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MESSAGE FROM THE CHAIRMAN

Assalamualaikum and *Salam Sejathera*

It is my great pleasure to welcome all the parcipants to the Postgraduate Symposium on Biocomposite Technology 2015.This event is a continuation of our last year event which was successfully organized. The Postgraduate Symposium on Biocomposite 2015 is organized by Laboratory of Biocomposite Technology, INTROP, Universiti Putra Malaysia, with the aims to promote an academic gathering focusing on renewable resources used in composites and to improve learning experience of the postgraduate students by providing an opportunity to present their research findings. The organising committee received more than 30 research papers related to biocomposite technology and related research fields. We congratulate all postgraduate students, supervisors and research advisors for their interest in participating in the Postgraduate Symposium on Biocomposite Technology 2015. We hope this symposium will become a fruitful forum and discussion platform for the benefits of postgraduate students itself. In this symposium, keynote and invited speaker presentations will be delivered by speakers who are experts in biocomposites fields. As encouragements to the participants, we offer special awards for the best paper, best presenter and the best poster presentation. I hope postgraduate students enjoy while organizing and participating in this symposium. I would like to thank Director of INTROP, Prof. Dr. Paridah Md. Tahir for supporting Postgraduate Symposium on Biocomposite 2015. I sincerely hope that this event wil be a success and benefited every participant. I would like to express my sincere gratitude to all members of the organizing committee for their hard work in making this symposium a success.

Thank you,

Professor Ir. Dr. Mohd Sapuan Salit
Chairman
Committee of Postgraduate Symposium on Biocomposite Technology 2015

PROGRAMME AND PRESENTATION SESSION SCHEDULES84

TIME	PROGRAMME
8.30-9.00	**Registration**
9.00-9.15	**Opening ceremony**
9.20-9.45	**Keynote /Invited Lecture sessions**
10.00-10.30	**Tea break**
10.30-12.30	**Parallel Sessions A1 and B1**
12.30-2.30	**Lunch and Zuhur Prayer**
2.30-4:15	**Parallel Sessions A2 and B2**
4:30-4:45	**Tea break**
4:45-5:00	**Prize giving ceremony and closing**

Time:	Activities:	
08:30	Registration	Ms Ana Salleza Md. Salleh Ms Nadlene Razali
09:00	Emcee speech	Mastura Mohammad Taha
	Doa	Muhammed Lamin Sanyang
09:15	Symposium chairman speech	Prof Ir Dr Mohd Sapuan Salit
	Opening speech	Prof. Dr. Paridah Md. Tahir
09:20	Keynote session: Chairman: Prof Ir Dr Mohd Sapuan Salit	
	Keynote Lecture: Assoc Prof Dr Md Enamul Hoque University of Nottingham Malaysia Campus	
09:45	Invited Lecture: Mr NorazlanMohd Ramli SIRIM BHD	
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	Session chairman: **Dr Mohammad Jawaid**	Session chairman: **Dr. Juliana Abdul Halip**
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10:45-11:00	**P2:** *A framework of integration fuzzy AHP and ahp in natural fibre reinforced composites material selection* **M.T. Mastura and S.M. Sapuan**	**P10:** *The suitable material for design of prosthetic leg socket* **M.H Nurhanisah, M. Jawaid and M.T Paridah**
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12:00-12:15	**P7:** *A Review: Application of polymer based composite for automotive bumper system* **Amira Farhana Mohamad Tar**	**P15:***Effects of various plasticizers and concentration on physical properties of cassava films* **Ahmed. F. Edhirej, S.M. Sapuan, M. Jawaid, Z. N. Ismarrubie**
12:15-12:30	**P8:** *Mechanical properties of woven kenaf reinforced Phenolic resin produced using a hot press technique* **Suhad D. Salman, Z. Leman, M.T.H. Sultan, M. R. Ishak and F. Cardona**	**P16:***Application of cassava and cassava composites* **Ahmed. F. Edhirej, S.M. Sapuan, M. Jawaid, Z. N. Ismarrubie**
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2:45-3:00	**P18:** *Exploration on compatibilizing effect of non-ionic, anionic and cationic surfactants on mechanical properties of high density polyethylene/low density polyethylene/cellulose biocomposites* **A.K. Sudari, A. A. Shamsuri, E. S. Zainudin, P. M. Tahir**	**P25:** *The effect of alkalization and silane coupling agent treatment on the water absorption of roselle fibre reinforced vinyl ester composites* **R. Nadlene, S.M. Sapuan, M. Jawaid, M.R. Ishak, L. Yusriah**
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3:45-4:00	**P22:** *Application of Halpin-Tsai Equation to Kenaf Reinforced Biopolymer Composites* **M.R. Mansor, S.M. Sapuan,M.A. Salim, M.Z. Akop, and M. M. Tahir**	**P29:***Study on Lead Free Composite Solders (Bi- 205Ag) as a Replacement for Classic Solder at High Temperature Application* **Nima Ghamarian, M.A. Azmah Hanim, Zulkarnain Zainal, Lim Hong Ngee**
4:00-4:15	**P23:** *Study on linear relation between energy used by internal mixer screw and torque or time of compounding kenaf core polypropylene composites* **Haniffah W.H., Khalina A, S.M. Sapuan, Bakar E.S.**	**P30:** *Porous bioceramics in bone implants, methods: review* **Mohammed Sabah, M.A Azmah Hanim, C.A.N. Jaafar, S. M.Tahir and M.Norkhairunnisa**
4:15-4:30	**P31:** *Kenaf fibre reinforced polymer composites for automotive and construction applications* **N. Saba and M. T. Paridah**	
4:30-4:45	**TEA BREAK**	
4:45-5:00	Closing ceremony (Auditorium) Prize giving: Best paper, Best presenter and Best Poster Awards	

ABSTRACTS
AND
FULL PAPERS

Advanced Biomaterials (Biocomposites) in Biomedical Applications

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Abstract

Apparently composites refer to the hybrid/artificial materials consisting of two or more constituent materials that produce different characteristics from the individual component materials. However, many of the natural/living systems are essentially examples of composites more appropriately biocomposites such as bamboo, wood, human/animal bodies and so on. Furthermore, most of the organs/tissues are also biocomposites e.g. bone, cartilage, skin etc. Currently, artificially developed biocomposites are increasingly entering into biomedical applications besides many other early-established industrial applications (e.g. automotive, locomotive, aerospace, military). Amongst numerous sectors of biomedical engineering, tissue engineering that often uses biomaterial/biocomposite along with living cells represents new medical therapies for injury and organ disease. This paper discusses the development and evaluation of tissue engineering scaffolds using hybrid biopolymers and rapid prototyping technology.

Keywords: Biocomposite, Biomedical Engineering, Tissue Engineering

Biocomposites as Alternative to Timber

Protecting the planet, one building at a time

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Abstract

From the rooftops of renowned hotels and neo-modern skyscrapers to the decks of meditation retreats, today's designs often utilise natural aesthetics through wooden surfaces and textures. But with the emerging consciousness of our impact on the environment, the challenge lies in bringing a natural touch to these edifices in a sustainable fashion. Biocomposite is emerging as a substitute for real timber in creating upmarket building materials particularly decking, flooring and wall claddings as well as furniture. The composite, which uses sawdust or rice husk mixed with plastics feedstock, offers the look and feel of wood, but with a lower impact on forests and greater resistance against weather, termites and rot. It can be produced in a wide range of shapes and used in many different types of projects without losing its dimensional integrity. Because of its composition, the material does not require any additional protective coating and is inherently non-toxic and low-maintenance. Compared with untreated wood, it is not easily susceptible to rot, and is highly resistant to water, termites and fungus.SIRIM's bio-composite decking material promises zero reliance on the wood derived from logging, while also providing the look of a natural surface for attractive decking and flooring.

A Brief Review on Thermoplastic Starch Reinforced Nanocellulose Film

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Abstract

Petroleum-based plastics widely used in polymer composite which cannot be recycled and accumulate as waste material. On the contrary, the thermoplastic starch film through weak in strength and easily affected by water, it subsists as an effective solution to petroleum-based plastic because it is renewable, poses no smell, colorless, tasteless and readily abundant. Moreover, the additions of nanocellulose into thermoplastic starch have shown drastic improvement in its mechanical properties and water permeability of the film. In this review, we focus to cover research carried out during 2009 to 2014 and aimed to highlight major findings on addition of nanocellulose to mechanical and water permeability of thermoplastic starch film.

Keywords: Thermoplastic starch; Nanocellulose; Mechanical properties; Water absorption

Introduction

There are various types of plastic packaging on the market such as those made of polyethyleneterephthalate (PET), high density polyethylene (HDPE), low density polyethylene (LDPE), polyvinylchloride (PVC), polypropylene (PP), polystyrene (PS) and are normally derived from petroleum sources. These plastics takes a long time to decompose biologically thus pose negative impacts on the environment and human health. Several life cycle assessments performed on plastic packaging revealed that, according to analytical comparisons of the six scenarios of plastic waste management, recycling option is the most preferable action[1]. However, plastic that is being dispersed into households are largely mixed, contaminated and damaged more than often causing problems for reuse and recycling of the materials [2]. Therefore, the emergence of environmentally friendly plastic or bio-plastic which does not need to be

recycled and biologically degradable provides an effective solution to the above problems.

Thermoplastic starch

Starch is potentially used for raw material in the manufacture of bioplastic. Due to isotropic property, it is scentless, tasteless, colorless and besides it is non-toxic to human and ecologyand also biodegradable [3]. However, it is naturally very brittle because of the existence of a strong cohesive bonds between the polymer molecules [4]. Therefore, plasticizer such as water, glycerol, sorbitol and aceticacidis added to the starch to reduce the brittleness, which helps to increase the flexibility and extensibility of starch by reducing intermolecular forces between the adjacent polymer chains [5]. The transformation of native starch to this thermos plastic starch occurs due to the disintegration of molecular order within the starch granules when high temperatures, mechanical stirring and plasticizers are added which subsequently caused the starch granules to swell and burst resulting the releasing of starch polymers contained therein. This polymer form hydrogen bonds with plasticizers and thus prevents them from realigning into crystallite formation again.

Thermoplastic starch reinforced nanocellulose

The addition of plasticizer into the starch does not only produce a stretchable and flexible film layer, but is also mechanically low, has barrier properties with high water uptake. This drives the starch into competitiveness disadvantage position compared to durable and water insoluble petroleum based plastic. To overcome the obstacles, nanocellulose is added to the mixture of starch and plasticizer. It is expected that with the addition of nanocellulose, it will increase the mechanical strength of the film composite. Nanocellulose can be obtained by two methods, namely through biological synthesis by using bacteria (bottom-up method) or extraction from plants (top-down method). It has high strength and stiffness besides low weight with a valuable sustainable property as it is derived from renewable sources. In addition, it decomposes naturally and bio-compatible, thus making it the best candidate for reinforcing the thermoplastic starch.

In literature, it is difficult to determine the true strength and modulus of cellulose crystals. Hence, theoretical calculations and numerical simulations have been used to estimate theaxial modulus of crystalline cellulose. Based on theoretical calculations, axial modulus of cellulose crystalis of 58G Pa where the value is close to the value owned by Kevlar (60 GPa). In addition, thenumerical simulation shows cellulosecrystalshavea high eraxial modulusof 180GPa compared to Kevlar which shows 125GPa. Tensile strength of nano cellulose shows the theoretical predictionsof 0.3 to 22GPa. This high valueis due to extended chaircon formation shown by cellulose crystal, as well as the high density of covalent bonds per cross-sectional area in addition to the high inter and intra molecular hydrogen bonding sites [6][7].

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Mechanicalproperties

The comparison between nano cellulose with cellulose fiber was acquired thatnanocelluloseprovides a better film properties, such as mechanical, barrier and morphology at the same percentage of filler [8]. The increasingtensilestrengthproved by many researches such as an increment of1.28-foldfor thermoplastic wheatstarchfilmreinforced2 wt% of oil palm empty fruit bunches nanocellulose [9], 1.9-fold for pea starch film reinforced 10 wt% hull peananocellulose [8], 1.08-fold for potato starch film reinforced 0.5 wt% nanocellulose [10] and more than doublingof the strength of the modified potato filmrein forced 0.5 wt% sisal nano cellulose [11] when compared to their respective neat films. The increase intensile strength is driven by good compatibility between these two types of poly saccharides in addition to high numberof activesites on nanocellulose surfaces which then allow more hydrogen bonds to be formed with the starch matrix. Hydrogen bonds formed between nano cellulose leads to adensepercolating cellulosic network that is fundamental for the reinforcing effects. Table 1 below shows some of the mechanical properties of thermoplastic
starch reinforced nano cellulose film derived from different mixing formula.

Water absorption properties

Film resistance against water attacks is one of the most important properties for food packaging applications, particularly those involving high water activity food. According to Tongdeesoontorn [14] water in solubility is a prerequisite to improve product intensity and improve moisture barrier property as well as overall shelf-life[14]. Thermo plastic starch film is highly hydrophilic in nature indicated by high water diffusion rate [15] but nano cellulose addition to thermos plastic starch film has been successful in lowering these properties. The films water uptake is decreased by 1.19-fold when added with 10 wt% nano cellulose [8], similarly a 1.92-fold decrease when added with 70 wt% nano cellulose [16], likewise by a 1.5-fold decrease when added with 10wt% nano cellulose [15]and by 1.14-fold decrease when added with 2 wt% nano cellulose [9]compared to their respective neat films.

Table 1: Mechanical properties of thermoplastic starch reinforced nanocellulose film derived from different mixing formula

Starch type	Nanocellulose origin	Nanocellulose extraction process	Nanocellulose properties	Fabrication technique	Mechanical properties							Testing method	Reference
Pea	Pea hull	Acid hydrolysis	7 to 12 nm in diameter, 240 to 400 nm long and 10wt% content inside the film	Solution casting	Properties	Neat starch film			Nanocellulose added film			Universal testing machine	[8]
					Tensile strength (MPa)	4.1			7.9				
					Elongation at break (%)	30.1			62.2				
Wheat	Oil palm empty fruit bunches fibre	Acid hydrolysis	1 to 100 nm in size, 2wt% content inside the film	Solution casting	Properties	Neat starch film			Nanocellulose added film			Universal testing machine according to ASTM D638-03	[9]
					Tensile strength (MPa)	3.66			4.68				
					Elongation at break (%)	1.79			1.19				
Potato	Commercial microcrystalline cellulose	High pressure homogenization	392 ±36 nm in size, 1wt% content inside the film	Solution casting	Properties	Nanocellulose added film			Nanocellulose and gum arabic added film			Texture analyser according to ASTM D882-91	[12]
					Tensile strength (MPa)	3.27 ± 1.1			4.79 ± 1.3				
					Elongation at break (%)	22.8 ± 2.9			36.6 ± 3.8				
Wheat	Oil palm empty fruit bunches fibre	Acid hydrolysis	1 to 100 nm in size, 2wt% content inside the film	Solution casting	Properties	Chitosan added film			Nanocellulose and chitosan added film			Universal testing machine according to ASTM D638-03	[13]
					Tensile strength (MPa)	3.96			5.25				
					Elongation at break (%)	30.27			25.71				
Potato	Commercial nanofiber	-	0.5wt% content inside the film	Hand lay-up and compression molding	Properties	A1	A2	B1	B2	C1	C2	Universal testing machine according to ASTM 882	[10]
					Tensile strength (MPa)	6.5	8.5	7.0	104	3.5	108		
					Elongation at break (%)	2.4	19.8	3.2	21.7	6.5	21.7		
					Young modulus (MPa)	20	215	60	250	30	456		
Potato (acetylated potato and maleated potato)	Sisal fibre	Acid hydrolysis	30.9 ±12.5 nm in diameter, 5wt% content inside the film	Solution casting	Elastic modulus 1-fold increase compared to neat potato starch film							Universal testing machine Instron according to ASTM D638	[11]

A1= neat starch film A2 = thermoplastic starch compressed with ramie mat
B1= nanocellulose added to thermoplastic starch B2 = B1 compressed with ramie mat
C1 = nanocellulose and polycaprolactone added to thermoplastic starch C2 = C1 compressed with ramie

Water molecules use capillary movement to flow along the fiber-matrix inter phase before diffuse into bulk resin. The existence of micro cracks form apathway for the water and facilitate the penetration [10]. Inside the thermos plastic starch film, there are spaces that are easily penetrated by water molecules because of loosely arranged polymer chains. Nano cellulose inhibits the movement and swelling of the starch, it acts like a sieve that slows down the water movement. It is said that the nano cellulose homogeneity is an important factor in lowering the water up take where more disperse nano cellulose produce higher water permeability film. Vigneshwaran (2011) reported that gum arabic was used to help spread out nano cellulose in starch matrix by lowering thes urface energy of nano cellulose therefore prevent them from stacking together and forms aggregation. 2-fold increase in water permeability observed in nano cellulose composite reinforced thermos plastic starch when mixed with gum arabic, compared to only 1.4-fold for the film without the additive. It is proved that nano cellulose in the starch matrix shows the homogenous dispersion [12]. In addition to nano cellulose, the starch itself also undergoes chemical modification to increase film water permeability. It is found that acetylated starch and maleated starch reinforced with 5 wt% sisal nanocellulose has successfully increase 30 percent of contact angle against water, resulting in a much less water sensitivity [11].

Conclusions

The application of thermoplastic starch reinforced nano cellulose as plastic packaging that poses sustainable propertiesmay solve problems of plastic waste accumulation. It has good mechanical properties and lower water uptake compared to neat starch film.

As far as research is concerned, many scientific researches prefer fabrication techniques using solution casting method which is suitable only for a small scale laboratory study. Mechanical mixing of thermoplastic starch with nano cellulose filler using conventional mixing techniques used largely in factories will invite new challenges. The homogeneity of nano cellulose can hardly be achieved through this process, but not impossible. This possibility can be prevailed by the addition of additive like gum arabic to help with the dispersion of the nanocellulose as discussed above. In addition, the film blow molding techniques commonly used for the production of plastic bag will likely produce weaker film compared to its solution casting counterpart. In this process, nanocellulose is only given a very short period of time to form cellulosic network. Yet the network is fundamental to the strength of the composite film. Therefore, pre-mixing of thermoplastic starch with nano cellulose before under going this process will optimistically shorten the time required for both to react. Regardless of the solution to the stated problems, it will definitely bring more innovations in the research field.

Acknowledgements
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References

[1] U. Arena, M. Mastellone, and F. Perugini, "Life cycle assessment of a plastic packaging recycling system," *Int. J. life cycle,* 2003.
[2] G. Davis and J. Song, "Biodegradable packaging based on raw materials from crops and their impact on waste management," *Ind. Crops Prod.*, 2006.
[3] S. Flores, L. Famá, and A. Rojas, "Physical properties of tapioca-starch edible films: Influence of filmmaking and potassium sorbate," *Food Res.*, 2007.
[4] J. Maran and V. Sivakumar, "Development of model for barrier and optical properties of tapioca starch based edible films," *Carbohydr.*, 2013.
[5] C. Romero-Bastida and L. Bello-Pérez, "Physicochemical and microstructural characterization of films prepared by thermal and cold gelatinization from non-conventional sources of starches," *Carbohydr.*, 2005.
[6] W. Hamad, "On the development and applications of cellulosic nanofibrillar and nanocrystalline materials," *Can. J. Chem. Eng.*, 2006.
[7] R. Xiong, X. Zhang, D. Tian, Z. Zhou, and C. Lu, "Comparing microcrystalline with spherical nanocrystalline cellulose from waste cotton fabrics," *Cellulose*, 2012.
[8] Y. Chen, C. Liu, P. Chang, X. Cao, and D. Anderson, "Bionanocomposites based on pea starch and cellulose nanowhiskers hydrolyzed from pea hull fibre: effect of hydrolysis time," *Carbohydr. Polym.*, 2009.
[9] M. Salehudin and E. Salleh, "Empty Fruit Bunch (EFB) Cellulose Nanofiber as Filler for Starch Based Biopolymer Reinforcement," *2013 CIGR Sect. VII*, 2013.

[10] S. Arun, K. Kumar, and M. Sreekala, “Fully biodegradable potato starch composites: effect of macro and nano fiber reinforcement on mechanical, thermal and water-sorption characteristics,” *Int. J. Plast.*, 2012.
[11] J. Morán, A. Vázquez, and V. Cyras, “Bio-nanocomposites based on derivatized potato starch and cellulose, preparation and characterization,” *J. Mater. Sci.*, 2013.
[12] N. Vigneshwaran, L. Ammayappan, and Q. Huang, “Effect of Gum arabic on distribution behavior of nanocellulose fillers in starch film,” *Appl. Nanosci.*, 2011.
[13] M. Salehudin, E. Salleh, S. Mamat, and I. Muhamad, “Starch based Active Packaging Film Reinforced with Empty Fruit Bunch (EFB) Cellulose Nanofiber,” *Procedia Chem.*, 2014.
[14] W. Tongdeesoontorn, “Effect of carboxymethyl cellulose concentration on physical properties of biodegradable cassava starch-based films,” *Chem. Cent.*, 2011.
[15] S. Karimi, A. Dufresne, and P. Tahir, “Biodegradable starch-based composites: effect of micro and nanoreinforcements on composite properties,” *J. Mater.* 2014.
[16] A. Svagan, M. Hedenqvist, and L. Berglund, “Reduced water vapour sorption in cellulose nanocomposites with starch matrix,” *Compos. Sci.*, 2009.

A Framework of Integration Fuzzy AHP and AHP in Natural Fibre Reinforced Composites Material Selection

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Abstract. Material selection is one of the process that include in design activity to determine the most preferred material for the product design according to product design specification. In this study, integration of fuzzy analytic hierarchy process and conventional analytic hierarchy process is proposed to select the material from natural fibre for reinforcement. Both of methods are used to get a consistent fuzzy decision and more accurate priority vector. The result will be applied in determination of the most preferred natural fibre according to the weight of each parameter that calculated from pairwise comparison matrix.

Keywords:material selection, fuzzy analytic hierarchy process, analytic hierarchy process, natural fibre reinforced

Introduction

In design process of natural fibre reinforced composite-based product environment, selection of type of natural fibre has to carefully determined in order to match with the product design specification since that the fibre's properties can be vary according to the origin of the fibre itself. There are a few methods that have been proposed and applied by a few of researchers. Analytic Hierarchy Process (AHP) seems to be quite a popular tool because of the simplicity of the algorithm. Besides, AHP has been widely applied in various fields such as logistics, marketing, military, agriculture, business and others. Thomas L. Saaty has developed this method to derive the scale of relative importance for alternatives and criteria [1]. This method has been used since it is developed because of the effectiveness of the weighting value to deal with complex decision making problem. The weighting values that obtained from this method are acceptable in objective or subjective evaluation which by using pair-wise comparisons that can derive accurate ratio and scale priorities [2]. Therefore, according to the studies in material selection process, researchers tend to use AHP as a tool to determine that best materials for their products [3-9].

In 1996, Da Yong Chang in his research has introduced Fuzzy Analytic Hierarchy Proses (FAHP) with extent analysis to obtain a crisp priority vector from triangular fuzzy comparison matrix [10]. This method is used by convert the linguistic assessment to triangular fuzzy number. The "extent analysis" here is referred to as a consideration of the extent of an object to be satisfied for the goal [11]. Although FAHP has been criticized by the architect of conventional AHP itself, Thomas L. Saaty in his papers [12-14], there are numerous researchers that are still using fuzzy judgment in FAHP to determine the importance weights in any kind of research field. Chang's article in 1996 has been cited by more than 1500 researchers till today as a proof that FAHP is applicable in all kind of research field. Thus, in this study, a framework of natural fibre composite material selection process will be proposed based on integration of FAHP and AHP approaches.

Methodology

Steps that are proposed in this study are shown in Figure 1. Fuzzy analytic hierarchy process and conventional analytic hierarchy process are applied in parallel. Initially, the hierarchy of attributes was constructed and each of the elements was comparing over another on pairwise basis. A nine-point scale is used as it is commonly used to show the judgment or preference between options as equally, moderately, strongly, very strongly, or extremely preferred. For FAHP, triangular fuzzy number technique is used to represent a pairwise comparison and a same scale of crisp number was applied for pairwise comparison matrix in AHP.

Fuzzy AHP with extent analysis

A triangular fuzzy number can be denoted as $M=\tilde{a}_{ij}= (l_{ij},m_{ij},u_{ij})$ where $l \leq m \leq u$, l and u stands for lower and upper value of the support M, respectively and m is the mid value of M. Triangular numbers are used to represent the assessment from equal to extremely preferred for scale M_1,M_3, M_5, M_7 and M_9 while M_2,M_4,M_6,M_8 are used to represent the moderate values.

Next, a triangular fuzzy comparison matrix expressed by

$$\tilde{A} = (\tilde{a}_{ij})_{n\times n} = \begin{bmatrix} (1,1,1) & (l_{12},m_{12},u_{12}) & \ldots (l_{1n},m_{1n},u_{1n}) \\ \vdots & (1,1,1) & \ldots \quad \vdots \\ (l_{n1},m_{n1},u_{n1}) & (l_{n2},m_{n2},u_{n2}) & \ldots \quad (1,1,1) \end{bmatrix}$$

where $\tilde{a}_{ij}=(l_{ij},m_{ij},u_{ij})$ and $\tilde{a}_{ij}^{-1} =(1/u_{ij},1/m_{ij},1/l_{ij})$ for $i,j =1,\ldots,n$ and $i \neq j$.

Analytic Hierarchy Process

In AHP, the common hierarchy is used and nine-point scale with conversion value from triangular fuzzy number to crisp number is used to construct pairwise comparison matrixes. The calculations to obtain priority vectors are by calculate the eigenvector of comparison matrix. The eigenvalue of the comparison matrix would be used to calculate the consistency index, CI and consistency ratio, CR of the judgments in pairwise comparison.

Discussion

The most commonly parameters that include in material selection requirements are strength, density, cost and environmental safety. In order to determine the most reliable and durable material, the selected material should has a highest value of strength among the other candidates. On the other way, density of the best material sometimes would contradict with the requirement that needed in the selected material. This would imply that some conflict just occurs for design engineer to choose the type of material that can fit with both required parameters. Contradiction among the required parameters would make the material selection process going complicated and false decision would cost more and the most suitable material could not be selected. Therefore, by applying FAHP together with AHP, the most important criteria would be considered first and more according to the value of weight that calculated using both methods. For instance, in a case of design of automotive component, selection of material for anti-roll bar (ARB) should consider the strength, stiffness, density, cost and from non-toxic material. According to the requirements from the customer after being weighted, performance of the selected material including strength and stiffness is on the top of priority rank during determination of the best material for ARB. However, the challenge is to select material that is lightweight which is contradicting with the higher value in strength and stiffness. Therefore, from FAHP and AHP the more important parameter would give more weight after normalization and this would help design engineer to do unbiased decision making and more logic.

Conclusions

Selection of the best material for a particular product design could be a complicated job if all the criteria are not identified accurately and not organized in systematic way. Besides that, the least preferred material could be selected if the most important parameter is considered as less important during decision making process. This would cause the team to do repetition task if the selected material fail to perform according to what have been decide earlier in product design specification. More time, more cost and more energy are required if false decision is made. Thus, fuzzy number could be used as a tool to select the most preferred material as the result is compared and analysed with crisp number. The consistency ratio is checked to prove that the decision from fuzzy number is parallel with crisp value.

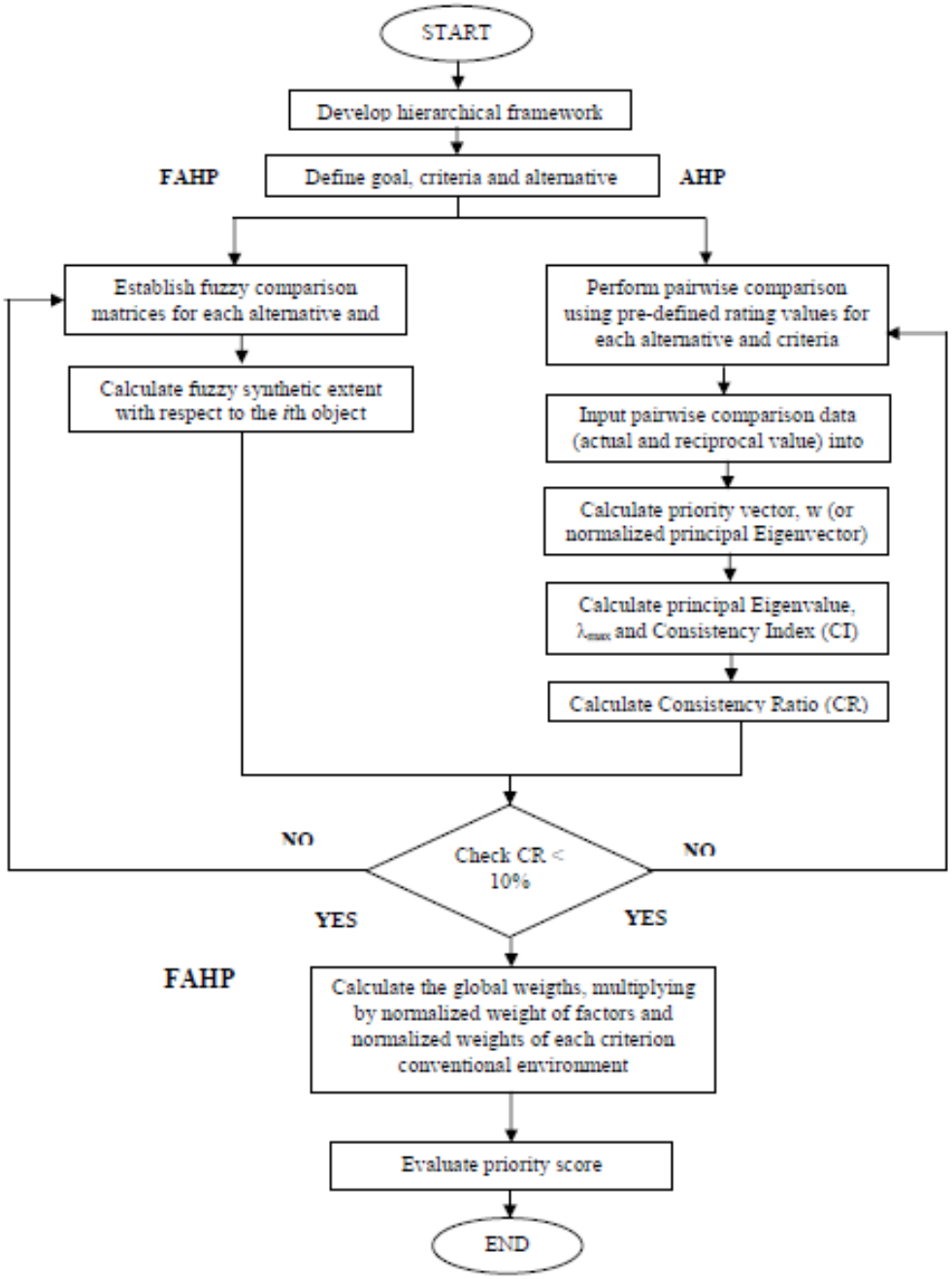

Figure 1: Methodology of integration FAHP and AHP

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References

[1] T. L. Saaty and L. G. Vargas, "Uncertainty and rank order in the analytic hierarchy process," *Eur. J. Oper. Res.*, vol. 32, no. 1, pp. 107–117, Oct. 1987.

[2] D. Dalalah, F. Al-oqla, and M. Hayajneh, "Application of the Analytic Hierarchy Process (AHP) in Multi- Criteria Analysis of the Selection of Cranes," vol. 4, no. 5, pp. 567–578, 2010.

[3] S. M. Sapuan, J. Y. Kho, E. S. Zainudin, Z. Leman, B. A. Ahmed Ali, and A. Hambali, "Materials selection for natural fiber reinforced polymer composites using analytical hierarchy process," *Indian J. Eng. Mater. Sci.*, vol. 18, no. August, pp. 255–267, 2011.

[4] M. R. Mansor, S. M. Sapuan, E. S. Zainudin, a. a. Nuraini, and a. Hambali, "Hybrid natural and glass fibers reinforced polymer composites material selection using Analytical Hierarchy Process for automotive brake lever design," *Mater. Des.*, vol. 51, pp. 484–492, Oct. 2013.

[5] M. R. Mansor, A. Hambali, M. D. Azaman, U. Teknikal, D. Tunggal, S. M. Sapuan, E. S. Zainudin, and A. A. Nuraini, "Material Selection Of Thermoplastic Matrix For Hybrid Natural Fiber / Glass Fiber Polymer Composites Using 2 . Material Requirements for Automotive Parking Brake Lever."

[6] B. A. A. Ali, S. M. Sapuan, E. S. Zainudin, and M. Othman, "Java based expert system for selection of natural fibre composite materials," vol. 11, no. October, pp. 1871–1877, 2013.

[7] M. R. Mansor, S. M. Sapuan, A. Hambali, E. S. Zainudin, and A. A. Nuraini, "Materials Selection of Hybrid Bio-Composites Thermoset Matrix for Automotive," vol. 8, no. 8, pp. 3138–3142, 2014.

[8] M. R. Mansor, S. M. Sapuan, E. S. Zainudin, A. A. Nuraini, and A. Hambali, "Application of Integrated AHP-TOPSIS Method in Hybrid Natural Fiber Composites Materials Selection for Automotive Parking Brake Lever Component," vol. 8, no. April, pp. 431–439, 2014.

[9] A. Mustafa, M. F. B. Abdollah, N. Ismail, H. Amiruddin, and N. Umehara, "Materials selection for eco-aware lightweight friction material," *Mech. Ind.*, vol. 15, no. 4, pp. 279–285, Jun. 2014.

[10] D.-Y. Chang, "Applications of the extent analysis method on fuzzy AHP," *Eur. J. Oper. Res.*, vol. 95, no. 3, pp. 649–655, Dec. 1996.

[11] C. K. Kwong and H. Bai, "Determining the Importance Weights for the Customer Requirements in QFD Using a Fuzzy AHP with an Extent Analysis Approach," *IIE Trans.*, vol. 35, no. 7, pp. 619–626, Jul. 2003.

[12] T. L. Saaty, "There is no mathematical validity for using fuzzy number crunching in the analytic hierarchy process," *J. Syst. Sci. Syst. Eng.*, vol. 15, pp. 457–464, 2006.

[13] T. L. Saaty and L. T. Tran, "On the invalidity of fuzzifying numerical judgments in the Analytic Hierarchy Process," *Math. Comput. Model.*, vol. 46, no. 7–8, pp. 962–975, Oct. 2007.

[14] T. L. Saaty and L. T. Tran, "Fuzzy Judgments and Fuzzy Sets," *Int. J. Strateg. Decis. Sci.*, vol. 1, no. 1, pp. 23–40, 2010.

[15] I. Bereketli and M. Erol Genevois, “An integrated QFDE approach for identifying improvement strategies in sustainable product development,” *J. Clean. Prod.*, vol. 54, pp. 188–198, Sep. 2013.
[16] D. Endah and H. Raharjo, “Evaluating Relationship Of Consistency Ratio And Number,” pp. 207–216, 2003.

Chemical Treatments of pineapple leave fibre for Natural Fiber-Reinforced Polymer Composites: A Review

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Abstract. Natural fibre based composites are being studied because of its physical and mechanical specification and environment concerning issues. Natural fibres are easily available, safe handling, low density and mechanically strong. Pineapple leave fibre (PALF) is waste material and abundantly available as compared to other natural fibres. Chemical, physical and mechanical properties of PALF are highly significant. Many types of surface treatments of PALF have been studied for the better interfacial adhesion between fibres and polymer matrices. PALF as a reinforcement compatible with polymers (thermoplastics and thermosets), and provide excellent physical and mechanical properties to developed polymer composites. In this review, we covered different method of natural fibre especially PALF treatment or modification reported in literature and its effect on structure, chemical composition, physical, and mechanical properties of PALF reinforced polymer composites and its applications.

Keywords: Pineapple leaves fibre (PALF), surface modification, PALF bio-composites and surface treatment

Introduction

Many natural fibres such as pineapple leaf (PALF), kenaf,sisal, coir, jute and ramie are prospective fibres to replace synthetic fibre or other conventional reinforced materials in composites. The natural fibres have very attractive characteristics such as stiffness [1] impact resistance[2], flexibility[3], and modulus[4]. Availability of naturals fibres are in abundant amount [5].In 2009, Thailand produces 1.894 million tonnes, the Philippines produced 2.198 million tones and Brazil produced only 1.43 million tonnes. In 2001 the production of Costa Rica, Cote d'lvoire and Philippines ware 322,000 tonnes, 188,000 tonnes and135,000 tonnes respectively[6].Generally agriculture materials and forest product produce 30-40% waste materials, which can be use by value added processing. The low density natural fibres are good alternative of glass fibres on low load bearer places [7], and have other properties like

economical, less density, low machining abrasion [3], non-carcinogenic [8], shock absorber[1][2], enhanced energy recovery[8]renewability and bio-degradability.
Pineapple leave fibre (PALF) is one of the most waste materials among natural fibres. Thailand is contributing major part of PALF production [9][10]. Tones of PALF are being waste as agricultural waste and soil dumping, which can be use by value added process. The expansion of bio-composites has amplified industrial usage that would release the possibilities to minimize the wastage of renewable materials. It promotes a non-food based market for agriculture industry[11]. PALF white in colour, smooth, and glossy as silk, medium length fibre with high tensile strength and it has a softer surface than other natural fibres and it absorb and maintain a good colour [12] Cellulose content of PALF is (70–82%), lignin (5–12%), ash (1.1%) and low microfibrill angle, which provides high specific modulus and strength[9][13]. Cellulose have high hydroxyl group which promote the hydrophilic character of natural fibres and resins are hydrophobic in nature, so the interfacial adhesion is very poor and prone to absorb moisture[14].Through using the surface improvement with coupling agent PALF can be an effective reinforcement for polymers[13].Mechanical properties of the composites depend on the compatibility of the fibres and matrix in interfacial bonds.

Natural fibres have been focused among industries, research scholars and composite firms for replacement of glass fibres to natural fibres. Natural fibres are very easy to access, economical, low density and eco-friendly. The rapid growth of natural materials and environment concern have been become a good indication of use of natural fibres in coming decades. In recent era, PALF is being use in effective manner for a polymer matrix to develop practical composites with good quality mechanical strength [15]. Recently, pineapple leaf fibres have been considered as eco friendly reinforcing agents. It is successfully being use in for polymer matrix to develop good composites with good mechanical properties. Individually pineapple leaf fibres (PALF) have outstanding mechanical properties.

Treatment

PALF is dipped with 2% detergent solution at 70°C for 1 hour, and then washed with running water, then dry it by using vacuum oven at 70°C. Thereare four deferent typesof surface modification to complete this modification process. First, treated fibre with detergent again treated with mixture of ethanol and benzene in the ratio of 1:2 for 72 hours at 50°C, then washed thoroughly with conductivity water and air dried. Second procedure of modification treated with 5% of NaOH solution for 1 hr at 30°C. Then washed with fresh water and dry it in open shed area.In third experiment, graft copolymerization of acrylonitrile (AN) onto defatted PALF is used [Cu_2+- IO_4 –] as starter at normal temperature (40°C) in aqueous condition. In last surface modification, defatted fibre treated with AN, acetone, and pyridine (as catalyst) at 60°C for 2 hr, then washing the fibres with acetic acid and acetone then water is used for washing and finally vacuum drying [16]It is known as cyanoethylated, allabove four surface modifications for preparation of non-oven mat composites[17]

PALF were dipped into different concentration 0.5, 1 and 5% (w/v) NaOH aqueous solution at room temperature for half an hour and then washed it in running water and finally wash with acidified water (HCI 0.1 M) for neutralize pH of fibre. Then treated fibres dried by using oven at 60°C for 24 hours.

Silane A 1100 [y-amino propyl tri(ethoxy) silane]

Acetone acidified water (95:5) and silane (3 ml) prepare a solution of hydrolyzed silane solution. Fibres immersed into for one and a half hours. The fibres colour changed in to yellow colour. Then kept the fibres into woven at 60°C for drying process.

Silane A 172 [vinyl tri(2-ethoxy methoxy) silane]

It is a combination of oven dried fibers, carbon tetrachloride, dicumyl peroxide (2% by wt of fiber) and vinyl tri(2-ethoxy methoxy silane) (4% by wt of fiber) was provided temperature for two hours with magnetic stirrer, then material was sieved and dried.

Permanganate (KMn04) treatment

Fibres is immersed into $KMnO_4$ solution in acetone in 5 different concentration (0.005, 0.01, 0.015, 0.02 and 0.05%)for I min. And then dried it into air woven. [18].

lsocyanate treatment (PMPPIC)

This method contains PMPPIC in different percentage level (5,8 and 10 by weight), fibres immersed into toluene containing chemical at 50°C for 30 minutes. Then fibres were dried by using oven at 70°C for 2 hours[19]

Bio-composite

Pineapple leave fibres is environment friendly reinforcing material for matrix to develop an effective composite with high mechanical strength, because of its outstanding mechanical properties, researchers are more focus on PALF containing thermoset[17], thermoplastic[20], biodegradable plastics[21], and natural rubber[22][13].

PALF-epoxy composite

PALF-epoxy composite is treated with three types of surface treatments.First,with NaOH (5% w/v NaOH aqueous solution), second, the deposition of diglycidyl ether of bisphenol A (DGEBA) from toluene solution (1% w/v DGEBA),and third is alkalization combined with the deposition of DGEBA (5% w/v NaOH/1% w/v DGEBA). These interfacial modifications enhance bonding strength and mechanical properties of PALF-epoxy composites. Treated PALF showed almost 2-2.7 times higher interfacial shear strength than untreated PALF-epoxy resin matrix. The fibres treated (alkalization combined with DGEBA deposition) showed strongest interfacial adhesion. Treated PALF with 5% NaOH and 5% NaOH/1% DGEBA showed better flexural and impact properties of unidirectional PALF-epoxy composites[15].

PALF reinforced low density polyethylene composite

PALF reinforced low density polyethylene composite has been studied on the effect on surface modification. Many chemicals treatment have been use such as NaOH, PMPPIC, silane and peroxide to modify the interfacial bonding. Mechanical properties has improved significantly, SEM studies revealed that fibre-matrix adhesion has fracture on surface. PMPPIC treatment minimised the hydrophilicity of fibre and improve the mechanical properties of composites.PMPPIC treatment of fibres exhibit maximum interfacialinteractions[19].

PALF-reinforced polyester composites

The study of PALF-reinforced polyester composites investigated themechanical propeties like tensile, flexural and impact behaviour of function of fibre loading and fibre surface modification. Surface modification of PALF has been investigated through dewaxing, alkali treatment,cyanoethylation and grafting ofANonto dewaxed PALF. The mechanical properties of PALF-reinforced polyester composites are optimised at a fibre loading of 30 wt%. AN grafted PALF composite exhibited maximum tensile strength (48.36 MPa) on 10 wt%. cyanoethylated PALF showed 41% and 27% more flexural and impact strength respectively than control composite[17].

Application

Surface modified PALF is renounce for making machinery parts like belt cord, conveyor belt cord, transmission cloth, air-bag tying cords and some cloths for industry uses [23]. PALF is very good for carpet making because of its chemical processing, dyeing behaviour, and aesthetically pleasing fabric [24]. The use of pineapple leaf fiber can be considered relatively as new in the paper manufacturing industry in Malaysia [25].Fibers from lignocellulosic sources are used in various applications such as building constructions, different types of boards like particle boards, insulation boards, etc. and many other sections like cosmetics, medicine and for other biopolymersand fine chemicals [26][27][28].

Conclusions

The utilization of pineapple leaf fibre in composite material is a new source of materials which have good mechanical and physical properties, economic, eco-friendly and recyclable. However, the main issues of PALF are its hydroscopic nature which is a big hurdle for fibre utilization as a reinforced material in polymer composites. Surface modifications of PALF require doing for improving interfacial adhesion of PALF with polymers in fabrication of polymer composites. Synthetic fibres can be replace or partially substitute with PALF in fabrication of composite products for different applications. We concluded from this review that several works reported on chemical modification of PALF, physical, and mechanical properties of PALF reinforced polymer composites and its hybrid. Pineapple is one of the natural fibre which having highest cellulosic content nearly 80%.These properties are suitable for its application as building and construction materials, automotive components, and furniture. From this review it clear that limited work carried out on thermal properties,

electrical properties, thermal conductivity, dynamic mechanical analysis and modelling of mechanical properties of PALF reinforced polymer composites

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References

[1] L. . Sherman, "Natural fibers: the new fashion in automotive plastics," *Plast Technol*, vol. 45, no. 10, pp. 62–68, 1999.
[2] C. Sydenstricker, T.H., Mochnaz, S., Amico, S, "Pull-out and other evaluations in sisal-reinforced polyester biocomposites," *Polym Test*, vol. 22, no. 4, pp. 375–80, 2003.
[3] S. Manikandan, K.C., Diwan, S.M., Thomas, "Tensile properties of short sisal fiber reinforced polystyrene composites," *J Appl Poly Sci*, vol. 60, pp. 1483–97, 1996.
[4] A. Eichhorn, S.J., Baillie, C.A., Zafeiropoulos, N., Mwaikambo, L.Y., Ansell, M.P., Dufresne, "Current international research into cellulosic fibres and composites.," *J Mater Sci*, vol. 39, no. 9, pp. 2107–31, 2001.
[5] t C. Maldas, D., Kokta, B.V., Ra, j R.G., Daneaul, "Improvement of the mechanical properties of sawdust wood fibre-polystyrene composites by chemical treatment.," *Polymer*, vol. 29, no. 7, pp. 1255–65., 1988.
[6] S. D. (FAOSTAT), "FAO (Food and Agriculture Organization of the United Nations)," *http://www. fao.org*. 2011.
[7] J. M. Kenny, *Natural fibre composites in the European automotive industry. Proceedings of the Sixth International Conference on Wood fibre-Plastic Composites*. 2001, pp. 9–12.
[8] R. Karnani, R., Krishnan, M., Narayan, "Biofiber-reinforced polypropylene composites," *Polym Eng Sci*, vol. 37, no. 2, pp. 476–82., 1997.
[9] N. Kengkhetkit and T. Amornsakchai, "Utilisation of pineapple leaf waste for plastic reinforcement: 1. A novel extraction method for short pineapple leaf fiber," *Ind. Crops Prod.*, vol. 40, pp. 55–61, Nov. 2012.
[10] S. Ketnawa, P. Chaiwut, and S. Rawdkuen, "Pineapple wastes: A potential source for bromelain extraction," *Food Bioprod. Process.*, vol. 90, no. 3, pp. 385–391, Jul. 2012.
[11] B. M. Cherian, A. L. Leão, S. F. de Souza, L. M. M. Costa, G. M. de Olyveira, M. Kottaisamy, E. R. Nagarajan, and S. Thomas, "Cellulose nanocomposites with nanofibres isolated from pineapple leaf fibers for medical applications," *Carbohydr. Polym.*, vol. 86, no. 4, pp. 1790–1798, Oct. 2011.
[12] and C. T. C. Py, J. J. Lacoeuilhe, *The pineapple-cultivation and uses*. Maisonneuve & Larose, Quae, 1987.
[13] N. Lopattananon, K. Panawarangkul, K. Sahakaro, and B. Ellis, "Performance of pineapple leaf fiber–natural rubber composites: The effect of fiber surface treatments," *J. Appl. Polym. Sci.*, vol. 102, no. 2, pp. 1974–1984, Oct. 2006.

[14] A. Alvarez, V. A., Ruscekaite, R.A, Vazquez, "Mechanical properties and water absorption behaviour of composite made from biodegradable matrix and alkaline treated fibre," *J Compos Mater*, vol. 37, no. 17, p. 1575, 2003.
[15] N. Lopattananon, Y. Payae, and M. Seadan, "Influence of Fiber Modification on Interfacial Adhesion and Mechanical Properties of Pineapple Leaf Fiber-Epoxy Composites," *Wiley Intersci.*, 2008.
[16] P. B. Samal RK, Mohanty M, "Effect of chemical modification on FTIR spectra; physical and chemical behavior of jute II.," *J Polym. Mater.*, vol. 12, pp. 235–40, 1995.
[17] S. Mishra, M. Misra, S. S. Tripathy, S. K. Nayak, and a. K. Mohanty, "Potentiality of Pineapple Leaf Fibre as Reinforcement in PALF-Polyester Composite: Surface Modification and Mechanical Performance," *J. Reinf. Plast. Compos.*, vol. 20, no. 4, pp. 321–334, Mar. 2001.
[18] J. George, S. S. Bhagawan, and S. Thomas, "Improved interactions in chemically modified pineapple leaf fiber reinforced polyethylene composites," *Compos. Interfaces*, vol. 5, no. 3, pp. 201–223, Jan. 1997.
[19] J. George, S. S. Bhagawan, and S. Thomas, "Improved interactions in chemically modified pineapple leaf fiber reinforced polyethylene composites," *Compos. Interfaces*, vol. 5, no. January 2015, pp. 201–223, 1997.
[20] J. George, K. Joseph, S. S. Bhagawan, and S. Thomas, "Influence of short pineapple fiber on the viscoelastic properties of low-density polyethylene," *Mater. Lett.*, vol. 18, no. 3, pp. 163–170, Dec. 1993.
[21] S. Luo and A. N. Netravali, "Interfacial and mechanical properties of environment-friendly ' green ' composites made from pineapple fibers and poly (hydroxybutyrate-co-valerate) resin," vol. 4, pp. 3709–3719, 1999.
[22] and J. C. T. B. Bhattacharyya, A. K Biswas, "Short pineapple leaf fibre reinforced rubber composites," *Plast. Rubber Process. Appl.*, vol. 6, pp. 119–125, 1986.
[23] and P. R. K. A. Basu, K. P. Chellamani, "Jute and pineapple leaf fibres for the manufacture of technical textiles," *Asian Text. J.*, vol. 12, pp. 94–96, 2003.
[24] A. Anon, "guide to unusual natural fibres: pineapple leaf fibre (PALF)," *Textiles*, vol. 21, no. 3, 1992.
[25] M. S. T. Y. Yusof, M. R. Ahrnad, M. S. Wahab, M. S. Mustapa, "Producing Paper using Pineapple Leaf Fiber," *Advnnced Mater. Res.*, vol. 383–390, pp. 3382–3386, 2012.
[26] V. G. Geethamma, K. Thomas Mathew, R. Lakshminarayanan, and S. Thomas, "Composite of short coir fibres and natural rubber: effect of chemical modification, loading and orientation of fibre," *Polymer (Guildf).*, vol. 39, no. 6, pp. 1483–1491, 1998.
[27] J. A Youngquist, A. M. Krzysik, B. W. English, H. N. Spelter, and P. Chow, "Agricultural Fibers for Use in Building Components," *Use Recycl. Wood Pap. Build. Appl.*, no. 38, pp. 123–134, 1996.
[28] B. Jayanta, "Plastics filled with agrowastes," *Pop. Plast. Packag.*, vol. 48, no. 11, pp. 63–74, 2003.

Design of a Natural Fibre Reinforced Composite Classroom Chairs and Desks For Polytechnic Students

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Abstract. This study is to design a natural fibre reinforced composite classrooms chairs and desks for polytechnic students. Polytechnic Kuching students (n=416) were completed questionnaire using form of a body chart. The students were asked to identify body areas experiencing discomfort and to rate discomfort on a scale rating (0 to 5). Rate 0 (no pain), rate 1-3 (pain) and rate 4-5 (very pain). Part of questionnaire consisted of a discomfort checklist in form of a body chart. The evaluation of the seating shows acceptable to the students and if there were any outstanding problems. Students was constructed by having chairs users rank order 10 statements about comfort and choose three responses which gave the most consistent equal interval scale. The results in the main response of discomfort of classroom chair indicated thatmost students agreed that they feel cramped, stiff, numb, sore and tender, unbearable pain, barely comfortable and feel uncomfortable.The project was identified such as evaluate the comfort level for classrooms chairs and desks for polytechnic students. Overall, the comfortable rate on sitting for chairs and desks by the students and to design of a natural fibre reinforced composite classrooms chairs and desks for polytechnic students.

Keywords: Design composite, classroom chairs and desks, polytechnic students.

Introduction

Chairs and desks have become an important physical element of the learning environment [1]. Education as part of the service sector has also been a concern for ergonomists since the seventies, and various models concerning its application have been published [2]. During the past decade ergonomic research has focused especially on the design of work furniture based on the biomechanics of the human body [3]. Anthropometric information for chair design is mainly concerned with providing data on the stature of the people for whom the seats are designed [4]. Also, school

furniture dimensions, within the context in which it is used, may have an impact on some physical aspects of the students [5].

Wood-composite-manufacturing factories are in constant search for new sources of fibers as raw materials to be used in their production programs; therefore, potential natural or synthetic fiber should be taken into account to satisfy the raw materials needed for uninterrupted production [6]. This study is of particular interest as it concerns polytechnic students rather than younger children reported in most previous studies in Malaysia and elsewhere.

Methodology

This cross-sectional study was focused the comfort level for chairs and desks in classroom at Polytechnic Kuching Sarawak. However, the respondents were selected by students final year who have fulfilled the inclusion criteria. Meanwhile for the anthropometric data, a standardized measurement method for human body taken by different measure comparable. The results of ergonomic evaluation level for chairs and desks used in the classroom. Then, the results of comfort evaluate rate on sitting for chairs and desks by final year students. In the same time, proposed design of natural fibre reinforced composite classroom chairs and desks for polytechnic students.

Results and Discussion

The results of comfortable evaluate rate on sitting for chairs and desks

This part of questionnaire consisted of a discomfort checklist in form of a body chart. The students was asked to identify body areas experiencing discomfort and to rate this discomfort on a scale rating (0 to 5). Rate 0 (no pain), rate 1-3(pain), rate 4-5(very pain). Besides, this evaluation of the seat to see if it was acceptable to the students and if there were any outstanding problems. The results of the chair comfort evaluation are highlighted below.

Table 1: Discomfort Checklist in the Form of a Body Chart

Discomfort Checklist in the Form of a Body Chart	Classroom
Neck or head	2.3
Shoulder	2.19
Upper back	2.46
Arms & hands	1.82
Low back	2.38
Buttocks	2.63
Thighs	2.11
Knees	1.77
Calf (leg below knee)	1.72
Ankles & feet	1.65

(Rating scale 0 – No pain, 1 to 3 – Pain, and 4-5 – Very pain)

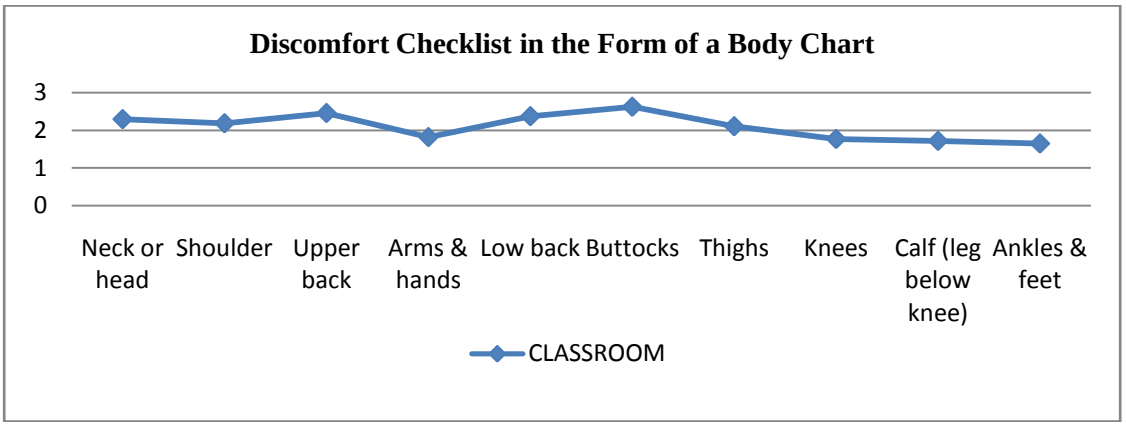

Figure 1: Discomfort Checklist in the Form of a Body Chart

This figure shows that symptoms of discomfort after long periods in the seat. Besides, this section was completed by 416 students [7]. Then mean hours in the seat at which time the students filled in their body chart was 2 hours. The body charts supported the checklist results in that the main areas of discomfort indicated for classroom chair were the upper back, buttocks and low back.

Table 2: General Comfort Scale

General Comfort Scale	Classroom
I feel cramped	2.27
I feel stiff	2.16
I feel numb	2.54
I feel sore and tender	2.38
I feel unbearable pain	2.21
I feel completely relaxed	1.83
I feel perfectly	1.80
I feel quite comfortable	1.89
I feel barely comfortable	2.28
I feel uncomfortable	2.43

(Rating scale 0 – Not agree, 1 to 3 – Agree, and 4-5 – Totally agree)

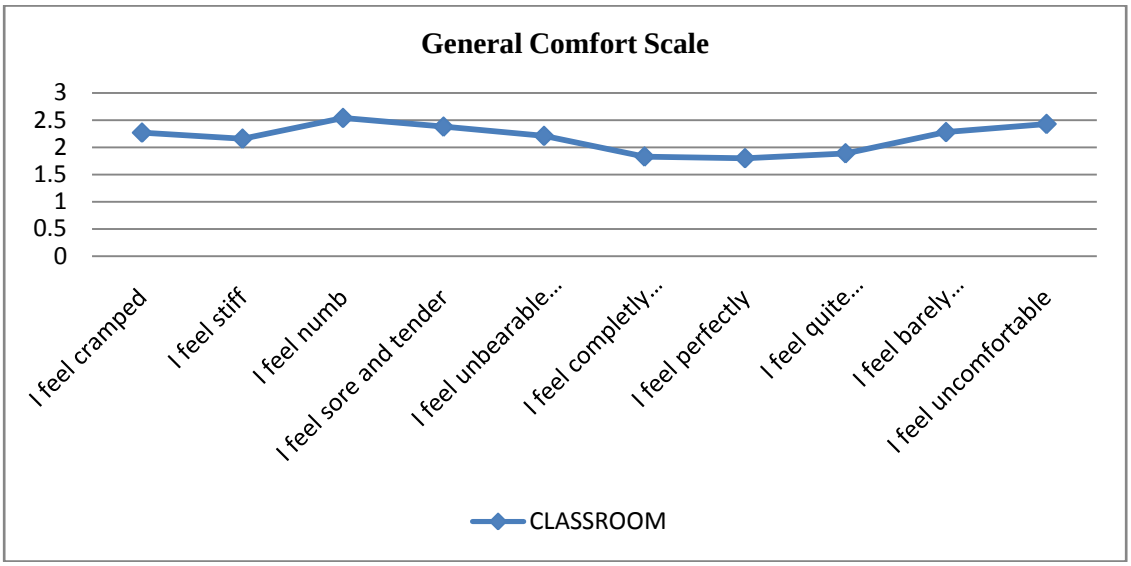

Figure 2:Discomfort checklist in the form of a body chart

General comfort scales have ranged from two point using adjective pairs (eg, rate the chair on your feelings and about comfort or discomfort. Besides, students was constructed by having chairs users rank order 10 statements about comfort and choose three responses which gave the most consistent equal interval scale. That response from 1 to 5 is just intolerable pain/discomfort and response from 6 to 10 just for noticeable pain/discomfort. The checklist results in that the main response of discomfort indicated for classroom chair their students mostly agree that they are feel cramped, stiff, numb, sore and tender, unbearable pain, barely comfortable and feel uncomfortable.

Conclusions

As conclusion, the students in these classrooms are discomfort to high level of stress on sitting using chairs and desks. Overall, the majority of the students feel uncomfortable. The recommended designs of a natural fibre reinforced composite classroom chairs and desks for polytechnic students to more comfortable and reduce cost manufacture.

Acknowledgements

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References

[1] M.G. Mohamed Thariq, H.P. Munasinghe, J.D. Abeysekara, Designing chairs with mounted desktop for university students: Ergonomics and comfort, *International Journal of Industrial Ergonomics* Vol. 40, 2010, pp. 8-18.

[2] M. Mokdad, M. Al-Ansari, Anthropometrics for the design of Bahraini school furniture. *International Journal of Industrial Ergonomics*Vol. 39, 2009, pp.728-735.

[3] G. Panagiotopoulou, K. Christoulas, A. Papanckolaou, K. Mandroukas, Classroom furniture dimensions and anthropometric measures in primary school. *Applied Ergonomics* Vol 35, 2004, pp. 121-128.

[4] H.I. Castellucci, P.M. Arezes, J.F.M. Molenbroek, Analysis of the most relevant anthropometric dimensions for school furniture selection based on study with students from one Chilean region. *Applied Ergonomics* Vol. 46, 2015 pp. 201-211.

[5] H.I. Castellucci, P.M. Arezes, J.F.M. Molenbroek, Applying different equations to evaluate the level of mismatch between students and school furniture.*Applied Ergonomics* Vol. 45, 2014, pp 1123-1132.

[6] H. R. Taghiyari, E. Bari, O. Schmidt. M. A.T. Ghanbary, A. Karimi, P. MD. Tahir, Effect of nanowollastonite on biological resistance of particleboard made from wood chips and chicken feather against Antrodia vaillantii, *International Biodeterioration & Biodegradation* Vol. 90, 2014, pp. 93-98.

[7] A. L. Mustapha, Pegawai Hal Ehwal Pelajar (Pengambilan & Data), Politeknik Kuching Sarawak.

Finite element analysis of torsional behaviour of filament wound kenaf yarn fibrecomposite hollow shafts

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Abstract. This paper presents a simulation study to investigate the behaviour of composite hollow shafts, with a specific focus on the maximum torsion capacity of the composite hollow shaft for different winding angles and stacking sequences. The results show that the torsion capacity is significantly affected by changing the winding angle and stacking sequences in the static torque test capacity properties. The maximum static torsion capacity of aluminum tube wound outside with kenaf yarn fibre/UP composite at a winding angle of 45º was the highest strength at 90º orientation.

Keywords: Kenaf yarn fibre,filament winding, hollow shafts, Finite elements.

Introduction

Kenaf fibre provides favourable advantages in terms of low cost and high raw material availability which suit perfectly the various applications of the composites industries. The current study focuses on the application of new materials, and is carried out to investigate the possibility of using kenaf as the fibre in a filament winding process reinforced with polyester. All the specimens used in this study were prepared using a filament winding process to determine the structural performance of the composite materials manufactured using the selected method.

Several findings have been reported concerning the best orientation angle and sequence in fabricating composite specimens using the filament winding method. Sevkat and Tumer [1] reported that the fibre orientation angle of 45º is the best for increasing the torsion stiffness. Mutasher [2],on the other hand, reported that the torsional capacity increases with the increase of the composite thickness. He also

reported that using a 45º sequence can withstand higher static torsion compared to a 90º sequence for hybrid aluminum/composite wound. The influence of the thickness and stacking sequence wound on the fatigue strength of the composites shaft has also been reported by Khalid et al. [3]. They found that for hybrid aluminum/composite shafts, the highest fatigue strength is obtained at +45/-45 fibre orientation. Apart from this, the selection of the final shaft geometry produced using the filament winding method also gives an effect on its torque performance. Lee et al. [4] reported achieving a 160% increase in torque capability using a one-piece hybrid aluminum/composite shaft construction compared to the conventional two-piece steel drive shaft configuration. The significant torque performance was also achieved at 75% less weight compared to the conventional two-piece steel drive shaft, indicating that a better lightweight property can also be obtained with the correct selection of the final fabricated shaft geometry.

Based on the above findings, it is highly possible to further increase the torsional properties of natural fibre composite shafts (such as from kenaf) through the selection and implementation of the best orientation angle and sequence in fabricating the composite specimens using the filament winding method. This will create further potential for natural fibre composites to be applied, especially for products which require both moderate strength and lightweight performances.

Materials and methods

Finite element analysis

Finite element analysis namely Abaqus 6.10 was used to verify the result obtained from the experiment. The meshing on kenaf composite hollow shaft was created from Abaqus software as shown in Figure 8. This module defines the material properties for the analysis and assigns those properties for the regarding part. Elastic orthotropic material model was used for simulating the elastic behaviour of composite. Therefore simulation model was able to give the maximum torque of predicted value for the given twisting angles. Four layers of filament wound kenaf yarn fibre reinforced unsaturated polyester hollow shaft composites were studied using FEA.

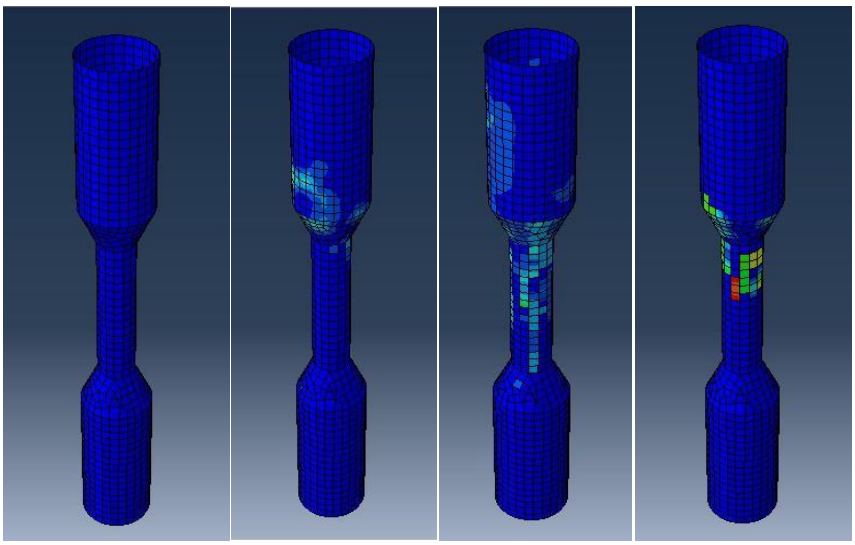
The material properties for each material were set. There were two materials used for this analysis; kenaf yarn fibre reinforced unsaturated polyester composite and aluminum (AA6063-T4). Both materials are elastic materials but for kenaf unsaturated polyester composite the elastic type was set as laminar and the values of Young's modulus (E_1 and E_2), poisson's ratio (υ_{12}) and shear modulus (G_{12}, G_{13} and G_{23}) were set. Meanwhile, the elastic type for aluminum (AA6063-T4) was set as an isotropic and the values of young's modulus and poisson's ratio were also set. The elastic constants for composite material and the mechanical properties of aluminum tube were obtained from Mutasher et al. [5].

In this analysis, two boundary conditions were assigned. The first boundary condition is at the bottom of the model was constrained to remain fixed ($U_1 = U_2 = U_3 = 0$) during the simulation. At the top of the model, second boundary condition was

applied. Since this model rotates at y-axis direction then, $U_1 = U_3 = 0$. Both boundary conditions were set with the cylindrical coordinate system (CSYS). The load was assigned as the shell edge load at the top of the model. Uniform distribution was set with shear traction and the magnitude was set as 10 000 N. At every step it will increase it will increased by one fold. A Tsai-wu failure criterion was set for this analysis.

Result and Discussion

Damage progression and stress counters of finite element simulation for a kenaf composite shaft are also shown in Fig. 1 (a-d). The effects of different winding angle and aluminum reinforcement on the torsion behaviour of the composite shaft and comparison between experimental and simulation is presented in Figure 2. Based on the results, the FEA result had lower angle of twist than the experimental results for the same torque. The kenaf hollow shaft composites show the FEA results of 45° kenaf composite were higher than experimental at the failure point of maximum torsion capacities where the results were 20 N m and 14 N m, respectively while for 90° kenaf composite, the results were 19 N m and 10 N m, respectively.



(a) (b) (c) (d)

Figure 1 : Damage progression and stress counters of finite element simulation for a kenaf composite shaft

However, a slight different trend was found at the failure point of composite kenaf reinforced with aluminum where experimental results were higher than FEA results . FEA result of 45° kenaf/aluminum composite was 33 N m which was approaching the experimental result of 30 N m. Similar to 90° kenaf/aluminum, it was found that the results of FEA and experimental were 28 N m and 26 N m, respectively.

Full comparison of torsional behaviour of kenaf aluminum and kenaf composites without aluminum at different winding angles of FEA result is also shown in Fig. 2. It shows the graph torque–twisting angle behaviour of four types of composite shafts. Based on the trend, the predicted torque for the composite shafts for both composite shafts reinforced with aluminum was significantly higher than composite shafts without aluminum while for the effect of winding angle , 45° showed better. These trends proved that the FEA results were similar with the results obtained by the experiment.

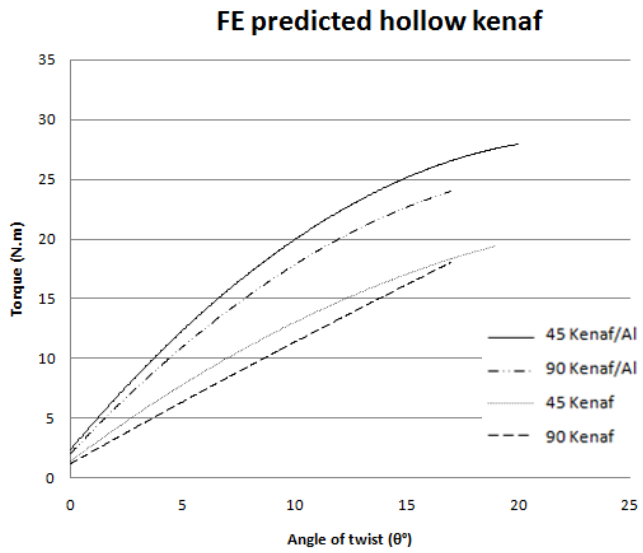


Figure 2 : Comparison of torsional behaviour of kenaf aluminum and kenaf composites without aluminum at different winding angles of FEA

In order to have a good understanding for both FEA and experimental results, the percentage result different of maximum torsion capacity between the experimental and FEA results is shown in Fig. 3. The result could be noted that the percentage differences of torsion capacity for 45° and 90° winding angles of composite shafts without aluminum were 26% and 45%, respectively while 9% for both composite shafts reinforced with aluminum.

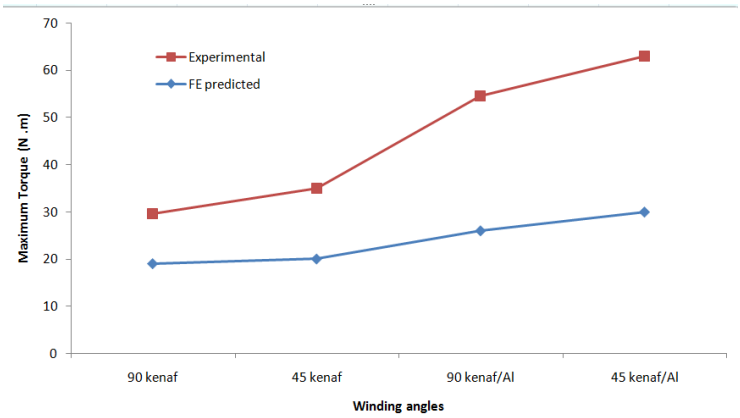


Figure 3 : The percentage result different of maximum torsion capacity between the experimental and FEA

Conclusion

A finite element study was carried out using Abaqus software 6.10 to verify the experimental results. The comparisons between the experimental and FEA results gave good agreement. The study also concluded the 45 degree winding angle of kenaf composite requires higher torque and can withstand higher static torsion compare to 90 degree. Torsional behaviour of the hollow shaft was also being influenced by the presence of the aluminium tube.

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References

[1] Wang X, Lou DYS, Zhang N. A helical tow model and numerical simulation of on-line thermal curing of thermoset composites in filament winding. Int J Heat Mass Transf 2004;47:4807–20.

[2] Khalid YA, Hamed AF, Sapuan SM. Pressure carrying capacity of reinforced plastic tubes. Polym Plast Technol Eng 2007;46:651–9.

[3] Meijer G, Ellyin F. A failure envelope for ±60_ filament wound glass fibre reinforced epoxy tubulars. Compos A Appl Sci Manuf 2008;39:555–64.

[4] Mutasher SA, Sahari BB, Hamouda AMS, Sapuan SM. Experimental study of bending fatigue characteristics of a hybrid aluminum/composite drive shaft. J Compos Mater 2007;41:2267–88.

[5] Khalid YA, Mutasher SA, Sahari BB, Hamouda AMS. Bending fatigue behaviour of hybrid aluminum/composite drive shafts. Mater Des 2007;28:329–34

Mechanical Properties of Tea Tree (*Melaleuca Alternifolia)*Fibre Reinforced Tapioca Starch Composite

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Abstract. We aim to utilize the tea tree fibre, a residue wastefrom the distillation process, to act as the reinforcement or filler in tapioca starch (TS) composite. The fabrication of tea tree fibres reinforced TS composite successfully developed using casting method. The mechanical properties are undergone in order to get the characterization of the composite. From the Young's modulus of mechanical test, reveal that tea tree branch reinforced tapioca starch (TTB/TS) was the stiffest, which is 5.63MPa (±0.091), followed by tea tree trunk reinforced tapioca starch (TTT/TS) with 4.78MPa (±0.04), tea tree leaf reinforced tapioca starch TTL/TS with 4MPa (±0.06) and TS with 1.25MPa (±0.037) respectively. The addition of 5% v/v of tea tree fibre as filler, the Young's modulus of TS improved 350.4% in TTB/TS, 282.4% in TTT/TS and 220% in TTL/TS. Above all, the tea tree fibre reinforced TS composites are better than TS composite in mechanical. Hence, all parts of the tea tree waste, namely tea trea leaf (TTL), tea tree branch (TTB) and tea tree trunk (TTT) have the value added potential fibres which can act as reinforcement or filler in a development of a green biocomposite.

Keywords: Tea tree, Melaleuca alternifolia, Natural fibre,Biocomposite, Agro waste, Reinforcement, Filler

Introduction

Native to Queensland to north-east New South Wales, Australia, *Melaleuca alternifolia* or commonly known as tea tree is a tall shrub or small tree in the plant genus *Melaleuca*. Famous of its oil, it has been used in a wide range of products, either as formulated or pure oil into many kinds of value-added products as a preservative, antiseptic, antibacterial, antifungal and even anti-pest agent [1, 2]. Tea tree oil is produced by steam distillation of the leaf, and the yield of oil is typically 1-2% of the wet weight of the plant [3]. Basically, after distillation, the tea tree leaf will be burned or will be composted. Richard [4] reported that in Australia, there are some companies dry the residue leaf after distillation, and then universally return it to the plantation as mulch. However, burning can cause environment pollution and the burning activities are monitored by local authorities. Based on these facts, it gives a

good consideration to use these left-over fibres or the agro waste which contain natural fibre as the filler in biocomposite [5].

There is a huge demand in using biofibre as reinforcer and/or biofibre in composite. Their highly specific stiffness, flexibility during processing and low cost (on a volumetric basis) makes them attractive to manufacturers. Various studies related to development of biocomposites reinforced by natural fibres had been done in Malaysia. From oil palm empty fruit brunch (EFB) [6], sugar palm tree [7, 8], and many more including banana pseudo-stem, coconut shell, kenaf, rice husk, coir, sugarcane bagasse, roselle, and wood [9]. No research work reported before regarding the utilization of tea tree fibre as the source for reinforcement or filler in biocomposite, except for its oil properties. Sánchez-González, et al., [10] have used tea tree oil in chitosan composite film in the making of antimicrobial film. Meanwhile, Liakos et al., [11] also successfully made film composite by using tea tree oil dispersed in sodium alginate (NaAlg) matrix with remarkable antimicrobial and antifungal properties. According to Abba et al., [12], various processing methods and conditions are used in the composite productions such as; compression molding process, injection molding, extrusion method, and thermoforming process. In this paper we are using casting method.

Material and methods

Preparation of materials

Throughout this study, tea tree fibres were taken from SEDIA's tea tree field at UNDP/GEF/SGP-IDS Demonstration Plot located at Mile 30 Kimanis, Papar coordinates of 5°
39' 27.95" N, 115° 54' 22.47' W [5]. Chainsaw was used to cut down the tree for easy ground harvesting of the fibres. The harvesting of the fibre was done manually using slashing knife. Fibre from different parts of the tree, which are tea tree leaf (TTL), tea tree branch (TTB), and tea tree trunk (TTT) were extracted as shown in Figure 2a to 2c. In order to obtain the fibre, every part of tea tree was cut separately and dried. The fibres themselves remain relatively unchanged during this process. Tapioca starch and glycerol were purchased from local market.

Fabrication of Composites

Tea tree fibres reinforced tapioca starch composites were prepared according to Chillo et al, [13] with slight modification. Tapioca starch (TS) and glycerol were mixed and dispersed in distilled water. The mixture contained 80% v/v of water, 15% v/v of glycerol and 5% v/v of tapioca starch. The mixture was heated under hot plate at 130^0C for 30 minutes under continuous stirring until the mixture gelatinized. After that, the mixture was cast in a mould sized 17cm x17cm x 1cm. The mixture was then dried under room temperature. The methods repeated for fabrication of tapioca composite reinforced tea tree fibres, by adding another 5% v/v of tea tree fibres. The tea tree fibres were added when the mixture gelatinized. The composite were labelled TS, TTL/TS, TTB/TS and TTT/TS respectively.

Mechanical properties

Tensile tests were carried out using universal testing machine type Gotech AI-7000 in accordance with ASTM D5083-(1996) [14]. The rate of testing used was 5 mm/min. 5 samples of size 10cm (L) x 1cm (W) x thickness in cm (T) were tested for different composite group.

Results and discussion

Figure 1 shows the tensile stress-strain behaviour of tea tree fibres reinforced tapioca starch composites for TS, TTL/TS, TTB/TS and TTT/TS. The deformation behaviour of the composites can be understood obviously through the tensile stress-strain curve. It is found that all type of composites give the brittle nature due to linearly increase in tensile stress at low strain follow by the drastic decrease shown in the tensile stress-strain curve. These phenomena might be due to the weakness reinforcement of the fibres which tend to break all at one strain parameter. From the figure, it is also found that TTB/TS exhibited higher stiffness compared to other composites due to its higher slope which can consider as the stiffest of material. The higher the slope, the more stiffness the materials are.

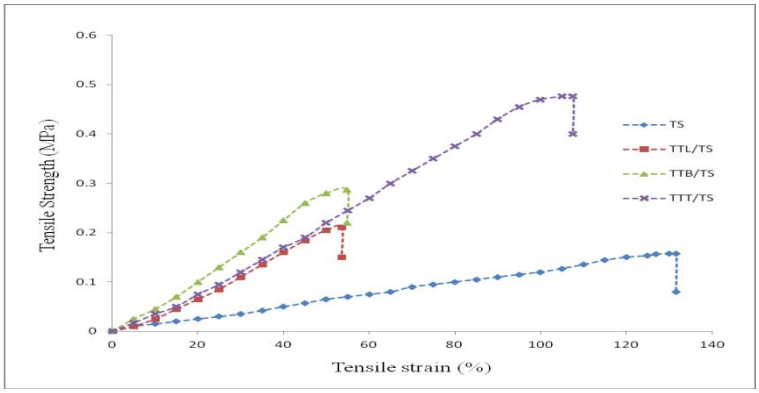

Figure 1: Tensile stress-strain curve of composite

Table 1: Mechanical properties of tea tree fiibres reinforced TS

Composite	Tensile strength (MPa)	Elongation at break (%)	Young's modulus 9 (MPa)
TS	0.157±0.01	131.6±5.01	1.25±0.037
TTL/TS	0.211±0.016	53.6±1.35	4±0.06
TTB/TS	0.287±0.015	54.8±1.61	5.63±0.091
TTT/TS	0.476±0.012	107.7±2.83	4.78±0.04

Table 1 shows the tensile strength of tea tree fibres reinforced tapioca starch. From the figure, it can be seen that TTT/TS was the highest of tensile strength with 0.476MPa (±0.012), followed by TTB/TS with 0.287MPa (±0.015), TTL/TS with 0.211MPa (±0.016) and TS with 0.157MPa (±0.01) respectively. Habibi et al., [15] support this result by the fact that mechanical properties are strongly influenced by the cellulose content as been discussed earlier. The addition of 5% v/v of tea tree fibre as filler, the

tensile strength of TS plastic improved 34.39% in TTL/TS, 82.80% in TTB/TS and 203.18% in TTT/TS.

Table 1 shows elongation at break of the composites. The ductility of a material determined from its elongation at break. The higher the value of elongation at break, the higher ductility of the material. It was found out that TS gave the highest value of elongation at break, which is 131.6% (±5.01), followed by TTT/TS with 107.7% (±2.83), TTB/TS with 54.8% (±1.61) and TTL/TS with 53.6%(±1.35) respectively. By the addition of 5% v/v of tea tree fibre, the ductility TS plastic decreased by 18.23% in TTT/TS, 58.35% in TTB/TS and 59.27% in TTL/TS respectively.

Table 1 shows the tensile modulus or Young's modulus of the composites. The gradient of the tensile vs elongation at break graph gives the tensile modulus. Tensile modulus indicates the relative stiffness property of a material [8]. Tensile modulus represents the ability of a material to resist deformation in tension. Ke and Yu [16]concluded that the higher the value of the tensile modulus, the stiffer the material is. From the Figure 3.4, TTB/TS was the stiffest, which is 5.63MPa (±0.091), followed by TTT/TS with 4.78MPa (±0.04), TTL/TS with 4MPa (±0.06) and TS with 1.25MPa (±0.037) respectively. The addition of 5% v/v of tea tree fibre as filler, the Young's modulus of TS improved 350.4% in TTB/TS, 282.4% in TTT/TS and 220% in TTL/TS.

In this study, the mechanical properties of TS are acceptable, as the results show it is near to previous study. By the addition of tea tree fibres, the mechanical properties are improved compared to TS even though the addition is only 5% v/v. The mechanical properties can be improved further if the vacuum process is undergone to avoid air bubbles [17]. As suggested by Oksman et al.,[18], poor dispersion of fibre in the matrix causing the fibre cannot perform its mechanical behavior effectively. Furthermore, as far as casting method is concern, it might not distribute all of the fibres evenly in the mould. Humidity is one of the most important factors affecting the mechanical properties of thermoplastic starch. For example, the mechanical strength of starch composites can reach 20 MPa in 0% RH; however, at high moisture conditions, the tensile strength may be below 1 MPa due to water plasticization [19].

Conclusion

From this study, the fabrication and characterization of tea tree fibre reinforced tapioca starch composites successfully developed and determined. All of the composite are superior compared to TS. The addition of 5% v/v of tea tree fibre as filler, the Young's modulus of TS improved 350.4% in TTB/TS, 282.4% in TTT/TS and 220% in TTL/TS. Hence, all parts of the tea tree waste, namely tea trea leaf (TTL), tea tree branch (TTB) and tea tree trunk (TTT) have the value added potential fibres which can act as reinforcement or filler in a development of a green biocomposite. The tea tree fibre, which considered agro waste, are successfully became the value added fibre, as it act as the reinforment in a composite. This gave a better option to industry in making green and renewable biocomposite.

References

[1] Southwell, I. and Lowe, R. 1999. *Tea Tree The Genus Melaleuca, Page 63-76, 81-89,* Netherland, Harwood Academic Publishers.

[2] Colton, R.T., Murtagh, G.J., Drinnan, J., and Clarke, B. 2000. *Tea tree oil*. Agfact P6.4.6. Second ed. NSW Agriculture, Orange.

[3] Carson, C.F., Hammer, K.A., Riley, T.V. 2005. Compilation and Review of Published and Unpublished Tea Tree Oil Literature. *Rural Industries Research and Development Corporation.* Publication No 05/151.

[4] Richard, L.D. The Australian tea tree oil industry. *In:* IFEAT International Conference 'Australia and New Zealand: Essential Oils and Aroma Chemicals - Production and Markets' 2003, 2-6 November Sydney, Australia.

[5] Rodney, J., Sahari, J., Mohd Kamal Mohd Shah., Review: Tea Tree (*Melaleuca Alternifolia*) As A New Material For Biocomposites. *Journal of Applied Science and Agriculture,10(3): 21-39, 2015*

[6] Abu Bakar, A., and Hassan, A.. 2009. Oil Palm Empty Fruit Bunch Fibre-Filled Poly (Vinyl Chloride) Composites. *In:* S.M. Sapuan, (eds.) *Research on Natural Fibre Reinforced Polymer Composites.* Serdang: Universiti Putra Malaysia Press.

[7] Sahari, J., Sapuan, S.M., Ismarrubie, Z.N., Rahman, M.Z.A. 2012. Physical and Chemical Properties of Different Morphological Parts of Sugar Palm Fibres. *Fibres & Textiles in Eastern Europe,* 20, 2(91) 23-26.

[8] Sahari, J., Sapuan, S.M., Zainudin, E.S., Maleque, M.A. 2013a. Mechanical and thermal properties of environmentally friendly composites derived from sugar palm tree. *Materials and Design,* 49, 285–289.

[9] Mohamad Awang, S.M. Sapuan, Riza Wirawan, and Hamim Izwan Hamdan. 2009. A review of the natural fibre reinforced polymer composite research. *In:* S.M. Sapuan, (eds.) *Research on Natural Fibre Reinforced Polymer Composites.* Serdang: Universiti Putra Malaysia Press.

[10] Sánchez-González, L., González-Martínez, C., Chiralt, A., Cháfer, M. 2010. Physical and antimicrobial properties of chitosan–tea tree essential oil composite films. *Journal of Food Engineering.* 98: 443–452.

[11] Liakos, I., Rizzello, L., Scurr, D.J., Pompa, P.P. 2014. All-natural composite wound dressing films of essential oilsencapsulated in sodium alginate with antimicrobial properties. *International Journal of Pharmaceutics*. 463:137– 145.

[12] Abba, H.A., Ismarrubie, Z.N., Sapuan, M.S. 2013. Review of Agro Waste Plastic Composites Production.*Journal of Minerals and Materials Characterization and Engineering,* 1, 271-279.

[13] Chillo, S., Flores, S., Mastromatteo, M., Conte, A., Lia Gerschenson, Del Nobile, M.A. 2008. Influence of glycerol and chitosan on tapioca starch-based edible film properties. *Journal of Food Engineering.* 88:159–168.

[14] ASTM D 5083 (1996). Standard test method for tensile properties of reinforced thermosetting plastics using straight-sided specimens. American Society for Testing Materials.

[15] Habibi, Y. El-Zawawy, W. Ibrahim, M.M. and Dufresne, A. 2008. Processing and characterization of reinforced polyethylene composites made with lignocellulosic fibres from Egyptian agro-industrial residues. *Composites Science and Technnology* .68:1877-1885

[16] Ke, J. and Yu, K. 2007. Fibre-reinforced cellulose acetate composites. (http://www.odec.ca/projects/2007/yuka7k2/tensile.html) (accessed on 23th April 2014).

[17] Espinel Villacrés, R.A., Flores, S.K., Gerschenson, L.N. 2014. Biopolymeric antimicrobial films: Study of the influence of hydroxypropyl methylcellulose, tapioca starch and glycerol contents on physical properties. *Materials Science and Engineering C.* 36: 108–117

[18] Oksman, K. Skrifvars, M. And Selin, J.F. 2003. Natural fibers as reinforcement in Poly (lactic acid) Composites. *Composites Sciences and Technology*. 63: 1317-1324.

[19] Liu, D., Zhong, T., Chang, P.R. Li, Kaifu, Wu, Q. 2010. Starch composites reinforced by bamboo cellulosic crystals. *Bioresource Technology.* 101: 2529–2536.

A review: application of polymer based composite for automotive bumper system

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Abstract. The use of composite materials has been growing rapidly in automotive industry. Nowadays, the use of composites mostly focused on automotive components such as automotive bumper system. Therefore, the research of the composite materials on the bumper system is carried out. The objective of this paper is to review the application of polymer based composite in automotive bumper system. There are 15 papers were reviewed that related to research area. The polymer based composites comprise of polymer mixed with the natural materials which result in different mechanical properties and the method to carry out the process of composite materials. Therefore, polymer based composite can be conclude as the top materials in application of the bumper system.

Keywords: polymer based composite, automotive bumper system

Introduction

In the worldwide automotive, the production rate is increasing which is about to reach 76 million cars every years reaching to 2020 [1].The main reason to use the polymeric composites in automotive is because of the need of reducing the fuel consumption and exhaust emission which give the outcomes of lightweight structure. Thus, an important component in automotive that uses polymer composite is a bumper system [2]. Polymeric materials are being applied in the automotive bumper system to satisfy the precise weight and performance requirement generally. Bumper system consists of three main parts; fascia, energy absorber and bumper beam [2]. The fascia is considered as a non-structural component which it cannot support the kinetic energy applied even though it is usually used for aesthetics and decreasing the aerodynamics drag force [5]. Since bumper fascia is a flexible and non-structural component, it influences the performance to unnecessary if the car has light weight under 2400lb with low cost. Therefore, the fascia is used with a foam energy absorber to support the fascia and to provide inexpensive energy absorber [4]. There are two types of bumper absorber which for lower impact that acts as a reversible energy absorber and the other one is when crashworthiness impact acts as an irreversible energy absorber [6]. When it comes to bumper beam, there are two different impacts occurred; high and low. The beam act to absorb the remaining impact force in low-impact test and provide the absorber with the bending resistance when high impact collision happens [6].

Materials and Methods

Materials

Polymer based composite consists of mixed elements of polymer and natural materials. One of the examples is the hybridization with glass fiber prepares a method to improve themechanical properties of natural fiber composites in application on automotive structural components. Hybrid material [5] using kenaf is invented from the Institute of Tropical Forestry and Forest Products (INTROP) in Universiti Putra Malaysia and the glass fibre from Fibreglass Enterprise (China) as consolidation and epoxy resin with hardener of 100:14 weight mixing ratio from LECO Corporation (USA) as the matrix [5].

Table 1: Hybrid materials mechanical properties

Material	Density	Strength	Modulus	Elongation at break	Reference
Kenaf	1.4	284-800	21-60	1.6	[8,9]
Glass	2.5	2000-3000	70	2.5	[8]
Epoxy	1.1-1.4	35-100	3-6	1-6	[10]

Clark et al [13] had done the research of glass fibre reinforced plastics on bumper beam which investigate the stress structure in the component. By the result, the material was 40% in mass fraction of glass fibre reinforced polypropylene. Besides, the study of differentiate the types of bumper beams offers different outcomes which resulting in polymer based composite of glass-mat thermoplastics (GMT) composite [14]. Some of the tests consider the section of the bumper to select the suitable materials. In developing a composite bumper beam, Cheon et al [15] applied two composite materials; the elbow section and the rest of bumper with carbon fiber epoxy composite and glass fibre epoxy composite materials respectively.

Methods

Every existing model that created in ANSYS 5.7 consists of a general strategy. Before creating and meshed the surfaces of the bumper structure, the Computer Aided Design (CAD) data is imported directly to ANSYS 5.7 to ensure the data can be read by the machines. A nonlinear explicit impact modelling elements is mostly used in LS DYNA ANSYS 5.7 [7]. SMC process is mostly used as the fabricating method to improve the mechanical properties of the materials. The short fibers are dissolved over the slurry resin passed through the sequential roller to make it denser, thinner and stable [5]. Suddin et al [12] presented Knowledge Based System (KBS) for the selection of suitable material of the automotive bumper beam by using screening and ranking process as selecting the material by its constraints and ranked the materials based on the objective using performance metrics respectively. Clark et al [13] described that as the experiment carried out, the automotive bumper beams three-dimensional models were developed and analyze using ABAQUS software.

Conclusions

As a conclusion, the review focus on thepolymer based composite to study the application on the automotive bumper system. Different types of composite gives different properties as the method to carry out the experimental. Each parts of the bumper systems has different structure which can be applied by the various choices of the polymer composite materials but the selection must fulfil the requirements of the other factors such as the mechanical properties and analysis of the test. Since the application has greatly implemented in the automotive industry, the polymer based composite can be categorized as the most suitable material for development bumper system besides contribute in reducing the cost, fuel consumption and exhaust emissions. The best development in the automotive offers the advantage in future.

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References

[1] WBCSD. Meeting the challenges to sustainability (mobility 2030). Geneva: World business council for sustainable development; 2004 April. Report No. 2-940240-57-4.

[2] S.M. Sapuan, M.A. Maleque, M. Hameedullah, M.N. Suddin, N. Ismail. A note on the conceptual design of polymeric composite automotive bumper system, Elsevier. 2005.

[3] W.D. Maahs, A.R. Janowiak, Composite bumper beams: comparing material choices, in: Proceedings of the Third Annual Conference on Advanced Composites, Detroit, Michigan, ASM Int, 15-17 September 1987, pp. 11-21.

[4]] D.L. Thurston, J.V.Camahan, in: A. Kusiak (Ed), Concurrent Engineering: Automation, Tool, Techniques, Wiley 1993.

[5] M.M. Davoodi, S.M. Sapuan, D. Ahmad, Aidy Ali, A. Khalina, Mehdi Jonoobi. Mechanical properties of hybrid kenaf/glass reinforced epoxy composite for passenger car bumper beam, Materials and Design 31 (2010) 4927–4932

[6] M.M. Davoodi, S.M. Sapuan, R. Yunus. Conceptual design of a polymer composite automotive bumper energy absorber, Materials & Design, Volume 29, Issue 7, 2008, Pages 1447-1452

[7] Ramin Hosseinzadeh, Mahmood M. Shokrieh, Larry B. Lessard. Parametric study of automotive composite bumper beams subjected to low-velocity impacts, Composite structure 68 (2005) 419-427

[8] Y. Xu, S. Kawata, K. Hosoi, T. Kawai, and Kuroda, S., Express Polymer Letter., 3(10) (2009) 657-664

[9] Z. Li, X. Zhou, and C. Pei., International Journal of Polymer Science., 2011 (2011) 1-7. Applied Mechanics and Materials Vol. 564 5

[10] A.K. Bledzki, A.A. Mamun, M. Lucka-Gabor and V.S. Gutowski., Express Polymer Letter., 2(6) (2008) 413-422.

[11] A. Hambali, S. M. Sapuan, N. Ismail, Y. Nukman. Material selection of polymeric composite automotive bumper beam using analytical hierarchy process. J. Cent. South Univ. Technol. (2010) 17: 244−256 DOI: 10.1007/s11771−010−0038−y
[12] Mohd Nizam Sudin, Mohd Ruzi Harun, Ahmad Tajudin Hamzah and Shamsul Anuar. A prototype of kbs for material selection in bumper beam design. (2007) Suranaree J. Sci. Technol. 14(3):215-222
[13] Clark G L, Bals C K, Layson M A. Effects of fibre and property orientation on 'C' shaped cross sections. SAE Technical Paper, 910049, 1991.
[14] Kumar A, Johnston C. Comparative performance of a compression: Molded I-section bumper beam with integrated mounting stays versus other GMT bumper design. SAE Technical Papers, 1998.
[15] Cheon S S, Choi J H, Lee D G. Development of the composite bumper beam for passenger cars. Journal of Composite Structures, 1995, 32: 491−499.

Mechanical Properties of Kenaf/Epoxy Composites

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Abstract. Natural fibres with variable properties are found in many engineering applications because of its low cost and biodegradability. There are many factors that can determine the properties of natural fibre composites. The effect of the kenaf fibre orientation (woven, non-woven mat, and unidirectional fibres) on the mechanical properties was evaluated, showing that the woven composites exhibit higher mechanical properties to the others. It was found that kenaf epoxy composites show lower strength than the neat epoxy resin.

Keywords: Natural fibre; kenaf; weight fraction; composites; flexural, impact strength

Introduction

Fibre reinforced polymer composites has been intensively used in various industries such as aerospace, marine, transportation and defence because of their advantages in term of strength and stiffness. Nowadays, there are trends in our society towards natures concern. Researchers also show their interest in using nature friendly materials in fibre reinforced polymer composites. Natural fibres reinforced polymer materials have been used in different applications. Fibres like pinus, sisal, flax, hibiscus sabdariffa, jute, etc. have all been proved to be good reinforcements in thermoset and thermoplastic matrices[1]. Innovative research was also conducted on other natural fibres such as; saccaharumcilliare fibres pine needle, durian skin, water hyacinth fibres and many more[2]. The use of natural fibres as renewable materials in polymer matrix composites will reduce the synthetic petroleum based fibres thus preserved the natural resources and the world as a whole.Natural fibres have many advantages over synthetic fibres such as biodegradable, low weight, low cost and acceptable properties. Natural fibres are now considered as serious alternative to synthetic fibres for use in various fields.Thakur and Thakur[3]in their review highlighted the advantages of natural fibre reinforced polymer composites such as

easy process, eco-friendly, and environmental awareness which encourage the use of natural fibres.

However, the disadvantages of natural fibres as reported in Khalil et al. [4] among others are; low modulus, low strength, and poor moisture resistance compared to synthetic fibre composites.Based on, tensile properties of the tensile modulus increases remarkably when using as unidirectional (UD) layers of kenaf fibers[5]. These results are due to the different kind of fabrics used in addition to void contents within the manufactured composites. In particular, the fibres, arranged along the load direction in the UD composites, give higher stiffness and strength than those of the mat composites (i.e. reinforced with short fibres randomly oriented).Kenaf fibres exhibited superior properties as reinforcement for polymeric composites under flexural loading conditions compared to other types of natural fibres[6]. Factors such as temperature, pressure and treatment time may affect the impact strength ofnatural fibre composites[7]. The mechanical properties of natural fibre composites can be related to the voids content within the manufacturing process[8].The main aim of this study was to investigate the mechanical properties kenaf hybrid composites with respect to fibre orientation. The results were compared with each other to find the best among them.

Materials and methods

Three kinds of kenaflayerhaving an areal weight equal to 660 g/m^2, were used as reinforcement of the composites. The first layer is woven kenaf which was produced by hand loom weaving process using kenaf yarns. The second layerconsists of randomly oriented fibresnon-woven mat. The third one is unidirectional yarn (UD) which is made of kenaf yarns, with length of 200 mm, oriented along 0 and 90^o. The composites were hand lay up with epoxy resin (DER331) of density 1.08g/m^3 cured with Jeffamine hardener. Pressure was applied using dead weights on the top of the mould 20cm x 20cm and cured at room temperature for 24 hours, followed by post-cured at a specified temperature. Volume fractions of kenaf in the composites were calculated by the following formula:

$$\boldsymbol{V_k} = \frac{\left(\frac{\boldsymbol{W_k}}{\boldsymbol{\rho_k}}\right)}{\left(\frac{\boldsymbol{W_k}}{\boldsymbol{\rho_k}}\right)+\left(\frac{\boldsymbol{W_m}}{\boldsymbol{\rho_m}}\right)} \qquad (1)$$

Density

After fabrication the density hybrid laminates were measured using the densimeter MD-200S, Mirage, according to the ASTM D792 standards. The void content was determined from the theoretical and the experimental density of the composites through Equation 1 and 2.

$$\text{Void contents (\%)} = \frac{\rho_{\text{theorethical}} - \rho_{\text{experimental}}}{\rho_{\text{theorethical}}} \times 100\% \qquad (2)$$

$$\rho_{\text{theorethical}} = \frac{1}{\left(\frac{w_f}{\rho_f} + \frac{w_m}{\rho_m}\right)} \quad (3)$$

where w_f is the fibre weight fraction, w_m is the matrix weight fraction, ρ_f is the fibre density, and ρ_m is the matrix density.

Tensile test

Tensile test was conducted to determine the stress-strain behaviour of the kenaf composite. The test was carried out using Instron 33R 4484 testing machine based on ASTM D 3039 on plates with a size of 200 mm x 25 mmx sample thickness for each composite. The samples were carefully cut from the laminate using wheel saw and finished to the accurate size. A standard head displacement at a speed of 5mm/min was applied. For each sample, three specimens were tested and average results were obtained.

Flexural test

The flexural test was conducted by 3-point flexure using Instron 33R 4484 tensile testing machine according to ASTM D 790-03. The rectangular samples of dimension 100 mm x 100 mmwere cut using circular saw. The speed of the crosshead was 2mm/min. Fivecomposite specimens were tested for each sample.

Impact test

Charpy test was performed on eight samples using an impact tester (Instron-CEAST 9050) with a hammer type 25J. Un-notched specimens from each composition, 5 specimens of 80mm×10mm×4mm were produced and tested.

Results and Discussion

Density and void

Voids are among the most common manufacturinginduced defects in composites, mainly due to the air entrapped during the composite manufacturing. Higher voids content resulted ina low resistance to water penetration and poor strength properties of composites. The density and void content are shown in Table 1. It was observed that the density of samples tested is almost similar because of low fibre content.

Table 1.Kenaf/epoxy composite investigated

Sample	Woven	Mat	UD
Thickness (mm)	9.57 ± 0.24	9.47 ± 0.08	10.65 ± 0.15
Fibre content (%)	4.72	4.79	4.73
Theoretical density (g/cm^3)	1.653	1.651	1.766
Real density (g/cm^3)	1.1537	1.1517	1.1553
Voids content (%)	0.302	0.302	0.346

Tensile properties

Tensile properties of composite samples were determined based on tensile strength and tensile modulus. Both parameters determine the greatest amount of stress a sample can withstand before failure occurred. The load-extension curves for hybrid composites with various layering sequences are shown in Figure 1.The curves determined the ultimate tensile strength and modulus of the composites. All the curves indicate non-linear behaviour and the point of deviation from linearity indicates failure initiation in kenaf layers.

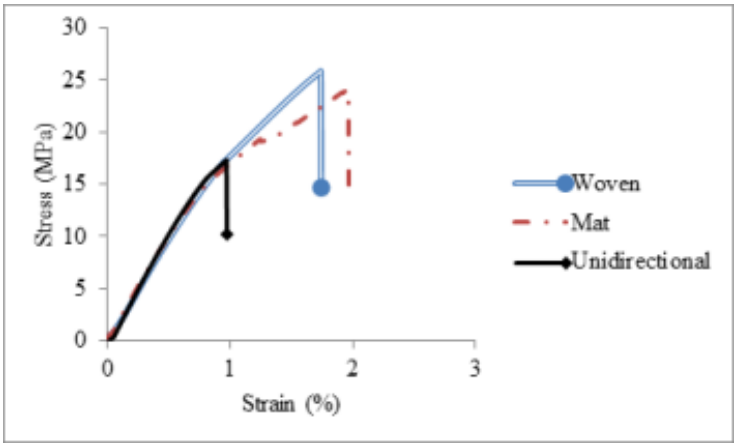

Figure 1. Stress-strain curves of samples tested.

Figure 2 shows the tensile properties of composites. All of the composites show lower tensile properties compared with tensile strength and modulus of neat epoxy which reported as 25.59MPa and 2.74GPa respectively[9]. Under tensile load, all three combinations exhibit sudden brittle failure after linear elastic region, with extensions up to approximately 1-2%. The tensile properties of sample tested are shown in Figure 3. Tensile test results show that when woven kenaf is used in combination with epoxy, the average tensile strength is 21.31 MPa. When the mat kenaf is used, the average tensile strength is 21.02 MPa, approximately 1.3% lower. The combination of UD kenaf, and epoxy reached a lowest tensile strength of 16.67 MPa, which can be attributed to poor bonding of fibre and matrix.Compared to the typical tensile strength of neat epoxy resins as reported previously [9] , the strength of woven, mat and UD kenaf/epoxy composites is approximately 16.70%,17.85% and 34.85% lower than neat epoxy respectively. Similar trends observed in gomuti/polyester composites as reported in [10]. They stated that the use of natural fibre as composite ingredients in polymer thermoset resins may not necessarily increase the tensile strength of the resin, but it reduces the amount of resin used with acceptable properties.

Flexural properties

Flexural strength determines the capability of laminated composites to withstand the bending before reaching the breaking point. **Fig.3** was plotted based on flexural testing of the composites. It shows typical load-extension curves for different samples. It is observed that both the flexural load-extension curves shows a linear increasing

trend and suddenly drops due to failure of specimen suggesting a brittle behaviour. The mat kenaf sample failed at a maximum stress of around 170N and extension of about 10mm while the woven sample failed at the maximum load of 141N and extension of 4mm.This figure clearly shows that the woven composites are much more rigid, and stronger. Based on the curves, mat samples experience a higher strain before breakage. Flexural strength and modulus of the composites are compared in Figure 4. The result indicates that mat kenaf composites haveslightly higher flexural strength(42.57MPa) compared to woven (42.30MPa) and UD samples (28.14MPa). Flexural modulus of mat kenaf samples was observed lowest compared to other samples.

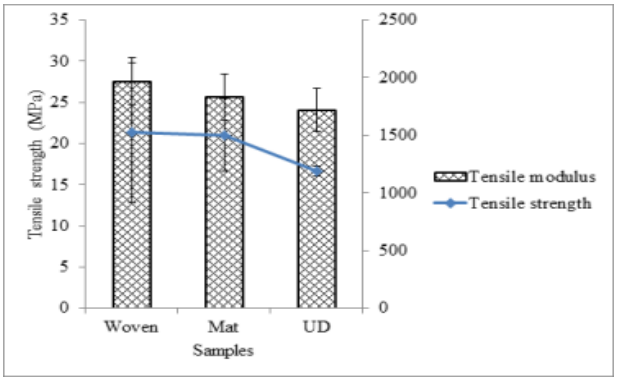

Figure 2. Tensile properties of kenaf/epoxy composites

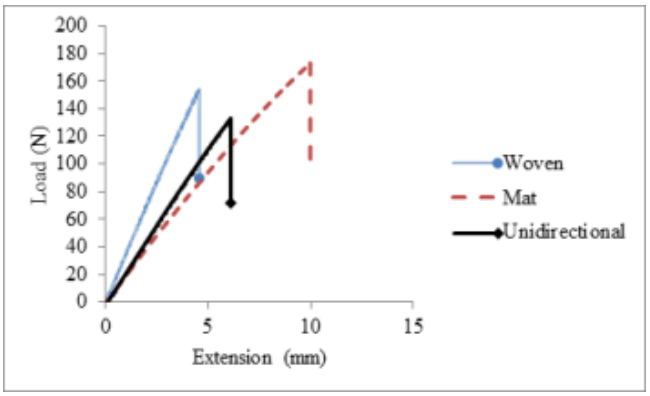

Figure 3. Flexural load-extension curves of the samples tested.

Impact properties

Charpy impact test was conducted to analyse the effect kenaf structure on the energy absorption capability of the hybrid composites. The toughness of composite or impact strength (kJ/m^2) was calculated by dividing the recorded absorbed impact energy with the cross-section area of the samples.Charpy impact testing has been used for impact toughness of natural fibre composites. The wovenand mat kenaf composites recorded higher impact strength, 21.32kJ/m^2 and 21.03 kJ/m^{2}respectively, compared with UD samples (16.67kJ/m^2).While examining the influence of fibre content on the mechanical and thermal properties of Kenaffibre reinforced thermoplastic

polyurethane, composites, El-Shekeil et al. [11] observed the negative effect on impact strengthas the fiber fraction increased.

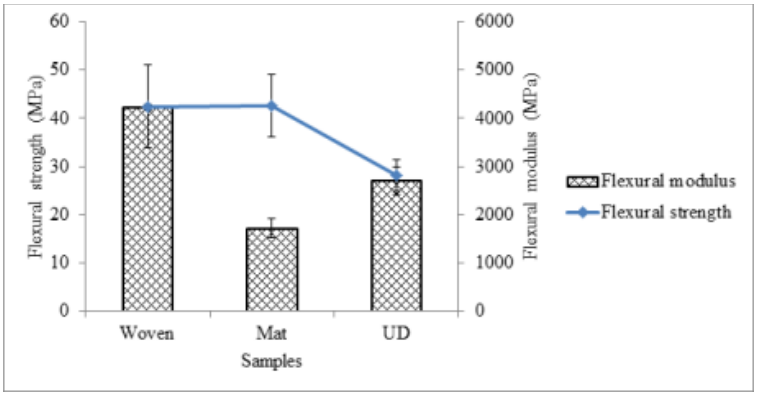

Figure 4. Flexural properties of the samples tested

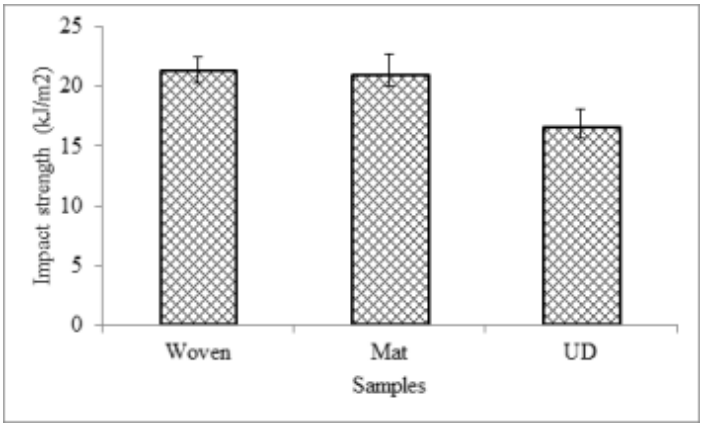

Figure5. Impact strength of samples tested

Conclusions

The mechanical properties of kenaffibre with an epoxy matrix were determined. Based on the results, the following conclusions can be drawn:

a) The laminates with woven fibresexhibited the higher strength, stiffness and impact strengthamong the investigated composites, followed closely by mat kenaf composites.
b) The density and void content are almost similar in the tested samples.
c) The tensile and flexural modulus properties of woven kenaf composites are the highest compared with other samples

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References

[1] A. S. Thakur, V. K., & Singha, Physico-chemical and Mechanical Characterization of Natural Fibre Reinforced Polymer Composites, *Iran. Polym. J.*, vol. 19, no. 1, pp. 3–16, 2010.

[2] R. Yahaya, S. Sapuan, M. Jawaid, Z. Leman, and E. Zainudin, Mechanical performance of woven kenaf-Kevlar hybrid composites, *J. Reinf. Plast. Compos.*, vol. 33, pp. 2242–2254, 2014.

[3] V. K. Thakur and M. K. Thakur, Processing and characterization of natural cellulose fibers/thermoset polymer composites., *Carbohydr. Polym.*, vol. 109, pp. 102–17, Aug. 2014.

[4] H. P. S. A. Khalil, S. Hanida, C. W. Kang, and N. A. N. Fuaad, Agro-hybrid Composite: The Effects on Mechanical and Physical Properties of Oil Palm Fiber (EFB)/Glass Hybrid Reinforced Polyester Composites, *J. Reinf. Plast. Compos.*, vol. 26, no. 2, pp. 203–218, Jan. 2007.

[5] V. Fiore, G. Di Bella, and a. Valenza, The effect of alkaline treatment on mechanical properties of kenaf fibers and their epoxy composites, *Compos. Part B Eng.*, vol. 68, pp. 14–21, Jan. 2015.

[6] B. F. Yousif, A. Shalwan, C. W. Chin, and K. C. Ming, Flexural properties of treated and untreated kenaf/epoxy composites, *Mater. Des.*, vol. 40, pp. 378–385, Sep. 2012.

[7] K. Saravanan and B. Dhurai, Effect of process parameters on impact strength of short jute fibre reinforced polypropylene composite board, *Indian J. Fibre Text. Res.*, vol. 38, no. March, pp. 92–95, 2013.

[8] N. M. Abdullah and I. Ahmad, Potential of using polyester reinforced coconut fiber composites derived from recycling polyethylene terephthalate (PET) waste, *Fibers Polym.*, vol. 14, no. 4, pp. 584–590, 2013.

[9] R. Yahaya, S. M. Sapuan, M. Jawaid, Z. Leman, and E. S. Zainudin, Effect of layering sequence and chemical treatment on the mechanical properties of woven kenaf–aramid hybrid laminated composites, *Mater. Des.*, vol. 67, pp. 173–179, 2015.

[10] A. Ticoalu, T. Aravinthan, and F. Cardona, Properties and behaviour of gomuti fibre composites under tensile and compressive load, in *Proceedings of the 22nd Australasian Conference on the Mechanics of Structures and Materials (ACMSM 22)*., 2011, pp. 531–535.

[11] Y. A. El-Shekeil, S. M. Sapuan, K. Abdan, and E. S. Zainudin, Influence of fiber content on the mechanical and thermal properties of Kenaf fiber reinforced thermoplastic polyurethane composites, *Mater. Des.*, vol. 40, pp. 299–303, 2012.

Kenaf fibre reinforced polymer composites for automotiveand construction applications

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Abstract. Proper utilization of the available natural resources and wastes became crucial for developing sustainable materials forautomotive and construction industries. Kenaf regarded as an industrial crop and belongs to family Malvaceae along with hibiscus (*Hibiscus hibiscum*L), hollyhock (*Althaearosea),* cotton (*Gossypiumhirsutum*L.) and okra (*Hibiscus esculentus),* are grown commercially in different countries including Malaysia. The plants possess wider range of adaptation to environments-climates, soils and rich source of cellulose compared to any of other fibre plant in profitable manufacture industry. Kenaf reveals a virtuous source of high and improved quality cordage fibre which can be processed into a variety of goods such as particle boards, fibre reinforced plastic components, pulp and paper, chemical absorbents and many others. Kenaf and its composites countless variability and its appropriateness make it suitable for utilizations as construction materials (such as boards of different densities, breadths, along with fire and insect resistance) and in automotive industries which wasrevealed by recent studies and research works reported in literature. Technical properties and performance, environmental, economic and social aspects strongly contribute toward adopting kenaf into the automotive and construction sector for developing various products.

Keywords: Natural fiber; Kenaf fiber; Polymer composite; Mechanical properties; Industrial applications.

Introduction

Industrial kenaf is a dicotyledonous plant usually cultivated for its bast fibre. Kenaf is commercially cultivated in more than 20 countries and more than 95 % of total productions are from China, India, and Thailand. Natural fibres such as, banana, hemp, flax, oil palm, kenaf, jute, sisal, etc. possess significant attention from past few years due to their environmentally green and renewable nature. Kenaf has been usefully utilized for a long period of time, traditionally as rope, canvas, sacking, etc. [1]. Kenaf stem consists of two parts, outer part known as bast fibres and inner part-core fibres. The bast fibres possess characteristic mechanical properties, making its suitable reinforcing materials to replace glass fibers as reinforcing materials in polymer composites[2]. Kenaf fibre regarded as one of the most important natural fibres, and is getting much attention from the composite industry as the potential polymer reinforcement's fibers. Kenaf fibres have lower thermal resistance as compared to synthetic fibres, similar to other natural fibers[1][3].Natural fibres reinforced composites are proposed to substitute synthetic fibres based composites due

to several advantages such as renewability, less abrasiveness to equipment, high specific properties, biodegradability and low weight and cost [4]thereby creating a positive environmental impact[5][6]. Recently publish review paper illustrated list of recent work made by the differentresearcher on the kenaf fibre reinforced polymeric (thermoset or thermoplastic resin) composite and also its hybrid composite [1]. Indoor panels and other interior components for high-end cars like BMW and Mercedes Benz and in composite material for the buildings or construction industry[7][8]. In this review paper we try to cover automotive and construction applications of kenaf fibre reinforced polymer composites.

Kenaf fibre Reinforced polymer composites

A study by researchers indicated that enhancement in flexural and impact strength, tensile as well as stiffness of the composites when reinforced with kenaf fibres [9]. In another research it also observed that composite kenaf sheets/poly-L-lactic acid (PLLA) obtained has large mechanical anisotropies such as Young's modulus and tensile strength[10]. In another research work, addition of kenaf fibres into the epoxy, thermal study results showed that the slightly improves both the thermal and charring stability of the samples. Alkalization also causes declination in the properties for the kenaf/epoxy composite [11]. Kenaf bast fiber reinforced poly (vinyl chloride) (PVC)/thermoplastic polyurethane (TPU) poly-blend (PVC/TPU/KF) composites have shown lower tensile strength and strain with increase in fiber content whereas Impact strength and thermal stability was decreasing with increase in fiber content[12].

Integrated industrial applications

The consideration and utilization of kenaf, being the matter of many investigation, study and projects from the late 1950s of the 20th century to explore it as industrial crop. Currently, the industrial use of kenaf fibres endures to expand its application from its ancient and historic appeal as a cordage crop (twine, rope and sackcloth) to variety of novel tenders including absorbents, building materials, paper products, livestock feed and auto industries[3].

Automotive applications

Due to the continuous increase in the cost of transportation, the need to produce lighter vehicles is quite important toward sustaining our current way of life (Fig.1) shows the car body made of kenaf reinforced composite from BMW and Toyota Auto Company. Toyota Boshoku Corporation has been using kenaf for automotive interiors since the mid-1990s. Many studies are also been made on utilizing kenaf fibers polymer composite in automotive industries.Fig.2 shows the 3D CAD model of a commercial passenger vehicle center lever parking brake design from kenaf glass fiber hybridized composite[13]. In the other study hybrid kenaf/glass epoxy composite are utilized in locomotive operational components such as bumper beam by improving the impact property[14]. Bumper system and kenaf bumper shown in Fig.3 (a-b) with its three main elements fascia, energy absorber and bumper beam [15]. Harusmas Agro Sdn. Bhd. (HASB) based in Sabah, Malaysia already initiated a kenaf project back in

the year 2000. In another important study commercialization of door trims using kenaf and petroleum-derived polypropylene (PP) resin are made. Fig.4 shows the door trim using kenaf base material. This combination reduced the weight while retaining the same level of durability as conventional products although achieved by establishing a unique technology to realize a high level of shock resistance and heat resistance. Another research involving the formulation of biodegradable composite using kenaf base material with biodegradable polylactic acid (PLA)resin to developed spare tyre covers and deck board (Fig.5) which is more environmentally friendly.

Source:http://opcionbio.es

Fig.1. Car body of BMW i3and Toyota made from Kenaf reinforced polymer composites.

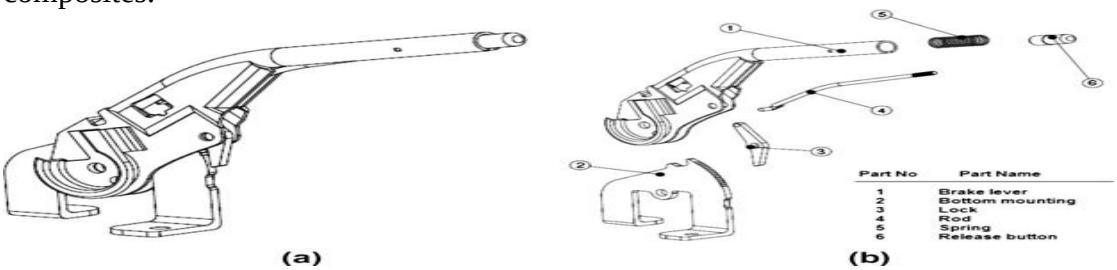

Fig.2. 3D CAD model of a commercial passenger vehicle center lever parking brake design in (a) assembly view, and (b) exploded view[13].

Fig.3. (a) Bumper system components[16]. Fig.3. (b) Kenaf bumper

(http://www.jeccomposites.com/news/composites-news/kenaf-fibre-future-harusmas-experience).

Fig.4. Door trim using kenaf base material

Fig.5.Deck board and spare tyre cover using kenaf (Source: http://www.toyota-boshoku.com/global/about/development/eco/kenaf/)

Construction & Housing applications

Wood and wood-based products can be replaced by kenaf/plastic compounds which are molded into lightweight panels in several applications. This is the first and economically priced plastic lumber for use as constructing materials in housing industry as a engineered materials. Fig.6 (a-b) showing the kenaf panels produced by KEFI Company and the insulated panel building materials made from kenaf (University of North Texas). Moreover, it is also used to make a strong, light weight, cement block with great insulation and effectively fireproof properties. Kenaf core blocks nowadays used to construct multi-story and solitary family homes, deprived of power tools. However, its impact strength is still higher, depicting a great probability of the utilization of kenaf fibre in hybrid natural fibre composites in many of the structural and non-structural components in construction and housing industries[14][9][17].Fig.7 shows the flooring panel and helmet developed from kenaf polymer compositeswhich are widelyused in construction and housing industries nowadays.

Fig.6. (a) Kenaf panels (KEFI Company). Fig.6. (b) Kenaf Insulated panel (Univ. of North Texas).

Fig.7. Flooring panel and helmet from Kenaf fibre reinforced polymer composites (INTROP-UPM).

Conclusions

Kenaf bast fibre has excellent tensile strength combined with superior flexural strength substantiated by several mechanical testing and research work enabling it to utilize in variety of application such as auto-industrial, light weight constructional applications besides customary products like yarns, fabrics, and ropes. Kenaf confirmed to be getting matures only within 5 months, eco-friendly, tolerant of environmental and drought stress and making it a versatile encouraging and economic crop for industrial applications in the upcoming years. Lightweight, insulative, as well as vibrational damping materials, are required in many industrial applications which can be fulfilled by kenaf/ kenaf hybrid thermoplastic or thermoset polymer composite.The commercial and profitable achievement of kenaf crop reflects prospective economic and environmental benefits. Although, kenaf polymer composite has been upgraded in the different sectors such as lethal toxic waste clean-up, removal of oil spills from water, soil remediation and many more.

References

[1] N. Saba, M.Jawaid, K.R.Hakeem, M. T. Paridah, A.Khalina, and O. Y. Alothman, Potential of bioenergy production from industrial kenaf (Hibiscus cannabinus L.) based on Malaysian perspective, *Renewable and Sustainable Energy Reviews* Vol. 42, 2015, pp. 446–459.

[2] M.T. Paridah, A.B. Basher, S. SaifulAzry, and Z. Ahmed, Retting process of some bast plant fibers and its effect on fiber quality: A review, *Bio Resources V*ol. 6, 2011, pp. 5260–5281.

[3] N. Saba, M. T. Paridah, and M. Jawaid, Mechanical Properties of Kenaf Fibre Reinforced Polymer Composite: A Review, *Construction and Building Materials*Vol. 76, 2015, pp. 87–96.

[4] A. N. Frone, S. Berlioz, J. F. Chailan, and D. M. Panaitescu, Morphology and thermal properties of PLA-cellulose nanofibers composites, *Carbohydrate. Polymers,* Vol. 91, 2013, pp. 377–384.

[5] Y. Chen, L. Sun, O. Chiparus, I. Negulescu, V. Yachmenev, and M. Warnock, Kenaf/ramie composite for automotive headliner, *Journal of Polymers and the Environment* Vol. 13, 2005, pp. 107–114.

[6] M. J. John, C. Bellmann, and R. D. Anandjiwala, Kenaf-polypropylene composites: Effect of amphiphilic coupling agent on surface properties of fibres and composites, *Carbohydrate Polymers V*ol. 82, 2010, pp. 549–554.

[7] M. S. Qatu, Application of Kenaf-Based Natural Fiber Composites in the Automotive Industry, *Society of Automotive Engineers Technical Paper*No. 2011-01-0215, 2011.

[8] R. Mahjoub, J. M. Yatim, A. R. Mohd Sam, and S. H. Hashemi, Tensile properties of kenaf fiber due to various conditions of chemical fiber surface modifications, *Construction and Building Materials*Vol. 55, 2014, pp. 103–113.

[9] H. Anuar and A. Zuraida, Improvement in mechanical properties of reinforced thermoplastic elastomer composite with kenaf bast fibre,*Composites Part B: Engineering V*ol. 42, 2011, pp. 462–465.

[10] T. Nishino, K. Hirao, M. Kotera, K. Nakamae, and H. Inagaki, Kenaf reinforced biodegradable composite, *Composites Science and Technology* Vol. 63, 2003, pp. 1281–1286.

[11] Z. N. Azwa andB. F. Yousif, Characteristics of kenaf fibre/epoxy composites subjected to thermal degradation, *Polymer Degradation and Stability***V**ol. 98, 2013, pp. 2752–2759.

[12] Y.A. El-Shekeil, S.M. Sapuan, M. Jawaid, and O.M. Al-Shuja'a,Influence of fiber content on mechanical, morphological and thermal properties of kenaf fibers reinforced poly (vinyl chloride)/thermoplastic polyurethane poly-blend composites, *Materials and Design*Vol. 58, 2014, pp. 130–135.

[13] M. R. Mansor, S. M. Sapuan, E. S. Zainudin, A. A. Nuraini, and A. Hambali, Hybrid natural and glass fibers reinforced polymer composites material selection using Analytical Hierarchy Process for automotive brake lever design, *Materials and Design*Vol. 51, 2013, pp. 484–492.

[14] M. M. Davoodi, S. M. Sapuan, D. Ahmad, A. Ali, A. Khalina, and M. Jonoobi, Mechanical properties of hybrid kenaf/glass reinforced epoxy composite for passenger car bumper beam, *Materials & Design* Vol. 31, 2010, pp. 4927–4932.

[15] S. M. Sapuan, M. A. Maleque, M. Hameedullah, M. N. Suddin, and N. Ismail, A note on the conceptual design of polymeric composite automotive bumper system, *Journal of Materials Processing Technology V*ol. 159, 2005, pp. 145–151.

[16] M. M. Davoodi, S. M. Sapuan, D. Ahmad, A. Aidy, A. Khalina, and M. Jonoobi, Concept selection of car bumper beam with developed hybrid bio-composite material, *Materials & Design* Vol. 32, 2011, pp. 4857–4865.

[17] A. Elsaid, M. Dawood, R. Seracino, and C. Bobko, Mechanical properties of kenaf fiber reinforced concrete, *Construction and Building Materials* Vol. 25, 2011, pp. 1991–2001.

Biocomposite Materials for Design of Prosthetic Leg Socket

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Abstract. In recent year, technological advancement has led to wider range of modern orthopedic and prosthetic device. Nowadays, composites are most widely used for upper- and lower- limb prostheses due to their superior strength and offer excellent biocompatibility. In this review, the use of fibre reinforced composite materials for prostheses are viewed.

Keyword: Composite; Modern orthopedic;Prosthetic devices.

Introduction

The recent technological advancement has led to wider range of application in modern orthopedic medicine and prosthetic devices. The demands on the prosthetics industry are several fold - comfort for the patient, biocompatibility, high performance and versatility to enable patients to lead as normal life as possible. At present, fibre reinforced polymer composites are the most widely used multiphase materials in orthopaedics. In addition, most of today's upper- and lower-limb prostheses are now made from composites with underlying polymer matrix. Due to their exceptional strength to weight characteristics, these types of materials are favorable as well as their superior biocompatibility [1].

Prosthesis is an artificial limb that replaced a missing part of body, such as the arm and leg [2]. The type of artificial limb used is determined largely by the extent of the amputation or loss and the location of the missing limb. Artificial limbs may be needed for a variety of reasons, including diseases, accidents, and congenital defects [3]. A congenital defect can create the need for an artificial limb when a person is born with a missing or damaged limb. Cancer, infection and circulatory diseases are the leading ailments that may lead to amputation. Moreover, industrial, vehicular and war related accidents are the leading causes of amputation in many developing countries, such as Africa. On the other hand, in most developed countries, such as the North America and Europe, diseases are the leading cause of amputation. Therefore, at present, the demand for prosthetic legs is high for many amputees around the world.

The advancement of design and manufacturing in the field of prosthetics has been notable due to the common demands from either the war victims in war-hit countries or those who are handicapped from birth [4]. Thus, as the human body changes over time due to growth or change in body weight, the artificial limbs have to

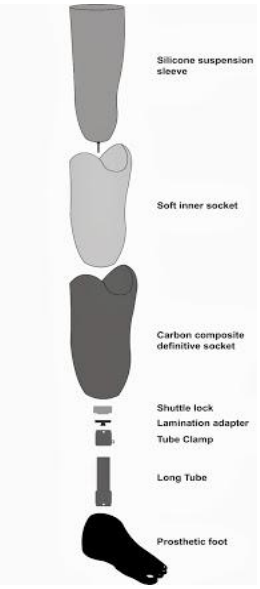
be replaced or adjusted periodically [5]. This constant need to change may become costly if the material used is expensive especially with regard to the production cost of the parts and components of the prosthetic legs.

Limb prosthesis characteristically has three main parts which the interface, the components, and the cover. As for the prosthetic legs, they consist of a socket, a pylon and a foot which make up the main parts. All these parts have different requirement and standards based on their usage such as strength, flexibility and aesthetic. Therefore, they are made of different materials - either synthetic or bio-based materials. However, none of the materials has used bio-based materials to produce the sockets of prosthetic legs at present [6]. Within this review, the use of fibre reinforced polymer composite in prosthetic devices is discussed. With particular emphasizes on sports and commercial prosthetic, the recent impact of composites on the design of upper- and lower- limb prostheses are reviewed. The use of natural composites is briefly together with their potential application.

Materials and Manufacturing of Prosthetic Leg Sockets

The prosthetic limbs are first prescribed by a medical doctor in conjunction with the prosthetist and the physical therapist's advice, similar with the way dentures or eyeglasses are recommended. Prosthetic sockets were fabricated from materials such as leather, wood, latex and metal before the introduction of today's advanced resins thermoplastics and composites. For centuries, certain types of wood or leather were carved, soaked, stretched and stitched into prosthetic forms. Once dried, sealed or lacquered, they proved to be very durable. Early leather sockets were often suspended in a structural metal or wood frame. Certain parts of the limb (e.g. the foot of the prosthesis) had traditionally been made of wood and rubber. Even at present, the foot of the prosthesis is made from urethane foam with a wooden inner keel construction, but the use of leather or wood, has been replaced by polypropylene-based materials (e.g. polyethylene, polypropylene, acrylics, and polyurethane) due to uneconomical and hazardous effects on the environment [7].

Prosthetic leg socket is upper most part of a prosthesis that makes contact with the residual limb and transfers the forces of walking to the ground. To produce a socket, the prosthetist has to capture the three dimensional (3D) shape of the residual limb by wrapping a cast around it while the residual limb is either loaded or unloaded, depending on the preference of the prosthetist. A positive mold of the residual limb is then acquired from the negative mold. The anatomical points of interest are identified on the positive mold and extra material is either added to relieve pressure at sensitive regions or removed to increase pressure at specific load-bearing locations [8]. The type of material used for construction is main important part to consider in the designing and fabrication of limb prosthetic. The comfort of the socket will be influenced by interface materials. The strength and weight will be affect structural materials of the overall prosthesis [9].



Trans-Tibial(below knee) prosthesis with components part [10].

In general, the technique of fabricate the sockets of prosthetic legs is initiated by constructing the positive cast of patient's residual limbs. This can be done using computer aided design and manufacturing (CAD/CAM) equipment and software, or manually by filling the negative impression of the amputee's residual limbs with a plaster mixture of Paris and other materials [11]. The socket fabrication process using lamination or sandwich layering technique basically conducted by sandwiching several layersof selected materials over a cast and between two layers of polyvinyl alcohol(PVA), polyvinyl chloride (PVC) or optional advanced materials. Then, theselected resin is injected into the material using a vacuum assistance. To avoid trapped moisture, the cast is ensured to be smooth with no sharp edges and dried or sealed. Fabrication process will be improved if readily available industry-standard acrylic resins are used [7]. Epoxy resins are commonly used with advanced laminating materials, such as carbon fibres, hybrid matt and stockinette, due to their mechanical strength in holding the number of layers together [12]. To ensure the capability of resin's optimum strength, the fibre orientation and the manufacturer's resin to matrix are the most important aspects to be considered in advanced socket fabrication, especially if it is made of a composite material. Lamination method of socket fabrication is suitable for natural-based biocomposites, since the proposed materials can be woven into a layer form [13]. The used of fiberglass, or in woven form it is called as glass fibre stockinette (tubular glass cloth), in this layers gives the most strength to the laminated socket. Therefore, it is proven that this particular layer is potentially to be replaced with the natural fibre materials. The twist to this exploratory experimentation is to replace the common synthetic fibres with natural fibre in the existing laminated material structure. Indirectly, this method provides a platform for proposing alternative materials which possess the same or better quality in order to lessen the cost of material while improving the strength of the socket.

The other technique for sockets fabrication which involves the vacuum casting technique is called seam draping socket [7]. This particular technique engages these steps: a) creating a positive cast of a patient's residual limb, b) positioning mechanical press on a distal end of positive cast, c) moulding a prosthetic socket component over the positive cast and mechanical press, and d) activating the mechanical press such that the prosthetic socket is pushed apart from the distal end of the positive cast [11]. Accordingly, a sheet of copolymer thermoplastic is first heated in a large oven and then vacuum starts to form around the positive mould. In this thermoplastic-heating process, the heated sheet is simply laid over the top of the mould in a vacuum chamber, and if necessary, the sheet is re-heated to ensure zero air tolerance space. This step requires that the air in between the sheet and the mould is totally sucked out of the chamber as well as collapsing the sheet around the mould and forcing it into the exact shape of the mould [11]. The type of material available for this thermoplastic-heating technique includes a clear thermoplastic material, a polypropylene polymer material, and a flexible thermoplastic material [14].Thissecond method of socket fabrication involves higher technology since it usesa special machine to soften the very high melting point of material used.

Natural Based Materials for Manufacturing of Prosthesis Leg Socket

The literature hasfound that natural fibres, such as kenaf and corn starch, can be reinforced withplastic in many other applications.Natural fiber is a material that can be recycled, more environmentally friendly, potentially abundant, and less costly than synthetic fibers and green composites. As such, it can support the concept of go green and back to nature [15]. The use of natural fiber reinforced composites (NFRCs) in orthopedic and prosthetic (O&P) would result in lighter weight, cost effective, sustainable, and widely available materials for use in these critical biomedical devices [16].

The lower manufacturing cost of prosthetic devices may permit the less fortunate and financially disabled wearers in most developing and Third World countries the chance to get affordable prosthetic legs. For this to happen, the cost of production should be lessened [17]. There are many studies carried out throughout the world to help building cheap yet eco-friendly prostheses. For instance, based on previous studies shown that kenaf can be processed into yarn and converted into composites of high impact strength to replace the fibre-glass as one of the layer in prosthetic socket fabrication [18]. Similar results studies have showed that ramie composite material have the potential to be further developed as an alternative material of socket prosthesis substitute socket prosthesis with fiberglass polyester composites (FGP), which are locally available, bio mechanically appropriate, as lightweight as possible, comfort and psychosocially acceptable [15]. Bamboo fibre reinforced composite can be replace the cotton and nylon composite that are currently used in O&P due to their superior strength and ductility [16].

Those successful findings suggest that the natural based materials applied in prosthetic applications could have potential to serve as a cost effective, practical, and environmentally sustainable material choice in O&P. These findings have inspired the present study to probably produce low-cost manufacturing of prostheses made of the

proposed composite. Additionally, it may prove that some components of prostheses can be locally produced, and more importantly, the quality of products is maintained. This can cut the cost of production and lessen the number of imported parts. At the same time, it is expected to contribute to the poor countries or poor people who cannot afford to have expensive artificial limbs.

Conclusion

The analysis of this literature review is to find out the suitable material in prostheses. Composites are now widely used in modern prosthesis device. Crucially, many advances have been seen in dentistry and the design of lower limb sports prostheses. The advantages of using fibre-reinforced composites for orthopedics purposes are associated with their exceptional specific strength characteristic and biocompatibility. The use of natural fibre-based biocomposite in prosthetic device will reduce the manufacturing cost in term of material costing and also to provide an eco-friendly, and yet maintainingthe features required for artificial limbs. Future generations of prosthetic devices are aiming to provide enhanced for upper- and lower- prosthetic in term of material and design, thus led the patient to have a good quality of life.

References

[1] M.-S. Scholz, J.P. Blanchfield, L.D. Bloom, B.H. Coburn, M. Elkington, J.D. Fuller, M.E. Gilbert, S.A. Muflahi, M.F. Pernice, S.I. Rae, J.A. Trevarthen, S.C. White, P.M. Weaver, I.P. Bond (2011). The use of composite materials in modern orthopaedic medicine and prosthetic devices: A review, *Compos Sci Technol* , 71, pp.1791 1803.

[2] Klodd, E., Hansen, A., Fatone, S., & Edwards, M. (2010). Effects of prosthetic foot forefoot flexibility on oxygen cost and subjective preference rankings of unilateral transtibial prosthesis users. Journal of Rehabilitation Research and Development, 47(6), 543-552.

[3] Horne, C.E., & Neil, J.A. (2009). Quality of life in patients with prosthetic legs: A comparison study. Journal of Prosthetics and Orthotics, 21(3), 154-159.

[4] Ramachandran, A., Lakshmi, S., Arun, N., Samith Shetty, A., & Snehalatha, C. (2010). Role of industries in the care of diabetic foot. International Journal of Lower Extremity Wounds, 9(3), 116-121.

[5] Kobayashi, L., Inaba, K., Barmparas, G., Criscuoli, M., Lustenberger, T., Talving, P., Lam, L., & Demetriades, D. (2011). Traumatic limb amputations at a level I trauma center. European Journal of Trauma and Emergency Surgery, 37(1), 67-72.

[6] Rayegani, S.M., Aryanmehr, A., Soroosh, M.R., & Baghbani, M. (2010). Phantom pain, phantom sensation, and spine pain in bilateral lower limb amputees: Results of a national survey of Iraq-Iran war victims' health status. Journal of Prosthetics and Orthotics, 22(3), 162-165.

[7] Myradal, P.J. (2009). Transtibial diagnostic prosthesis fabrication: Part one and two. O&P Business News. Retrieved from http://www.oandpbusinessnews.com

[8] Muller M, Staats TB, Leach M, Fothergill I. Total surface bearing transtibial socket design impression techniques. J Proc 2007; retrieved at http://www.oandp.org/publications/jop/2007/2007-49.asp

[9] Jack E. Uellendahl (1998). Prosthetic Primer: Materials Used in Prosthetics Part I & II. A Publication of the Amputee Coalition of America. Volume 8. Issue5.

[10] Figure of Trans-Tibial (below knee) prosthesis with components part, retrieved from http://www.propaedics.co.za/Prosthetics

[11] Faustini, M.C., Neptune, R.R., Crawford, R.H., Rogers, W.E., & Bosker, G. (2006). An experimental and theoretical framework for manufacturing prosthetic sockets for transtibial amputees. IEEE Transactions on Neural Systems and Rehabilitation Engineering, 14(3), 304-310.

[12] Rogers, M.S.B., Gordon, B., Mario, F., Richard, R.N., & Richard, C. (2007). Case report: Variably compliant transtibial prosthetic socket fabricated using solid freeform fabrication. Retrieved from http://www.me.utexas.edu/~neptune/Papers/jpo20(1).pdf.

[13] Rosalam Che Me, Rahinah Ibrahim, and Paridah Md. Tahir. Natural based biocomposite material for prosthetic socket fabrication. Universiti Putra Malaysia, 2012.

[14] Mak A.F.-T., Zhang, M., & Leung A.K.-L. (2007) Artificial Limbs. Comprehensive Structural Integrity, Chapter 9.08, pp. 329-363.

[15] A.P. Irawana, T.P. Soemardib, K. Widjajalaksmic and A.H.S. Reksoprodjob. Tensile And Flexural Strength Of Ramie Fiber Reinforced Epoxy Composites For Socket Prosthesis Application. International Journal of Mechanical and Materials Engineering (IJMME), Vol.6 (2011), No.1, 46-50.

[16] William Slocumb. Bamboo Fiber Reinforced Composites for Prosthetics and Orthotics. Department of Biomedical, Chemical, and Materials Engineering. 2014. Retrieved from http://www20.csueastbay.edu/programs/csr/files/docs/2014%20CSU%20Award%20Winner%20Summaries/WilliamSlocumb-2014%20Summary.pdf.

[17] Jensen, J. S., & Raab, W. (2006). Clinical field testing of vulcanized Jaipur rubber feet for trans-tibial amputees in low-income countries. Prosthetics and Orthotics International, 30(3), 225-236.

[18] Rosalam Che Me. Design and application of kenaf-based polymer composite in prosthetic leg socket fabrication. Universiti Putra Malaysia. 2012. pp 91.

Sub-Critical Water's Application in Oil Extraction From Oil Palm Empty Fruit Bunch (OPEFB)

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Abstract. Sub-critical water (sub-cw) was used to extract oil and other valuable product from spikelets of Oil Palm Empty Fruit Bunch (OPEFB). The spikelets was treated with sub-cw between 180-280°C and a reaction time of 2 and 5 minutes. The yield of oil extracted was strongly affected by the reaction temperature and time. Sufficient reaction time allows a complete reaction to occur. Highest yield of oil was obtained at 240°C and reaction time of 5 minutes, which was recorded at 0.0747g-oil/g-dry EFB. The oil obtained through this method was 84.4% from one obtained using soxhlet extraction. It is expected that the yield will continue to increase as the reaction time increases.

Keywords: Sub-critical water, oil palm empty fruit bunch, soxhlet

Introduction

In Malaysia, the average yield of fresh fruit bunch (FFB) is 20 tonnes/ha. For every ton of processed FFB, 23% are the OPEFB [1]. The whole OPEFB is considered as waste. This means around 4.6 ton OPEFB are produced for every hectare. In the year 2011 itself, Malaysia had a total of 4.917 million hectares of oil palm plantation [2] which means 22.6 million tons of OPEFB are produced in that year only. OPEFB is known as lignocellulosic waste produced during oil extraction in palm oil mill [3] and is available as a substrate in cellulose production [1].

Besides that, based on Hur Far Engineering Work Sdn. Bhd. under MPOB, the composition of OPEFB comprised of 70-75% moisture, 23-25% fibre and 3.5-4% oil (based on wet basis). Thus in total fibre present, cellulose makes up 30-40% of the OPEFB which is the highest portion followed by hemicellulose and lignin [4]. Eventhough the amount of oil in every tonne of OPEFB is approximately 4% on wet basis, it can be very significant if the oil can be extracted out from tonnes of OPEFB produced.

Theoretically, sub-cw is defined as liquid water that lies at temperature between its atmospheric boiling point and less than its critical point (T= 374°C and P = 21.8MPa) [5]. For sub-cw treatment, it is known that pressure have not much effect on the reaction [6]. Water can be used effectively as solvent due to alteration of their physico-chemical properties at higher temperature and pressure. However,

temperature plays an important role in the extraction efficiency. High temperature and pressure induce the nonpolar solvent behavior of water, thus making organic compound miscible in it. This sub-cw are said to give better yield than other conventional method like soxhlet extraction [7]. The two physico-chemical properties of water which are affected at higher temperature are the dielectric constant and ion production constant of water.

Water at room temperature has a very high dielectric constant of 80. Interestingly, the dielectric constant of water becomes 29 at 250^0C close to dielectric constant of ethanol at room temperature ($\varepsilon = 27$) **[6]**. It is known that ethanol is one of a good solvent for extraction, thus by having property similar to ethanol, it enables water to behave like ethanol. Owing to the excellent physic-chemical characteristics of sub-cw, it is expected that residual oil can be extracted from OPEFB, thus increase the extraction efficiency in mill operation.

Materials and methods

Materials

Chemicals used for molten salt bath were sodium nitrite (industrial grade) and potassium nitrate (industrial grade) with ratio of 1:1. Besides that, phenol (crystal), hexane, acetone (R&M Chemicals) and sulphuric acid, H_2SO_4(98%) (Merck, Germany) were also used in this research. Distilled and deionized water were prepared in the process laboratory, Faculty of Engineering UPM.

Methods

Preparation of the sample and moisture analysis

Oil Palm Empty Fruit Bunch (OPEFB) were obtained from Sri Langat Mill in Dengkil. The sample obtained was a fresh wet EFB which has not been pressed. The spikelets of the OPEFB were then cut from the stalk and were cut into small pieces of about 4-8mm long. Three of 5g from cut samples were prepared in a clean beaker which has been weighed. All the beaker containing sample were then put into dryer at 80°C to remove moisture from sample. The beaker were weighed every 24hours until it reach a constant weight.

Sub-critical water reaction

A batch laboratory-scale sub-critical fluid extraction system was used for this study. The reactor used was the stainless steel batch reactor (SUS316, i.d 16.5 mm x 150.4 mm) capped with Swagelok fitting. The reactor was rinsed three times with tap water and lastly using the distilled water. Then, the reactor was dried, and weighed before filled in with sample. Molten Salt bath (Celcius 600H, Tomasu Kagaku) was preheated up to a temperature between 180°C to 280°C for a few minutes. 1g of cut EFB were inserted into the stainless steel tube batch reactor using spatula and later filled with 5g distilled water. Then air was drawned out by purging argon gas before weighed. After the reactor cap was tighten, it was weighed again to obtain the actual amount of water inserted. The reactor was then placed slowly into the preheated salt bath, shaked for few minutes (reaction time of 2 and 5 minutes). The pressure of the

reaction which assumed to be constant during the reaction is measured using the steam table. After reaching the reaction time, reactor was quickly quenched in flowing tap water at room temperature. Reactor was quenched to terminate the reaction. The reactor content was transferred into test tube and photos were taken. Samples in test tube were centrifuged to obtain few phases which were the oil, aqueous and solid phases.

Recovery of oil extracted by sub-cw

The significant layer of oil on top of water was recovered using hexane solvent for a few times until it appear colourless. The hexane containing oil was then filtered and dried in fume hood for about 2 days. Weight of oil remained were recorded. The oil was then diluted back with hexane for analysis purpose. Yield of oil was defined as in eq. (1):

$$Yield\ of\ oil = \frac{W_{oil}}{W_D\ EFB} \qquad (1)$$

Where W_{oil} refer to weight of oil and W_D EFB refers to dry weight of EFB used which refers to EFB's wet weight multiplied by (1-Moisture Content).

Soxhlet extraction

This conventional method using hexane solvent to extract oil from OPEFB was used as benchmark for this research. Approximately 14g of cut spikelets were inserted into thimble and underwent extraction for about 6 hours. The hexane containing oil were then filtered to remove impurity and dust, and let to dry in the fume hood. The yields of oil obtained from soxhlet extraction were calculated as in eq. (1).

Results and Discussion

Moisture Content

The samples were dried in dryer at 80°C for several days until the weight obtained was constant. 80°C were chosen because lower temperature than that will require longer time for the weight of sample to become constant. The trend of the average moisture content reduction was shown in Figure 1.

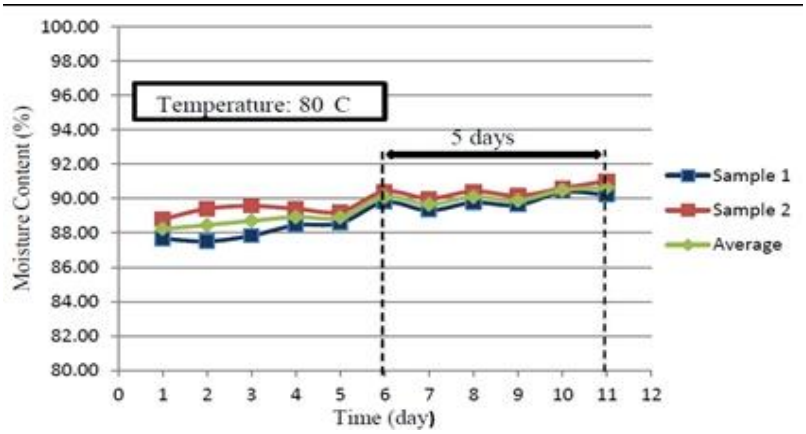

Figure 1: Time course of moisture content of OPEFB during drying process

Figure 1 clearly shows the moisture content was within 90.10-90.60% for wet basis. However, the moisture content of the dry basis was approximately 47.07% which covers almost half of the OPEFB weight. However, different OPEFB may have different moisture content. For example, Hur Far Eng. reported that the moisture content was 75% (wet basis). The moisture content (dry basis) obtained was used to calculate the dry weight of the sample.

Extraction of oil by sub-critical water

The reactions were carried out at temperature range from 180°C to 280°C with interval of 5°C, and 20°C and reaction time of 2 and 5 minutes. Each reaction temperature and time gives different results as shown in Figure 2. The yellow color of the oil in hexane also can be seen clearly as the temperature increases up to 240°C.

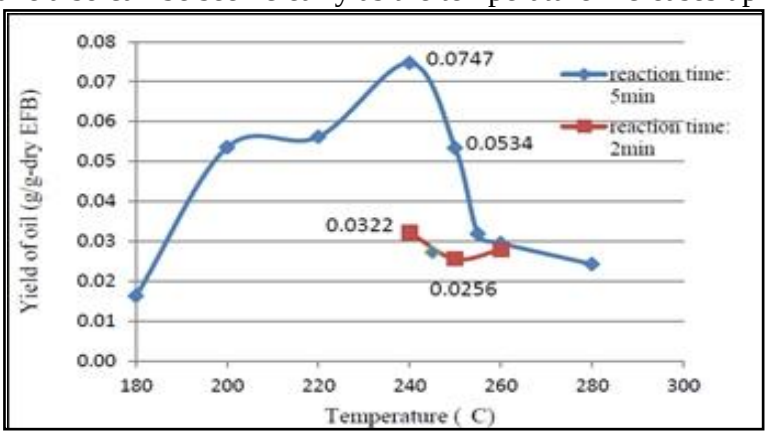

Figure 2: Effect of reaction temperature and time on the yield of oil extracted.

Figure 2 shows that the yield of oil was higher when reaction time by sub-cw was 5 minutes compared to 2 minutes at every temperature. Significant peak was observed at 240°C for reaction time 5minutes, while no peak can be seen when reaction time was 2 minutes. The highest yield of oil was observed at 240°C (0.0747 g-oil/g-dry EFB). It is because at this temperature, the dielectric constant of water reduces until near to

ethanol (at room temperature), makes it a good solvent. Thus, extraction power of oil was higher at 240°C than at other lower and higher temperature. Figure 3 shows the comparison for yield of oil at different reaction time and temperature.

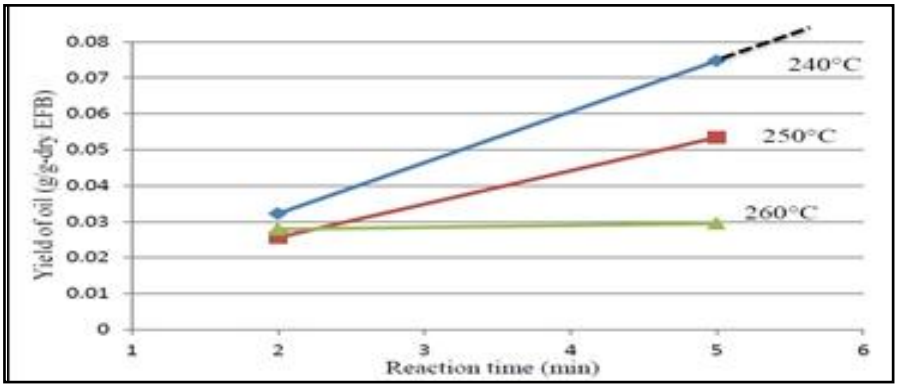

Figure 3: Effect of reaction temperature and time on the yield of oil extracted

From figure 3, all reactions were increasing when reaction time was increased from 2 to 5 minutes. Very steep increase was observed at 240°C reaction. Since no peak was observed at any of the reaction temperature and time, it is believed that the yield of oil will increase if the reaction time for 240°C reaction is more than 5 minutes. The yield of oil obtained by sub-cw was compared to 0.0885g-oil/g-dry EFB, one obtained through soxhlet extraction. Even though yield of oil obtained through soxhlet; 8.85% was higher than one obtained through sub-cw at 240°C, the difference was not too significant. It is expected that longer reaction time may results in higher yield of oil near to one obtained by soxhlet. However, different OPEFB used may give different result due to its quality. For example, the oil extraction rate (OER) from the fresh fruit bunch were affected by several factors such as ripeness, age of palm tree, type of soil and others [8]. Furthermore, [9] reported that yield of palm oil extracted from OPEFB using soxhlet extractor was 8% which was slightly lower than one obtained through this research by 0.85%.

Conclusions

The highest yield of extracted oil was obtained at 240°C which is 0.0747 g/g-dry EFB. The yield of oil obtained by sub-cw at this temperature was almost close to yield of oil extracted by conventional method, soxhlet extractor. This shows that the sub-cw was able to achieve more than 90% extraction of oil if the optimum reaction temperature and time are known. Our study shows that the sub-cw is an alternative solvent to other organic solvent commonly used in extraction. It is better than hexane as hexane is carcinogenic and hazardous to health. Meanwhile, other solvent like ethanol is quite expensive compared to water since water is easily obtained.

Acknowledgement

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References

[1] I. A. Udoetok, Characterization of Ash made from Oil Palm Empty Fruit Bunches (oefb) (2012). *International Journal of Environmental Sciences, 3*(1). doi: 10.6088/ijes.2025030131033

[2] Malaysian palm oil industry. (2012) *Malaysian Palm Oil Council*. Assessed on 20 April 2014. Available at: http://www.mpoc.org.my/Malaysian_Palm_Oil_Industry.aspx

[3] B. Kavitha, P. Jothimani and G. Rajannan. (2013). Empty Fruit Bunch- A Potential Organic Manure for Agriculture. *International Journal of Science, Environment and Technology. 2* (5)

[4] D. Zhang, Y. L. Ong, Z. Li, and J. C. Wu. (2012). Optimization of Dilute Acid-Catalyzed Hydrolysis of Oil Palm Empty Fruit Bunch for High Yield Production of Xylose. *Chemical Engineering Journal. 181-182.*

[5] Subcritical water. (2009). *Compressed Fluid Technologies.* Assessed on 2 October 2013. Available at: http://www.greenfluids.org/html/Techniques/hot_water.html

[6] P. Omid. (2010) Production of Valuable Materials from Rice Bran Biomass using Subcritical Water. *Doctoral Thesis at Osaka Prefecture University.*

[7] J. Kronholm, K. Hartonen, M. L. Riekkola. (2007). Analytical extractions with Water at Elevated Temperatures and Pressure. *Trends in Analytical Chemistry, 26*(5).

[8] L. Y. M. Elaine and B. Adam. (2010). A Qualitative Approach of Identifying Major Cost Influencing Factors in Palm Oil Mills and the Relations towards Production Cost of Crude Palm Oil. *American Journal of Applied Sciences. 8* (5).

[9] A. A. Salema, and F. N. Ani. (2012), Pyrolysis of Oil Palm Empty Fruit Bunch Biomass Pellets using Multimode Microwave Irradiation. *Bioresource Technology 125*, 102-107.

Mechanical Properties of Palm Composites:Date, Bertam, Coconut and Oil Palm –A Review

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Abstract. Nowadays, natural fiber is an interesting option and it is the most widely applied fiber in composite technology. This research paper aims to enlighten the existing properties in natural fiber composites especially the same species; palms. Additionally, this research paper reviews the related papers from the year 2006 until 2013. Finally, a summary of mechanical properties that have been identified for treated and untreated fibers.

Keywords: Date, Bertam, Oil palm, Composites, Mechanical properties, Strength, Impact.

Introduction

The natural fiber can be dividedinto three types which are vegetables, animals, and minerals. Several plants from which fibers can be sourced are hemp (*Cannabis sativa*), coconut (*Cocosnucifera*), flax (*Linumusitatissimum*), sisal (*Agave sisalana*), and jute (*Corhoruscapsularis*). Animal fibers are natural fibers that consist largely of particular proteins. Instances are silk, hair including wool, and feathers. The example of mineral fiber is asbestos which is used in insulating houses. However, in the automotive industry, the vegetable category is the most popular. This category canbe divided into three groups;

a) Fruit fibers are extracted from the fruits of the plant, they are light and hairy, and allow the wind to carry the seeds such as cotton and coir.
b) Bast fibers are found in the stems of the plants. Usually they run across the entire length of the stem and are therefore very long. Some examples of bast fibers are flax, kenaf and hemp.
c) Leaf fiber is rough and sturdy and form part of the plant's transportation system, they are also called leaf fibers [1]. Sisal and bertam are the example of these groups.

The properties of natural fibers are affected by some factors such as type of fibers, moisture content and form of fibers. Additionally, natural fibers are affected regarding to growth species, crop cultivation, location and local ultimate, harvest, supply (transportation supply, age of fiber) and retting or extracting process [2,3]. Eugeissonatristis known as bertam is one of the plant in the group which is have many benefits in medical and housings sector [4-7]. Usually, it has 6-10 m tall large

ascending leaves with a green leaflet and spiny leafstalks. In Malaysia, the fronds of bertam usually use as wall of chalets or resorts. Moreover, consuming it fruits can relieve one from fever; the sap from the stem is used for insect sting. The leaves are used as thatch and the leafrachis are used for fish traps and fishing poles [6]. Other example plants are of the same species such as oil, coconut and date palm.

Figure 1: Bertam plant (a), and Bertam leaves (b)

Methods

The method section describes in detail how the study was conducted. Contents of articles published on the international journals discussing about the oil, coconut, date and Bertam composites were identified. The authors have designed a structured process for selection appropriate articles. The research of the articles was carried out using online databases and library services. This was done by reading the articles and reflecting upon their appropriateness for the topic studied. The information such as palm species, part of fiber, and properties were identified, and then discuss the findings and finally draw conclusions.

Analysis and discussions

The analysis regarding to some papers related with the palms fibers. The table 1 shows the mechanical properties of bertam, oil palm, date, and coconut palm. Bertam have tensile, flexural and impact strength for untreated fiber composites with polyester. Bertam composites, coconut and oil palm have used the untreated fiber to examine the properties.

Conclusions

As conclusion, date fiber has tensile and flexural modulus highest than other palms. But, in this case, date fibers have done treatments. For untreated fiber, the oil palm bunch has the highest value of tensile modulus and bertam has the highest value of

flexural modulus and impact strength. The fiber treatment has improved the mechanical properties of fiber composites.

Table 1: Tensile modulus, flexural modulus and impact strength for palms.

No	Composites	Part of fiber	Treated/ untreated	Mechanical properties			References
				Tensile Modulus (MPa)	Flexural Modulus (MPa)	Impact Strength (kJ/m^2)	
1	Bertam	Leaves	Untreated	204	1827	12.29	[5][6][7]
2	Date	Trunk	Treated	1500	1590	-	[8]
		Rachis	Treated	1090	1590	-	[8]
		Petiole	Treated	1050	1610	-	[8]
		Pits	Treated	319	398	-	[14]
3	Coconut	Coir	Treated	24.8	-	-	[3]
		Fruit (shell)		-	-	4.76	[15]
		Fruit	Untreated	250	-	-	[17]
4	Oil palm	fruit	treated	-	-	4.6	[15]
		bunch	untreated	1430	3300	-	[16]

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References

[1] V.Vilay,M.Mariatti, R. Mat Taib, and M.Todo,Effect of fiber surface treatment and fiber loading on the properties of bagasse fiber-reinforced unsaturated polyester composites. Journal of Composites Science and Technology, Vol.68, 2008, pp. 631-638.

[2] P.M. Tahir, A.B.Ahmed, S.O.A.SaifulAzry and Z.Ahmed, Retting process of some bast plant fibers and its effect on fiber quality: a review. Peer-Reviewed Article Vol. 6(4), 2011, pp. 5260-5281

[3] M.M. Kabir, H. Wang, K.T.Lau and F. Cardona, Chemical treatments on plant-based natural fiber reinforced polymer composites: an overview, Journal of Composites: Part B. Vol.43, 2012, pp. 2883-2892.

[4] A.M.Syafinieand A.N. Ainuddin, Biomass and carbon estimation of Eugeissonatritis. SainsMalaysiana. Vol.42(10), 2013,pp. 1461-1466.

[5] M.Asyraf, and Z.Rahmad, 2012. The distribution of palms and pandans in TelukBahang permanent forest reserve, Penang. Pertanika Journal of Tropical Agricultural Science. Vol.35(1), pp 85-91.
[6] B.J. Chee, Medicinal properties and common usages of some palm species in the KampungPeta community of Endau-Rompin National Park, Johor. Journal of Tropical Medicinal Plants. Vol.6(1), 2005, pp. 79-83.
[7] H.D.Rozman, M.J.Saad, and Z.A. MohdIshak, Flexural and impact properties of oil palm empty fruit bunch (EFB)-polypropylene composites- the effect of maleic anhydride chemical modification of EFB. Journal of Polymer Testing, Vol.22, 2001, pp. 335-341.
[8] I.Zaman, A.E. Ismail and M.K. Awang,Influence of fiber volume fraction on the tensile properties and dynamic characteristics of coconut fiber reinforced composite, Journal of Science and Technology Vol. 1(1), 2011, pp. 55-72.
[9] M.H.Norhidayah, A. Hambali and Y. MohdYuhazri, A review of current development in natural fiber composites in automotive applications, Applied Mechanics and Materials Vol. 564(2014), pp. 3-7.
[10] M.H. Norhidayah, A. Hambali and Y. MohdYuhazri, Impact Behaviour of the Bertam Fibers Reinforced with Unsaturated Polyester,Advances in Environmental Biology, Vol. 8(8) 2014, Pages: 2753-2755
[11] M.H. Norhidayah, A. Hambali and M.Y.Yuhazri, Study the effect of particle size on the water absorption behavior and density of Polyester/ Bertam fiber composites, Australian Journal of Basic and Applied Sciences, 8(15) Special 2014, Pages: 374-376
[12] M.H. Norhidayah, A. Hambali and M.Y. Yuhazri, The effect of fiber size on the mechanical properties of bertam/unsaturated polyester composites, Proceedings of the International Conference on Design and Concurrent. Melaka, Malaysia, 22-23 September 2014, pp. 1-5. (paper in proceedings)
[13] S. Mahdari, H. Kermanian and A. Varshoei, Comparison of mechanical properties of date palm fiber-polyethylene composite, BioResources Vol. 5(4), 2011, pp. 2391-2403.
[14] A. Ghazanfari, S. Emami, S.Panigrahi and L.G. Tabil, Thermal and mechanical properties of blends and composites from HDPE and date pits particles, Journal of Composite Materials, Vol.42(1), 2008, pp. 77-89.
[15] S. I. Durowaye, G. I. Lawal, M. A. Akande, and V. O. Durowaye, Mechanical properties of particulate coconut shell and palm fruit polyester composites, International Journal of Materials Engineering, Vol. 4(4), 2014, pp. 141-147.
[16] M.Y.MohdZuhri, S. MohdSapuan, I. Napsiah, and W. Riza, Mechanical properties of short random oil palm fiber reinforced epoxy composites,Journal of Malaysian Science, Vol.39(1), 2010, pp. 87-92
[17] R. Kannan, C. MohdAs'ri, Y. MohdYuhazri and HaeryipSihombing, Effect on mechanical properties of hybrid blended coconut coir/ paraffin waz/ LDPE. Journal of Integrated Engineering Vol.3(2), 2011, pp. 63-67.

Experimental investigation of Lateral crushing on supporting and non-supporting plate composite hexagonal tubes

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Abstract. In this work, both supporting and non-supporting plates of natural non-woven kenaf reinforced epoxy composite hexagonal tubes were fabricated by a hand lay-up method. Lateral crushing tests were carried out under quasi-static compression load using the universal testing machine (UTM) type (INSTRON MTS 3382) with a capacity load of 100 kN and with constant speed of 15 mm/min. Results showed that the supporting tubes have better specific energy absorption (SAE) for all tested specimens, and exhibited better crashworthiness approximately 60% compared to the non-supporting ones. . Results also demonstrated that all specimens have been failed with longitudinal failure mode.

Keywords: Supporting plate, Specific Energy absorption, Kenaf fibre

Introduction and previous studies

Natural fibers have been gradually used in the automobile manufacturing sectors for several reasons like the weight saving, low density, corrosion resistance, availability, being renewable, environmentally friendly and ease of handling. Several studies have investigated the effect of using natural fibers on the structural crashworthiness. Alkbir et al [1] had experimentally studied the effect of geometry on the crashworthiness parameters of natural kenaf fiber reinforced composite hexangular tubes, they found that the composite tube with $\beta = 60°$ exhibited the highest specific energy absorption capability.Bartosz et al. [2], on their part, reported that four main fracture modes under compressive loading were observed for hemp yarn reinforced cylinder composite tubes, namely micro buckling, diamond shape buckling, concertina shape buckling, and progressive crushing. Libo yan et al. [3], investigated empty and polyurethane-foam filled natural flax /epoxy tubes under quasi-static lateral crushing. They reported that the foam filled tubes with more fabric layers displayed better crashworthiness compared to the empty tubes.This work considers the behavior of

non-woven kenaf fiber reinforced hexagonal composite tubes with a supporting and non-supporting hexagonal tubes subjected to quasi-static lateral loading.

Material and experimental procedure

The non-woven kenaf fiber and epoxy resin materials were used to fabricate the composite hexagonal tubes and the supporting plates inside each tube of the composite tubes' walls was fabricated with a thickness of 4 mm of NKF/epoxy. Samples of specimens and fabrication process are shown in Figure 1.

Figure 1. Fabrication process

Results and discussion

Load- displacement response

In the current study, kenaf/epoxy composite tubes with supporting plate and different hexagonal side angles were fabricated. The lateral load–displacement curves of the hexagonal tubes are demonstrated in Figure 2. Commonly, the tubes behaved approximately elastically till the first peak load, then immediately the load started to drop while the tubescorresponding to the initiation of collapsing.Three regions can be seen from the load- displacement curves. The first one is the pre- crushing stage. The second region represents the stable- crushing stage, whereas the third one is the post-crushing stage. The load carrying capacity missed its resistance after the first crash, then tubes start to resist as a shock absorbers. At the crushing stage, the disparity in curves denoted that the changing hexagonal side angles have an effect on the load carrying capacity. However, it can be mentioned that at the initial crushing stage, the highest crushing load value was recorded due to the supporting plates inside the hexagonal tubes.

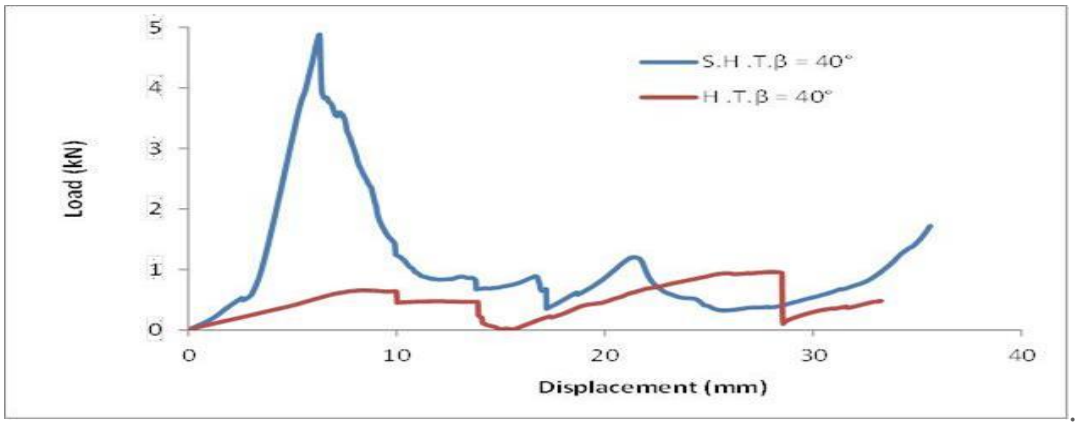

Figure 2. Load–displacement curve for hexagonal composite tubes with supporting and non-supporting plate

Table 1. Energy absorption capability for hexagonal composite tubes

Type of material	Type of ship	Type of test	SAE (J/kg)
Non-woven Kenaf /epoxy	Non supporting Hexagonal	Lateral test	68.362 to 95.59
Non-woven kenaf/epoxy	Supporting Hexagonal	Lateral test	163.71 to 241.6

Failure modes

The failure mode is a significant factor to evaluate the crashworthiness of the reinforced fiber composite tubes as energy absorbing. For tubes crashed in a progressive failure mode, the deflections of the load applied to the tubes as a purpose of displacement was small and thus offer a stable deceleration.Figure 3 shows the load–displacement for relationS-H-β 50^{o}. Thehexagonalnon-woven kenaf/epoxy tubes have been placed between two rigid platesunder lateral crushing load. They well behaved as a circular which deformed elastically at the pre-crushing stage as shown in Figure 3a. A sudden drop of the load was then observed after reaching the peak load value, which is due to the fracture of the middle supporting plate layer as shown in Figure 3 (b-c).Thenfour longitudinal cracks wereimmediately spotted. The fracture lines werecompletely formed on all sides of the tubes and supporting plates. Once of the crack lines were established close to the interface between the tube and the flat platens, more fluctuations of load were observed at the last stages of the crushing due to the supporting plate's resistance

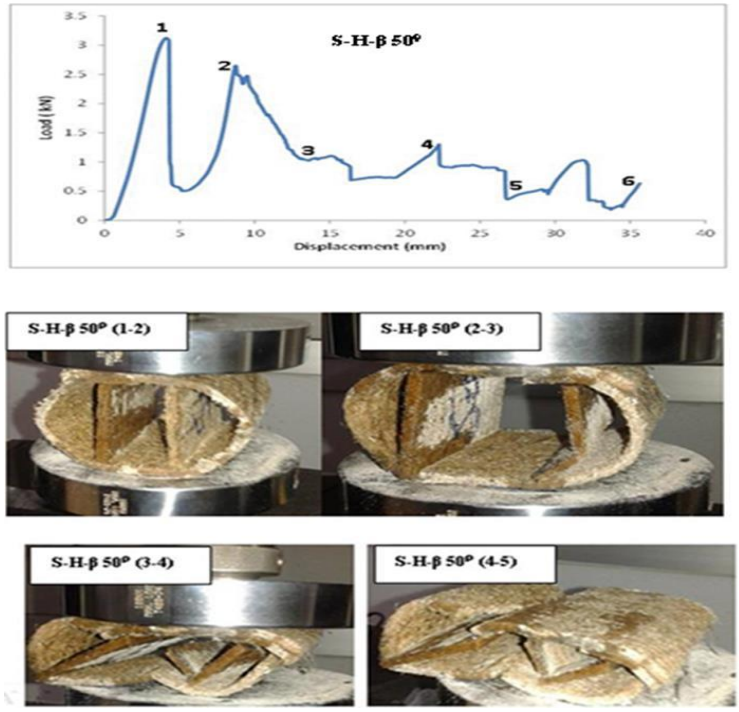

Fig. 3. Load–displacement curves and deformation history of supporting specimens.

Microscopic Investigation

The macroscopic investigation was carried out to investigate the behavior of the specimen outer skin of the non-woven hexagonal kenaf fiber /epoxy tube. This can be seen in Fig 4. It can be noticed that all cracks mechanisms were occurred closely to each other. Moreover, the damage initiated by fiber cracking was relatively long with longitudinal behavior due to the continuous appliedload E. Mahdi et al. [4].

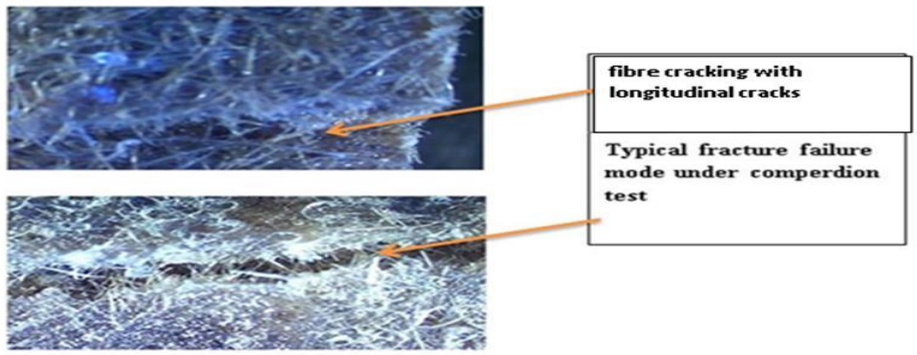

Fig 4. Futures in the fracture surface of the non-woven kenaf fibre /epoxy packing walls.

Conclusions

The lateral compressive loading of both plates supporting hexagonal tubes and empty hexagonal tubes subjected to quasi-static lateral loads were carried out experimentally.

Upon on the current lateral crushing test results and compared with previous studies, the following conclusions can be made:

- The use of plate-inside tube can increase the specific absorbed energy (SAE) for all the tubes with different side angle. That is; the specific energy increases as the hexagonal angle increases from 40^{o} to 60^{o}. .
- All tested specimens under lateral compression load had four longitudinal fracture lines. These fracture lines were formed diametrically opposite to each other.
- Tubes with supporting plate showed higher energy absorption (approximately 60%) compared to non-supporting tubes.
- Specimens with non-supporting plate exhibited lower peak and average crushing loads.

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References

[1] M.F.M. Alkbir, S.M. Sapuan, A.A. Nuraini, M.R. Ishak .Effect of geometry on crashworthiness parameters of natural kenaf fibre reinforced composite hexagonal tube . Mater Des, 60 (2014), pp. 85–93.

[2] Bartosz T., Węcławski, Mizi Fan,David Hui . Compressive behaviour of natural fibre composite. Compos Part B 67 (2014), pp. 183–191.

[3] Libo Yan,, Nawawi Chouwa , Krishnan Jayaraman . Lateral crushing of empty and polyurethane-foam filled natural flax fabric reinforced epoxy composite tubes. Compos Part B-ENG 2014; 63: 15–25.

[4] E. Mahdi,T.A. Sebaey. Crushing behavior of hybrid hexagonal/octagonal cellular composite system: Aramid /carbon hybrid composite. Mater Des 2014;63: 6–13

Seaweed as Biofiller for Polymer Composite: A review

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Abstract. Seaweed is one of the most important agriculture revenue for seaside country such as China, Japan, Philliphine, East Malaysia, etc. It has wide range of application from raw food, food texturing, industrial gelling, medical, cosmetics, and pharmaceutical. However, harvesting and extraction of seaweed mucilage will produce huge amount of waste which is yet to be utilized. On the other hand, other wild species of seaweed which grown at shallow seawater also create problem for marine industries. Application of natural fiber on polymer composite had received much attention from researchers and seaweed is one of them. Thus, this paper focuses mainly on the significance of seaweed as reinforcement material for polymer matrix.

Keywords:seaweed, composite, natural fiber, biocomposite

Introduction

Conventional material such as petroleum based plastic are widely used for variety of products ranging from packaging, electronic products, toys, utensil, automotive interior, etc. This material are very stable, in fact, it requires extremely long time to degrade once it was discarded. In recent years, disposal of non-biodegradable plastic waste have been a major issue for environment since numerous kind of daily used product were manufactured by this non-biodegradable materials. Thus, the need arise to develop alternative materials which is environmental friendly during processing, usage, and disposal as well. Natural resources has attracted large interest due to their potential to be utilized as new biodegradable material. Agricultural waste such as oil palm empty fruit bunches, rice husk, sugar cane bagasse, and sugar palm fiber have been used to develop biocomposite[1]–[4]. Furthermore, marine plant such as seaweed which can be found abundant also had

shown great potential as biocomposite material [5]. Eventhough there are research that had been conducted on seaweed as composite material, however, there are a lot of other seaweed species which the potential has yet to be found.
This article provides an academic review on the latest advances in research on seaweed application as reinforcement for polymer composite.

Seaweed

Seaweeds are macroscopic algae which can be found at the bottom in relatively shallow sea waters. In general, they grow on solid substrates such as rocks, dead corals, shells and other plant materials [6]. For agricultural purposes, seaweed were grown on rope to make ease of cultivation process, this technique were used all over the world including Malaysia.

Seaweeds grow in every sea provided suitable seawater and climate condition. However, there is only few species being used for food and other applications. The most common seaweed being cultivated all around the world including Gracilaria sp, Gelidium sp, Laminaria sp, Eucheuma sp, Aschopyllum sp, Chondrus sp, and Undaria sp. East Malaysia, particularly Sabah, is one of the cultivator for Eucheuma sp.

Application of Seaweed

Mainly, seaweed were used as traditional food for Asian country particularly for China and Japan. However, there are numerous application of seaweed mucilage which is extracted from seaweed such as carrageenan, agar, and alginate. These are known as polysacharide of seaweed which mainly used as thickening agent for food as well as industrial purposes[6].

On the other hand, these polysacharide were also used for cosmetic and healthcare purposes which further increase the demand for cultivation of this marine plant all around the world. However, this positive scenario also created a drawback by increasing the amount of agricultural and industrial waste at the same time which requires optimum utilization of it.

Seaweed as Biofiller in Polymer Composite

Recently, researchers has paid attention to seaweed as one of potential reinforcement material for polymer. This is due to the presence of seaweed waste from cultivation, mucilage extraction, and bioenergy production residue[7]. There are various kind of seaweed which the potential has been studied previously including Ulva armoricana [5], [8], Sargassum tenerrium ([9], Gelidium Elegance [10], Laminaria japonica, Enteromor phacrinite [7], and also red and brown seaweed which the scientifc name was not mentioned by the researchers[11].

In general, seaweed only required a simple process in order for it to be transformed to reinforcement form. The main process includes drying of harvested seaweed at temperature range of 60^{o}C to 100^{o}C for 24-48 hours [5], [12], [13]. Dried seaweed were then crushed into particle form by using pulverizer in order to make ease of mixing process for composite fabrication. However, study conducted by Luan et al., (2010) were using boiling and bleaching method in order to remove mucilage and to obtain fiber of red seaweed namely Gelidium Elegance[10]. It should be noted

that seaweed is a large family of macroalgae, thus, every particular species have their unique physical and properties which might affect the results of extraction. Mainly, seaweed in particle form were mixed with polymer matrix by means of extruder or mixer and followed by compression or injection moulding in order to fabricate the composite [7], [12], [14].

One of the most important reason to develop composite material is to improve the mechanical properties such as strength and rigidity. This were then followed by physical, thermal, and chemical stability of material. Albano et al., (2005) conducted study on seaweed residue incorporation in HDPE showed that seaweed had good characteristic to increase the rigidity of the matrix. Meanwhile, Barghini et al., (2010) showed that seaweed had improve both mechanical and thermal properties of material by its addition as a filler in poly(tert-hydroxy-butyrate) (PHB)[5]. On the other hand, Chiellini et al., (2008) found that addition of green seaweed had no effects on the tensile strength of polyvinyl alcohol (PVA), but, remarkably improve the tensile modulus which proves the strengthening effect of seaweed as a reinforcement material[8]. However, the mechanical strength was compromised as the seaweed content increase to higher percentage of composite which is attributed to the presence of imperfections and loss of cohesion in composites [8].

Hassan et al., (2008) had fabricated hyrid composite consists of seaweed and rice straw as reinforcement in polypropylene(PP) matrix[14]. The results showed that incorporation of seaweed into rice straw/PP composite had further increased the tensile, bending, and impact strength of the composite which proves the comptability and strengthening effect of seaweed as reinforcement in the matrix. In terms of water absorption behavior, addition of seaweed into rice straw/PP composite also had improve the water barrier properties of the composite. According to Hassan et al.,(2008) the lowest uptake of water by the composites indicate that more OH group of cellulose content in the fibers of the composites being blocked by their interaction with the PP matrix, hence hindering them from being accessed by water. Furthermore, addition of seaweed causes degradation rate of the hybrid composite to decreased and lower tensile strength losses were experienced after simulating weathering test. This findings show that seaweed does not only improves the mechanical properties of material, but, also improves the physical and chemical stability when being exposed to environment.

On the other hand, study by Sim et al., (2010) showed that the dynamic mechanical and thermomechanical prop erties of PLA and PP polymer were increased by incorporation of seaweed fiber[11]. This finding showed that seaweed provide good adhesion to the matrix which was then confirmed by morphological analysis. Chitra and Kumari (2012) found that incorporation of red seaweed and brown seaweed as filler for PP matrix had slightly decreased the tensile strength but successfully increase the tensile modulus and thermal stability of the composite[9].

Another study by Luan et al., (2010) showed that incorporation of seaweed into PP matrix had successfully increased the shore hardness, tensile modulus, and flexural modulus of the composite. This is because seaweed fibers are ductile, and the micro fibrils have a spiral orientation with respect to the fiber axis [15]. This study also reported application of Maleic anhydride polypropylene (MAPP) as

compatibilizer for fiber-matrix interface. The finding showed that MAPP had futher improve the mechanical properties of this composite by eliminating weak boundary layers by chemical bondings at the matrix-fiber interface [15].

Conclusions

In general, application of seaweed as reinforcement material for polymer composite had shown a positive trends such as improvement of mechanical, thermal, and physical properties of the polymer matrix. In addition, seaweed incorporation into polymer matrix also had improve the degradation characteristic by minimizing the strength losses due to environmental exposure. These valuable findings by previous studies proves that seaweed has a great potential to be used as reinforcement material, however, the number of study regarding seaweed reinforcement are limited and there are various kind of species which the potential has yet to be explored. Furthermore, incorporation of seaweed with biodegradable polymer is also an important area of research which could solve the waste degradation problem being encountered nowadays. However, most of the study reviewed in this paper were using non-biodegradable polymer as the matrix. Thus, the future of seaweed application as reinforcement material should be explored extensively especially on the biodegradable polymer in order to produce greener environment for future generation.

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References

[1] M. Jawaid, H. P. S. Abdul Khalil, and a. Abu Bakar, "Woven hybrid composites: Tensile and flexural properties of oil palm-woven jute fibres based epoxy composites," *Mater. Sci. Eng. A*, Vol. 528, no. 15, 2011 pp. 5190–5195.

[2] M. R. Ishak, S. M. Sapuan, Z. Leman, M. Z. a Rahman, U. M. K. Anwar, and J. P. Siregar, "Sugar palm (Arenga pinnata): Its fibres, polymers and composites.," *Carbohydr. Polym.*, Vol. 91, no. 2, 2013, pp. 699–710.

[3] H.-S. Yang, H.-J. Kim, J. Son, H.-J. Park, B.-J. Lee, and T.-S. Hwang, "Rice-husk flour filled polypropylene composites; mechanical and morphological study," *Compos. Struct.*, Vol. 63, no. 3–4, 2004, pp. 305–312.

[4] W. N. Gilfillan, D. M. T. Nguyen, P. a. Sopade, and W. O. S. Doherty, "Preparation and characterisation of composites from starch and sugar cane fibre," *Ind. Crops Prod.*, Vol. 40, 2012, pp. 45–54.

[5] A. Barghini, V. I. Ivanova, S. H. Imam, and E. Chiellini, "Poly-(ε-caprolactone) (PCL) and poly(hydroxy-butyrate) (PHB) blends containing seaweed fibers: Morphology and thermal-mechanical properties," *J. Polym. Sci. Part A Polym. Chem.*, Vol. 48, no. 23, 2010, pp. 5282–5288.

[6] R. Gade, M. S. Tulasi, and V. A. Bhai, "Seaweeds: A Novel Biomaterial," *Int. J. Pharm. Pharm. Sci.*, Vol. 5, no. 2, 2013, pp. 40–44.

[7] Y. H. Jang, S. O. Han, I. N. Sim, and H.-I. Kim, “Pretreatment effects of seaweed on the thermal and mechanical properties of seaweed/polypropylene biocomposites,” *Compos. Part A Appl. Sci. Manuf.*, Vol. 47, 2013, pp. 83–90.

[8] E. Chiellini, P. Cinelli, V. I. Ilieva, and M. Martera, “Biodegradable thermoplastic composites based on polyvinyl alcohol and algae.,” *Biomacromolecules*, Vol. 9, no. 3, 2008, pp. 1007–1013.

[9] N. Chitra and R. Kumari, “Studies on Polypropylene Biocomposite with Sea Weeds,” *Res. J. Pharm. Biol. Chem. Sci.*, Vol. 3, no. 3, 2012, pp. 1165–1170.

[10] M. W. Lee, S. O. Han, and Y. B. Seo, “Red algae fibre/poly(butylene succinate) biocomposites: The effect of fibre content on their mechanical and thermal properties,” *Compos. Sci. Technol.*, Vol. 68, no. 6, 2008, pp. 1266–1272.

[11] K. J. Sim, S. O. Han, and Y. B. Seo, “Dynamic mechanical and thermal properties of red algae fiber reinforced poly(lactic acid) biocomposites,” *Macromol. Res.*, Vol. 18, no. 5, 2010. pp. 489–495.

[12] C. Albano, a. Karam, N. Domínguez, Y. Sánchez, J. González, O. Aguirre, and L. Cataño, “Thermal, mechanical, morphological, thermogravimetric, rheological and toxicological behavior of HDPE/seaweed residues composites,” *Compos. Struct.*, Vol. 71, no. 3–4, 2005, pp. 282–288.

[13] M. M. Hassan, M. Mueller, D. J. Tartakowska, and M. H. Wagner, “Mechanical Performance of Hybrid Rice Straw / Sea Weed Polypropylene Composites,” *J. Appl. Polym. Sci.*, Vol. 120, 2011pp. 1843–1849.

[14] M. M. Hassan, M. Mueller, and M. H. Wagners, “Exploratory study on seaweed as novel filler in polypropylene composite,” *J. Appl. Polym. Sci.*, Vol. 109, no. 2, 2008, pp. 1242–1247.

[15] L. Luan, W. Wu, M. H. Wagner, and M. Mueller, “Seaweed as novel biofiller in polypropylene composites,” *J. Appl. Polym. Sci.*, 2010, p. n/a–n/a.

Effects of various plasticizers and concentration on physical properties of cassava films

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Abstract. The present study investigated effects of plasticizer (Fructose, Ethylene glycol, Tri-ethylene glycol and Tri-ethanolamine glycol) on physical and mechanical properties of cassava starch-based edible films. Films were obtained from solutions containing gelatinized starch, plasticizers and water by casting and evaporating water at 45^{o}C. Thickness, density, Water content and solubility in water of films were measured, the result show that the density increased with increasing plasticizer content except for TEA. Effects of plasticizers on physical properties of films were largest for TEG and smallest for EG. High contents of plasticizers resulted in changes in physical properties of films probably due to phase separation and crystallization.

Introduction

The increase in non-biodegradable waste material and the difficulty in recycling most of the available synthetic packaging have been pushing researches toward the development of new biodegradable materials, which are suitable for packaging. In this regard, the production of edible films may play an important role in food preservation [1,2].Several studies have been carried out on the use of starches from different sources to obtain films and coatings with different properties. The reported results indicate that these carbohydrates are promising materials in this regard [3-5].Film characteristics are dependent on the cohesion of the polymeric matrix, which in turn is dependent on the structure of the polymer chains, the film obtainment process and the presence of plasticizer agents. The most used plasticizers for starch-based films are glycerol [6-8].Addition of plasticizers, affects film properties such as water sorption, mechanical properties and glass transition temperature (Tg). Simultaneously as plasticizer content of a starch film increases, water content of the film decreases at low relative humidity (RH) whereas water content of the film increases significantly at high RH [2]. The purpose of this study was to investigate effects ofvarious plasticizers and plasticizers contents on physical andmechanical properties of cassava starch-based films.

Materials and methods

Materials

Native cassava starch,Starch was extracted from cassava tubers; the tubers were washed, peeled and grated. The resulting paste was mixed with water and the solution

was filtered on a clean cloth. The white precipitate (starch) was then recovered, sun-dried and used to prepare edible films.Fructose, Ethylene glycol, Tri-ethylene glycol and Tri-ethanolamine glycol were used as plasticizers.

Film preparation and characterization

Starch films were prepared through the casting technique using a film-forming solution containing 5g of cassava starch. Fructose, Ethylene glycol, Tri-ethylene glycol and Tri-ethanolamine glycol were used as plasticizers, at concentrations of 0.15, 0.30 and 0.45 g/g dry starch. The mixture was heated to 80 ^{o}C in a thermal bath under constant stirring.Air bubbles formed during heating were removed by placing the film forming solution into a desiccator under vacuum until there was no bubble and poured homogenously onto circle plates. The plates with the film forming solution were then dried in an oven with air circulation, at 45^{o}C. The dry films were removed from the plates and characterized.

(A) (B) (C)

Fig.1:Cassava film preparing at INTROP lab using different plasticizers. A: cassava film with various plasticizers in casting plate, B: cassava film after removing from plate, C: cassava film sample.

Film thickness

The film thicknesses were determined according to the method of Cao, Fu, and He (2007) using a digital micrometre (Mitutoyo Co., Japan) from the average of five random measurements performed for each film and an average value was calculated. [9]

Film density

Square samples of the films (20 x20 mm), the film density was determined directly from the film weight anddimensions (volume) and the values considered were theaverage of ten determinations. Film density was calculated by dividing the film weight by the film volume, where the film volume was calculated by multiplying the film area by the thickness [5].

Water content

Film moisture content (WC) was determined through the weight loss. Film samples were weighed (w1), dried at 105 ^{o}C for 24 h, and weighted (w2) again. Water content

was determined as the percentage of initial film weight lost during drying and reported on a wet basis according to Eq1.The temperature was chosen to avoid loss of plasticizer.

$$WC(\%) = \frac{W1-W2}{W1} X100 \quad (1)$$

Where: WC (%): Water content (%); W1: initial weight and W2: final weight after drying

.

Film solubility in water

Water solubility of films was made in triplicate and measured following the method of ShojaeeAliabadi et al. (2013). Film samples were dried at 90 ◦C for 24 h in a laboratory oven and then weighted to determine initial solid content. Pre-weighed film samples (1 cm × 3 cm) were immersed under constant agitation in 50 ml of distilled water for 6 h at 25 ◦C. After that period, the remaining pieces of films were filtered and dried at 90 ◦C to constant weight (final dry weight). The water solubility (%) of the film was calculated according to followingEquation: [10]

$$WS(\%) = \frac{Wi-Wf}{Wi} X100 \quad (2)$$

Where: WS (%): Water solubility (%); Wi: initial dry weight and Wf: final dry weight

Results and discussion

Thickness and density of the starch films

There was significant difference in the film thicknesses and density among treatments, there were increased upon plasticizer addition and showed very small variations. The thicknesses of films plasticized with fructose rangedfrom 210 ± 15µm (0.15g F/g starch) to270 ± 20µm (0.45 g F/g starch), while the density of dry films variedfrom 2.07 % g/cm³ (0.15g F/g starch) to1.73g/cm³ (0.30 g F/g starch). On the other hand,the thicknesses of films plasticized with Ethylene glycol rangedfrom 97.5 ± 12 µm (0.15 g EG /g starch) to 300 ± 10 µm(0.45 gEG /g starch), while the density of these films afterdrying varied from 3.41 g/cm³ (0.15 g EG/g starch)to 2.67 ± 0.12 g/cm³ (0.30 g EG/g starch). Moreover, The thicknesses of films plasticized with Tri-ethylene glycol ranged from 190 ± 12 µm (0.15g TEG/g starch) to410 ± 16 µm (0.45 gTEG/g starch), while the density of dry films varied from 1.72 % g/cm³ (0.30g TEG/g starch) to0.69g/cm³ (0.45 g TEG/g starch). On the other hand, the thicknesses of films plasticized with Tri-ethanolamine glycol ranged from 155 ± 9 µm (0.15 g TEA /g starch) to 193 ± 10 µm (0.45 g TEA /g starch), while the density of these films after drying varied from 167 g/cm³ (0.15 g TEA/g starch) to 404 ± 0.12 g/cm³ (0.45 g TEA/g starch).

Table 1: Physical properties of cassava films incorporated with various concentrations of plasticizer.

Plasticizer type and content		Thickness (mm)	Density (g/cm^3)	Water content (%)	Solubility in water (%)
F This study	15%	0.21	2.07	10.32	23.7
	30%	0.1875	1.73	10.96	33.9
	45%	0.27	1.74	12	42.7
EG This study	15%	0.0975	3.41	13.95	24.6
	30%	0.225	2.67	19.82	19.8
	45%	0.30	----	20.12	----
TEG This study	15%	0.19	----	18.23	----
	30%	0.2625	1.72	20.94	24.1
	45%	0.4175	0.69	29.11	27.1
TEA This study	15%	0.155	1.67	10.08	---
	30%	0.17	2.44	11.14	45
	45%	0.193	4.04	11.48	41.9
GLY Belibi, et al. (2014) [11]	30 %	0.193	----	11.8	23
	45 %	0.210	----	41.1	32.1

Where: F=Fructose, EG=Ethylene glycol, TEG=Tri-ethylene glycol, TEA = Tri-ethanolamine glycol and GLY = Glycerol

Water Content and Solubility in Water

When plasticizersconcentration increased from 15 to 30 and then to 45% (w/w)the moisture content of cassava films increased significantly due to the water holding capacity of plasticizers [12]. Films with a higher concentration of plasticizer showed higher densities and the type of plasticizers also influenced the film densities.Film solubility in water increasing with the increasing plasticizers concentration, The Tri-ethanolamine glycol has the greatest effect on solubility in water (45%) for (0.30 g TEA /g starch). In contrast, the Ethylene glycol has the lowest effect with (19.8%) for (0.15 g EG /g starch).

Conclusions

Cassava starch-based films plasticized with various plasticizerswere prepared by a casting method. Plasticizer typeand content were found to affect physical properties of the starch films. Watercontents increased with increasing plasticizer contentand TEGhad the greatest effect on water content. However, the TEA has the greatest effect on solubility in water.

References

[1] Lu, Y., Weng, L., & Cao, X. (2006). Morphological, thermal and mechanical properties of ramie crystallites-reinforced plasticized starch biocomposites. Carbohydrate Polymers, 63, 198–204.

[2] Mylla¨rinen, P., Partenen, R., Seppa¨la¨ , J., &Forssell, P. (2002). Effect of glycerol on behaviour of amylase and pectin films. Carbohydrate Polymers, 50, 355–361.
[3] Mali, S. (2002). Produc¸a˜o, caracterizac¸a˜o e aplicac¸a˜o de filmespla´sticosbiodegrada´veis a base de amido de cara´ . Doctor Thesis. Cieˆncia e Tecnologia de Alimentos, Departamento de Tecnologia de Alimentos e Medicamentos. UniversidadeEstadual de Londrina, Brazil, 362 f.
[4] Mylla¨rinen, P., Partenen, R., Seppa¨la¨ , J., &Forssell, P. (2002). Effect of glycerol on behaviour of amylase and pectin films. Carbohydrate Polymers, 50, 355–361.
[5] Larotonda, F. D. S., Matsui, K. N., Sobral, P. J. A., &Laurindo, J. B. (2005). Hygroscopicity and water vapor permeability of Kraft paper impregnated with starch acetate. Journal of Food Engineering, 71(4), 394–402.
[6] Gontard, N., &Guilbert, S. (1996). Bio-packing: Technology and properties of edible and/or biodegradable material of agricultural origin. Boletim da SociedadeBrasileira de Cieˆncia e Tecnologia de Alimentos, 30(1), 3–15.
[7] Krochta, J. M., & Miller, K. S. (1997). Oxygen and aroma barrier properties of edible films: A review. Trends in Food Science and Technology, 8, 228–237.
[8] Lourdin, D., Coignard, H., Bizot, H., & Colonna, P. (1997). Influence of equilibrium relative humidity and plasticized concentration on the water content and glass transition of starch materials. Polymer, 38, 5401–5406.
[9] Cao, N., Fu, Y. H., & He, J. H. (2007). Preparation and physical properties of soy protein isolate and gelatin composite films. Food Hydrocolloids, 21, 1153e1162
[10] Shojaee-Aliabadi, S., Hosseini, H., Mohammadifar, M. A., Mohammadi, A., Ghasemlou, M., Ojagh, S. M., et al. (2013). Characterization of antioxidant–antimicrobial _-carrageenan films containing Saturejahortensis essential oil. International Journal of Biological Macromolecules, 52(1), 116–124.
[11] Belibi, P. C., Daou, T. J., Ndjaka, J. M. B., Nsom, B., Michelin, L., & Durand, B. (2014). A comparative study of some properties of cassava and tree cassava starch films. Physics Procedia, 55, 220-226.
[12] Rhim, J. W., Gennadios, A., Weller, C. L., & Hanna, M. A. (2002). Sodium dodecyl sulfate treatment improves properties of cast films from soy protein isolate. Industrial Crops and Products, 15(2), 199–205.

Application of cassava and cassava composites

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Abstract. Cassava is a species of plant which has different purpose of use. It is used to produce various foods, bio-fibres, bio-composites and biopolymers. Besides, it now is used as a renewable energy source as starch through multi process. The most popular products produced through cassava are food, biopolymer and flour although there are many other products that cassava produces. The main focus of this paper is on the importance of fibres and polymers since they are attributed to be extremely biodegradable and durable. Moreover, it is easy to process them as they are naturally available.

Keywords: Cassava; Bio-products; Cassava composite

Introduction

Cassava is one of the staple foods for about 800 million of population in South America, Asia, the pacific islands and Africa, it is very significant for these countries [1-3].Besides, an advantage of cassava is that it can be grown on marginal lands and bad soils as it is tolerant to bad weather and drought.Cassava has a wide range of usage with respect to many industrial applications. Popular examples include production of ethanol and biofuel which has become quite a focus by researchers in the last decade with respect to limited reserve of fossil oil [4].Cassava plays a particularly important role in agriculture in developing countries, especially in sub-Saharan Africa, because it does well on poor soils and with low rainfall, and because it is a perennial that can be harvested as required[5]. With the increase in awareness levels about the environment, the interest to develop biodegradable material via renewable sources seems to be growing. Since starch is attributed to offer a favourable combination of cost, availability and performance is known to be very effective and valuable as compared to other polymers [6]. Recently, there has been a rapid increase in the composite components' use in sports, leisure and automotive. This has generally focused on renewable and sustainable reinforced composites[7].

Cassava Application

There are multiple uses of biodegradable polymer materials. Examples include agriculture, medicine, automotive, packaging and drug release. There is an increased awareness of environmental responsibility with respect to the industry as well as the consumers. The processes that make use of greater development of biopolymer

materials via natural resources, appears to be most promising [8]. Cassava is also sued as the raw material to produce cakes, traditional foods and cassava starches.Cassava also provides significant energy especially to the households with low incomes, as compared to other food items. Furthermore, cassava is also used to prepare many household food items like paste, sauce, biscuits and sago bread as shown in figure1.

Fig.1: Household food prepared from cassava; A: cassava bread, B: cassava cake. C: cassava pasta

Bio-products from Cassava

Acetone, cellulose, and ethanol from cassava

There are various uses of cassava and it used extensively. Examples include using it in the production of ethanol and biofuel, which has now become a focus point by many researchers over the last ten years, with respect to the reserve of limited fossil oil [4].

One of the latest developments in the field of biochemical engineering pertains to the use of the biodegradable waste or generating more significant products like organic acids, xylenes enzyme, bio polymer and antibiotics [9, 10]. Cassava has recently been found with greater uses as it can be used in the production of xylenes through Bacillus subtitles with the use of cassava bagasse which is submerged in fermentation. The greatest production occurred when cassava bagasse was used as this process is economical. There was greater stability of the enzyme when the alkaline level was at 600C and pH 8. There is a huge industrial potential in the thermostable and alkaline xylenes that is produced via Bacillus subtitle [11]. Furthermore, kraft PA utilized to form a composite which is quite like cardboard. In this process, a fibre like residue which was highly rich in bagasse was used. The percentages of cassava bagasse and Kraft paper in this production was 90% and 10% respectively.

Cassava Starch

Starch is one of the most widely available and cheapest agricultural products which can be fully degradable in several environments[12]. It is because of these characteristics that starch is examined as a polymer in several applications. The creation of thermoplastic starch involves temperature and pressure extrusion and/or moulding. The degradation of pure-starch polymer takes place very rapidly in a composting atmosphere.

Several thermoplastic starch composites are generated on the basis of combining starch and vinyl alcohols, and such polymers have a tendency of being more stable. However, there is an inverse relationship in the biodegradability of such composites and their starch content [13].

Fig.2:Cassava root and Cassava starch. A: cassava root, B: cassava starch after filtration, C: cassava starch

Cassava bagasse

Cassava bagasse, a by-product of cassava starch industrialization, was examined by Pasquini et al. (2010) as a new raw material for obtaining cellulose whiskers. The by-product is essentially made of cellulose fibres (17.5 wt %) and residual starch (82 wt %). The study showed that it is possible to attain high value-added products from an agricultural waste residue. Those cellulose whiskers that had a high aspect proportion were removed from the cassava bagasse directly and were used in the process of nano-composite films alongside a natural rubber matrix. The inclusion of filler led to a considerable improvement in the storage tensile modulus [14].

The industrial applications to generate cassava starch use the solid residue (bagasse) that is obtained from the extraction that consists of fibrous materials and starch which has not been extracted. Substantial amount of bagasse is generated in the industry, which is around 900 kg of bagasse, with 85% moisture for every tonne of processed root. The renewable solid waste material, cassava bagasse is developed from processing industry and is present in lands close to the processing unit. However, it has a larger organic content and bio-degradability which leads to greater pollution [9]

Fig.3:Cassava Bagasse. A: cassava root, B: cassava bagasse before drying, C: cassava bagasse after drying

Cassava Peels

Cassava, one of the basic foods in the tropical regions, is normally utilized in traditional food items, cakes, etc. There is a significant amount of cassava production; however, very small quantity is used in traditional food industries, while the remaining is utilized in the form of raw materials in cassava starch industries. The production of cassava starch usually involves a significant amount of solid wastes (cassava peel), and if these solid wastes are directly released, significant environmental issues can arise[15].Cassava peel is a thin brown outer layer that has a thicker leathery parenchymatous inner layer. This is the foremost waste that is

developed in cassava processing that take place in either food or other industrial products. The cassava peel can form around 10-20% of the wet weight of the roots [16] which suggests that it has a huge potential to be used in biotechnological and industrial processes since it is available in large quantities and is not very expensive. These peels are, however, considered to be a waste substance and are directly released which leads to severe environmental issues[17, 18].[10, 19] had another investigation to study the influence of adding cassava peels and coal ash on the compressed earth bocks engineering characteristics (CEB). This mixture comprises 5% of coal ash to the clayed soil was seen to have the greatest result of coal ash (7.5%) where the cassava peels were 2.5%. Cassava peels are found in great quantities in the wastes and they can be used to make CEBs. This way, the environmental problem can be reduced [20]. Cassava peeling ash (CPA) is used in cement for the concrete work. The most effective mix was observed to be 15 to 20% CPA with respect to the safety and strength of the concrete. As a conclusion, it was said that concrete which comprises of 30% CPA is stable and can be used in most of the concrete work. Such an alternative use will promote greater value economically to the cassava peelings.

Fig.4: Cassava Peel. A: cassava root, B: drying cassava peel, C: grinding cassava peel

Cassava polymer

Cassava starch has been extensively used to produce films and the results indicated that these carbohydrates are promising materials in this regard. (Chen & Lai, 2008;Kaisangsri, Kerdchoechuen, &Laohakunjit, 2012; Kechichian et al,2010; Mali, Pelissari et al., 2012; Souza et al.). Native cassava starch was extracted from cassava tubers and used to prepare edible films. Using different types of plasticizers.

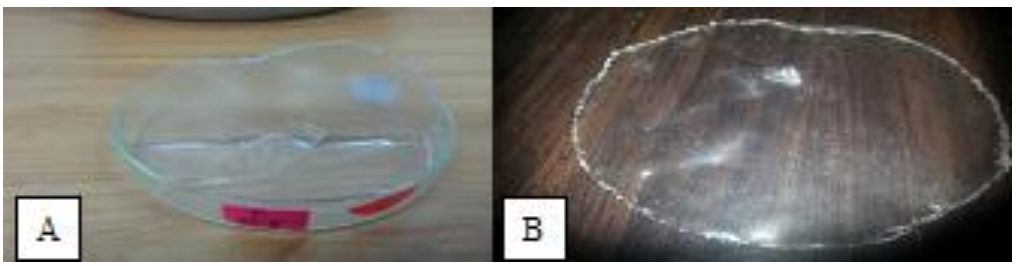

Fig.5:Cassava starch film prepared at UPM lab. A: plate of cassava film, B: cassava film.

Cassava composite application

Cassava starch has been successfully expanded under extrusion conditions. Due to its low bulk density, a little modification is needed so that its moisture content is increased. Twin screw extrusion is recommended for direct expansion of cassava starches. Cassava starch can be expanded in moulds, at 200-240°C for 1-3 minutes, to form into package utensils, such as bowls. [21]
Parra et al (2004) produced film from blending cassava starch with glycerol and PEG, the films were transparent, homogeneous and flexible. The average thickness of the films was 0.012 mm.[22]

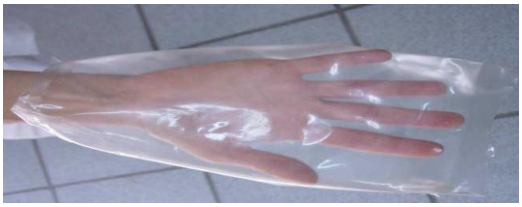

Fig.6: Illustrative picture of cassava starch edible film.[22]

The components of the formulations (starch, chitosan and glycerol) were mixed. In the first stage of the extrusion process, the mixtures were extruded and pelletized twice to obtain good homogenization (Fig. 1a). Next, the reprocessed pellets were used to manufacture the film by blown extrusion (Fig. 1b).[23]

Fig.7: Film production by extrusion: (a) acquisition of pellets and (b) formation of tubular film.[23]

Conclusions

Cassava starch is available widely and it is quite cheap; therefore, cassava starch is being used in a wide variety of application and it is expected that the use of cassava would increase in the near future [24].Efforts have been made to use cassava fibres as an alternative to the synthetic fibre because of its morphological and chemical properties, particularly in those countries where limited wood resources are available. The concept of 'from waste to wealth and recyclable material' has been promoted by the increased deposits of organic wastes like cassava bagasse and peels. In applications like production of enzymes, organic acids, feed, etc. the use of cassava bagasse can be economical.

References

[1] Gleadow, R.M., et al., *Growth and nutritive value of cassava (Manihot esculenta Cranz.) are reduced when grown in elevated CO2.* Plant Biology, 2009. 11(s1): p. 76-82.
[2] FAOstat, F., *agriculture organization of the United Nations.* Statistical database, 2009.
[3] Iyer, S., D.S. Mattinson, and J.K. Fellman, *Study of the early events leading to cassava root postharvest deterioration.* Tropical plant biology, 2010. 3(3): p. 151-165.
[4] Lu, Y., Y. Ding, and Q. Wu, *Simultaneous saccharification of cassava starch and fermentation of algae for biodiesel production.* Journal of Applied Phycology, 2011. 23(1): p. 115-121.
[5] Stone, G.D., *Both Sides Now.* Current Anthropology, 2002. 43(4): p. 611-630.
[6] Abdillahi, H., et al., *Influence of citric acid on thermoplastic wheat flour/poly (lactic acid) blends. II. Barrier properties and water vapor sorption isotherms.* Industrial Crops and Products, 2013. 50: p. 104-111.
[7] Wambua, P., J. Ivens, and I. Verpoest, *Natural fibres: can they replace glass in fibre reinforced plastics?* composites science and technology, 2003. 63(9): p. 1259-1264.
[8] Chauhan, A., *Environment-friendly biodegradable polymers and their applications.* Malaysian Polym. J, 2012. 7: p. 62-67.
[9] Pandey, A., et al., *Biotechnological potential of agro-industrial residues. II: cassava bagasse.* Bioresource Technology, 2000. 74(1): p. 81-87.
[10] Sugumaran, K., et al., *Cassava bagasse—a low cost substrate for thermotolerant xylanase production using Bacillus subtilis.* Int J ChemTech Res, 2013. 5(1): p. 394-400.
[11] Matsui, K.N., et al., *Cassava bagasse-Kraft paper composites: analysis of influence of impregnation with starch acetate on tensile strength and water absorption properties.* Carbohydrate polymers, 2004. 55(3): p. 237-243.
[12] Whistler, R., *History and future expectation of starch use. Starch: Chemistry and Technology, RL Whistler, JN BeMiller, EF Paschell*. 1984, academic Press, Orlando, FL.
[13] Glenn, G.M., et al., *Heat expanded starch-based compositions.* Journal of agricultural and food chemistry, 2007. 55(10): p. 3936-3943.
[14] Pasquini, D., et al., *Extraction of cellulose whiskers from cassava bagasse and their applications as reinforcing agent in natural rubber.* Industrial Crops and Products, 2010. 32(3): p. 486-490.
[15] Sudaryanto, Y., et al., *High surface area activated carbon prepared from cassava peel by chemical activation.* Bioresource Technology, 2006. 97(5): p. 734-739.
[16] Obadina, A. and S. Abiola, *Fungal enrichment of cassava peels proteins.* African Journal of Biotechnology, 2006. 5(3): p. 302-304.
[17] Aderemi, F. and F. Nworgu, *Nutritional status of cassava peels and root sieviate biodegraded with Aspergillus niger.* Am.-Eurasian J. Agric. Environ. Sci, 2007. 2: p. 308-311.

[18] Okpako, C., et al., *Proximate composition and cyanide content of cassava peels fermented with Aspergillus niger and Lactobacillus rhamnosus.* Journal of Food Agriculture And Environment, 2008. 6(2): p. 251.
[19] Villamizar, M.C.N., et al., *Effect of the addition of coal-ash and cassava peels on the engineering properties of compressed earth blocks.* Construction and Building Materials, 2012. 36: p. 276-286.
[20] Oladipo, I., J. Adams, and J. Akinwande, *Using Cassava Peelings to Reduce Input Cost of Concrete: A Waste-to-Wealth Initiative in Southwestern Nigeria.* Universal Journal of Environmental Research & Technology, 2013. 3(4).
[21] Poovarodom, N. and S. Praditduang, *The development of packaging from cassava starch.* Packaging Directory Thailand. The Thai Packaging Association, 1999: p. 41-42.
[22] Parra, D., et al., *Mechanical properties and water vapor transmission in some blends of cassava starch edible films.* Carbohydrate Polymers, 2004. 58(4): p. 475-481.
[23] Pelissari, F.M., et al., *Constrained mixture design applied to the development of cassava starch–chitosan blown films.* Journal of Food Engineering, 2012. 108(2): p. 262-267.
[24] Westby, A., *Cassava utilization, storage and small-scale processing.* Cassava: biology, production and utilization, 2002: p. 281-300.

Mechanical properties of woven kenaf reinforced Phenolic resin produced using a hot press technique

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Abstract. Kenaf fiber reinforced composite shows a bright future, among other natural fiber reinforced composites due to its availability and readiness to be used with various manufacturing processes. Unidirectional long fibers or randomly oriented short fiber shapes are the most common type of kenaf fibers that were investigated with either epoxy or polyester resin in previous works. This work evaluates the tensile and flexural properties of plain woven kenaf fabric with Phenolic resin, in order to assess their suitability as lignocellulosic reinforced polymer composites. The hot press manufacturing technique was used to prepare the specimens and eight specimens were prepared for each test, (five replications were adopted). The results show that the plain woven kenaf/phenolic composites possess a good tensile and flexural strength and they are good candidates as reinforcement material in many applications. In addition, the tensile and flexural strength of WKFPH composite were found to be higher than the tensile and flexural strength of woven kenaf/epoxy composite, while the elongation at break exhibited by WKFPH composite was almost same.

Keywords: Plain woven kenaf, Phenolic resin, mechanical, natural-based composite.

Introduction

In the past two decades a number of researchers have sought to use natural fibers with polymer matrix composite (PMC) have received considerable attention both in the literature and industry. Many of the woven natural fabrics are rising as a viable option to glass fiber reinforced composites in industrial applications like packaging, paper making and composite materials with many uses, including as parts in automotive, aircraft, building materials, agriculture, furniture and defense industry application and sport equipments [1]. One of the popular natural fibers is kenaf fiber, which is an annual plant due to its rapid growth, average yield of 1700 kg/ha [2].

Kenaf fibers have the advantage that they are renewable resources, low cost, light, plentiful, non-abrasive, and non-hazardous, therefore they can serve an excellent reinforcing agent for plastics [3]. Kenaf fiber possesses moderately high specific strength and stiffness that could be utilized as reinforcing materials in polymeric resins to make useful structural composite material. Kenaf has been already adopted as automobile parts due to lightweight and good mechanical properties [4].

Woven fabrics are formed in particular by interlacing of fiber bundle (yarns) to form a fabric layer, which offered advantages in terms of good dimensional stability and high packing density. The use of the plain weave technique can add structural strength to the material because it increases both the strength and the ability of energy absorption capacity.Whilst articles and even books on the overall properties of natural fiber reinforced composites have been published, the authors have concluded that a specific article on the overall characteristics of plain woven kenaf fiber reinforced Phenolic resin composites, has not yet been published and give value addition to enhance its use. In an experimental study, the tensile and flexural strength of the unidirectional kenaf fiber composites were investigated by Ochi [5] with different fiber content. Experimental results showed that the weight of composites increased linearly up to a fiber content of 50%. Several attempts have been made to study the influence of fiber content on mechanical properties of kenaf bast fiber reinforced thermoplastic polyurethane (TPU) composites with different fiber loadings was investigated by Shekeil et al. [6]. It was concluded that a 30% fiber loading display the best tensile strength, whilst the tensile modulus, thermal stability, hardness, and flexural strength increased with increase of fiber content, but the strain decreased.

Even though kenaf fibers have the potential to supplement synthetic fibers in polymer composites, limitations arise with respect to mechanical performance and moisture absorption when natural fibers are used [7]. This study is a part of an ongoing study to achieve the advantages of using kenaf fibers and woven pattern on the tensile and flexural properties in order to draw a brief guideline for future development on using woven kenaf fibers.

Materials and Methods

In order to study the behavior of woven kenaf fibers with Phenolic resin (WKFPH) on the mechanical properties of composites, tensile and flexural test were carried out. Kenaf fiber is the main fiber that's used in this study and was supplied by ZKK Sdn Bhd, Malaysia. Table 1 shows the properties of plain woven kenaf that used in this study.The composite samples of WKFPH were made with 40% kenaf fiber weight content by using a hot press technique which leads to better fiber-to-resin bonding, as shown in Figure 1. Eight specimens were fabricated for each test, five replications were considered.

Table 1: Properties of plain woven kenaf.

Characterization	Woven kenaf
Thickness, t (mm)	2 ± 0.2
Weight (g/m^2)	890
Density (g/cm^3)	1.2
Warp density (warp/inch)	12
Weft density (weft/inch)	12
Wavelength, λ (mm)	4.2
Inter-yarn fabric porosity (ε)	0.274
Moisture Content (%)	8.353
Water Uptake (%)	148.86
Average breaking strength (MPa)	100.64
Average maximum strain (%)	17.3

Figure 1: The composite specimenby using the hot press technique.

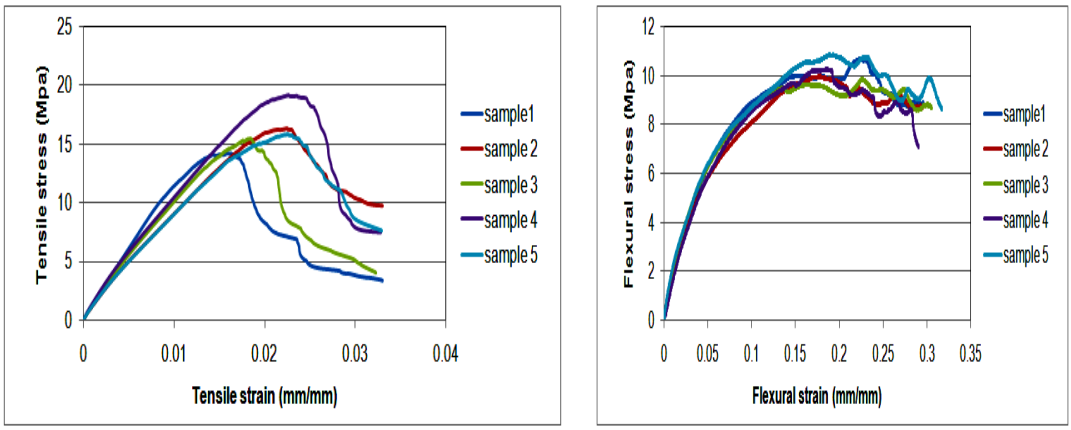
Mechanical properties of WKFPH

Tensile and flexural testing were carried out in the composite laboratory of the Mechanical Department, Universiti Putra Malaysia, according to ASTM D 3039 and ASTM D-790 standard [8, 9], to determine the ultimate tensile strength and ultimate flexural strength of the WKFPH. Tensile specimens were cut to the 250mm × 25mm × 7mm, rectangular sectional area flat strip and gage length 170mm. The rectangular shape three-point bending specimens were prepared with dimensions of 127mm × 12.7mm × 7mm. The distance between supports (span length) was calculated as per the standard, with a ratio of 16:1. Both tests were conducted by using a universal testing machine INSTRON 3365 with the capacity of 100 KN, and crosshead speed 2 mm/min with replication 8 times.

Results and Discussions

Figure 2 (a) shows the tensile strength of WKFPH, the tensile stress curve is shown linearity in the first phase followed by non-linearity up to fracture. Similar trend had been reported in the study done by Khan et al. [10] on the jute fabric reinforced composites. The maximum tensile strength and modulus of WKFPH composite in this study is 19.19 MPa and 889.22 MPa, which is found to be higher than the tensile strength of woven kenaf/epoxy composite which were 16.46MPa and 500.1 MPa [11]. The similar finding was also observed in the flexural strength of WKFPH by using a 3- point flexural test, as shown in Figure 2 (b). It is noted that the flexural stress curve is shown linearity in the first phase followed by non-linearity up to fracture, the staircase region, which lead to the sudden rupture of the specimens. The ultimate flexural strength and flexural modulus were 10.86 Mpa and 282.96 respectively. Generally, the increase in tensile strength and modulus of the composites is attributed to differences in the load-distribution properties. The maximum tensile strain was 2.26%, while the maximum flexural strain was 0.14%, because the flexural effect can

create an interlocking structure which could result in constraints for the extension of the kenaf fiber along the directions.



(a) (b)

Figure2: (a) Tensile properties ofWKFPH of composites, (b) Flexural properties of WKFPH of composites.

Conclusions

The woven kenaf composite material is fabricated with Phenolic resin (WKFPH) to study the tensile and flexural properties with more emphasis. It was concluded that WKFPH composites possess a good tensile and flexural strength; therefore it is possible to be a good candidate as reinforcement material in many applications. In addition, the tensile and flexural strength of WKFPH composite was found to be higher than the tensile and flexural strength of woven kenaf/epoxy composite, while the elongation at break exhibited by WKFPH composite was almost same.

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References

[1] Y.A. El-Shekeil, S.M. Sapuan, M.W. Algrafi., 2014. Effect of fiber loading on mechanical and morphological properties of cocoa pod husk fibers reinforced thermoplastic polyurethane composites. Materials and Design 64, 330–333.

[2] N. Saba, M.Jawaid, K.R.Hakeem, M.T.Paridah, A.Khalina, O.Y.Alothman., 2015. Potential of bioenergy production from industrial kenaf (Hibiscus

cannabinus L.) based on Malaysian perspective. Renewable and Sustainable Energy Reviews 42, 446–459.
[3] Imtiaz Ali, Krishnan Jayaraman, Debes Bhattacharyya, 2014. Implications of fiber characteristics and mat densification on permeability, compaction and properties of kenaf fiber panels. Industrial Crops and Products 61. 293–302.
[4] Qatu MS. Application of kenaf-based natural fiber composites in the automotive industry. In: Proceedings of SAE 2011 world congress and exhibition. Detroit; April, 2011. (paper in proceedings)
[5] Shinji Ochi., 2008. Mechanical properties of kenaf fibers and kenaf/PLA composites. Mechanics of Materials 40, 446–452.
[6] Y.A. El-Shekeil, S.M. Sapuan, M. Jawaid, O.M. Al-Shuja'a. Influence of fiber content on mechanical, morphological and thermal properties of kenaf fibers reinforced poly(vinyl chloride)/thermoplastic polyurethane poly-blend composites. Materials and Design 58 (2014) 130–135.
[7] Sgriccia N, Hawley MC, Misra M. Characterization of natural fiber surfaces and natural fiber composites. Compos – Part A: Appl Sci Manuf 2008; 39: 1632–7.
[8] ASTM D 3039-10:2010. Standard test method for tensile properties of polymer matrix composite materials.
[9] ASTM D 790: 2010. Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials.
[10] Khan GMA, Terano M, Gafur MA, et al. Studies on themechanical properties of woven jute fabric reinforcedpoly(l-lactic acid) composites. J King Saud Univ – EngSci 2013.
[11] R Yahaya, SM Sapuan, M Jawaid, Z Leman andES Zainudin, (2014). Mechanical performance of wovenkenaf-Kevlar hybrid composites. Journal of Reinforced Plastics and Composites published online 7 November 2014.

Exploration on Compatibilizing Effect of Non-Ionic, Anionic and Cationic Surfactants on Mechanical Properties of High Density Polyethylene/Low Density Polyethylene/Cellulose Biocomposites

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Abstract. In this study, three types of surfactants specifically cationic, anionic and non-ionic at different weight percentages were added into high density polyethylene/low density polyethylene/cellulose (HDPE/LDPE/cellulose) biocomposites via melt mixing. The cationic, anionic and non-ionic surfactants that used were hexadecyltrimethylammonium bromide (HTAB) and sodium stearate (SS) at 4 to 20 wt.%, whereas sorbitan monostearate (SM) at 1 to 5 wt.%, respectively. The mechanical testing results exhibited that the addition of HTAB increasedtensile stress and modulus, while SS deterioratedmechanical properties, whilst SM increased impact strength and tensile extension of the biocomposites. Based on the mechanical properties results, optimum weight percentages of HTAB and SM were 12 wt.% and 4 wt.%, respectively. The compatibilizing effect was found in HTAB, whereas SM did not show compatibilizing effect but instead plasticizing effect. However, neither compatibilizing nor plasticizing effect was exhibited by SS.

Keyword: high density polyethylene, cellulose, biocomposite, surfactant, hexadecyltrimethylammonium bromide, sodium stearate, sorbitan monostrearate

Introduction

Numerous studies have been done in finding way to improve the incompatibility between natural fillers and polymer matrices of the biocomposites. This is importantas the mechanicalproperties of the biocomposites will improve as well. The polarity difference of the natural fillers and polymer matrices has led to poor interfacial adhesion between them. Based on their chemical structure, PE is non-polar and hydrophobic [1] whereas cellulose are naturally polar and hydrophilic [2]. However, recent researchers had used surface active agents (surfactants) as compatibilizers for polymer composite and blend systems [3-4]. The amphiphilic properties of the surfactants have enabled it to compatibilize between the polar natural filler and the non-polar polymer matrix [5]. The surfactants used in this research are hexadecyltrimethylammonium bromide, HTAB (cationic), sodium stearate, SS (anionic) and sorbitan monostearate, SM (non-ionic). The selection of these surfactants are based on their similarity which, for instance, their structure consisted of single long alkyl chain (C_{16} to C_{17}), environmentally friendly, low toxicity and most

importantly they are not decompose during melt processing of PE. The chemical structure of HDPE/LDPE, cellulose and surfactants are showed in Fig. 1.
In this research, the main objectives are to explore the compatibilizing effect of the surfactants (cationic, anionic and non-ionic) on the mechanical properties of the HDPE/LDPE/cellulose biocomposites. Besides, these three types of surfactants that have been used in the prepared biocomposites were also studied and compared.

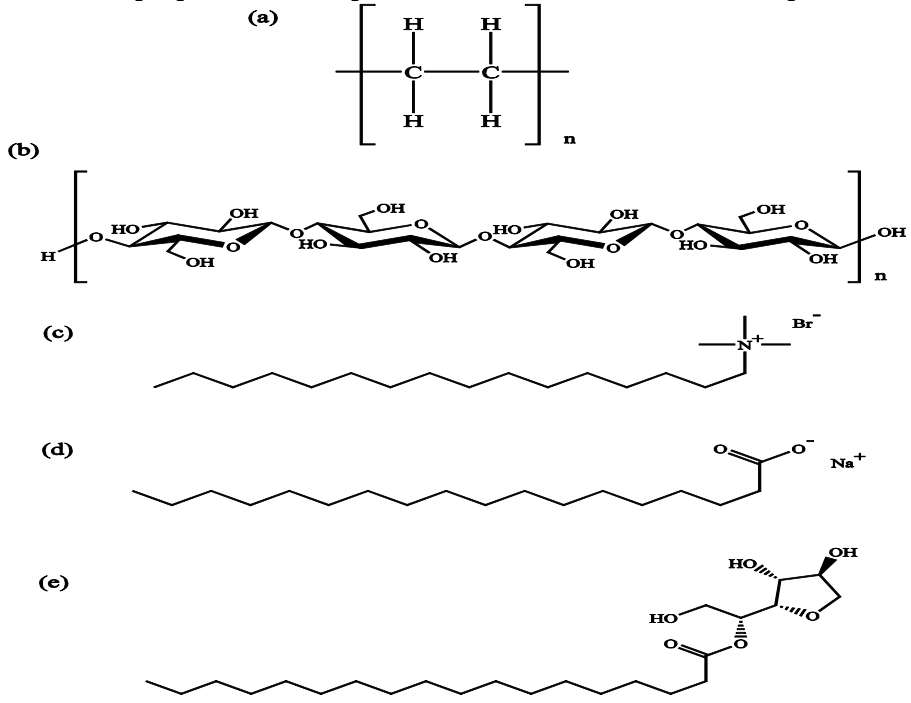

Figure 1. Chemical structures of (a) HDPE/LDPE, (b) cellulose, (c) HTAB, (d) SS, and (e) SM surfactants.

Materials and Methods

Materials

Cellulose microcrystalline powder in size of 20 µm was procured from Sigma-Aldrich Malaysia Sdn. Bhd. High density polyethylene, HDPE (Etilinas HD5740UA) was purchased from Polyethylene Malaysia Sdn. Bhd. Low density polyethylene, LDPE (Titanlene LDC800YY) was bought from Lotte Chemical Titan Malaysia Sdn. Bhd. Three different types of surfactants that were acquired from Sigma-Aldrich Malaysia Sdn. Bhd. are hexadecyltrimethylammonium bromide, HTAB (≥99%), sodium stearate, SS (≥88%) and sorbitan monostearate, SM (≥98%).

Methods

Preparation of HDPE/LDPE/cellulose Biocomposites

The biocomposites were prepared via melt mixing process by using Brabender internal mixer equipped with real-time processing torque recorder. The temperature, rotor speed and time duration for melt mixing process were set at 150ºC, 60 r.p.m and 12 min respectively. The composition of premixed HDPE/LDPE and cellulose was

fixed at 60/40 weight ratio. The weight percentages of HTAB and SS were varied from 4, 8, 12, 16 and 20 wt.%, whereas SM was varied from 1, 2, 3, 4 and 5 wt.% relative to the content of biocomposites. The biocomposite compounds obtained from internal mixer were molded into 1 mm sheet by using hot press machine. The compression molding procedures involved preheating of the biocomposite compounds at 150ºC for 7 min, and subsequently compressed for 2 min at the same temperature before they were cooled under pressure for 5 min. The sheet of HDPE/LDPE/cellulose biocomposite without addition of surfactants was also prepared for comparison purpose. The weight proportion of HDPE/LDPE/cellulose biocomposites without and with addition of HTAB, SS and SM surfactants are exhibited in Table 1.

Table 1: The weight proportion of HDPE/LDPE/cellulose biocomposites without and with addition of HTAB, SS and SM.

Sample	[HDPE/LDPE]/cellulose (60/40)	Surfactant	Weight proportion (wt.%)
PE/Cell	100	-	0
PE/Cell/HTAB4	96	HTAB	4
PE/Cell/HTAB8	92	HTAB	8
PE/Cell/HTAB12	88	HTAB	12
PE/Cell/HTAB16	84	HTAB	16
PE/Cell/HTAB20	80	HTAB	20
PE/Cell/SS4	96	SS	4
PE/Cell/SS8	92	SS	8
PE/Cell/SS12	88	SS	12
PE/Cell/SS16	84	SS	16
PE/Cell/SS20	80	SS	20
PE/Cell/SM1	99	SM	1
PE/Cell/SM2	98	SM	2
PE/Cell/SM3	97	SM	3
PE/Cell/SM4	96	SM	4
PE/Cell/SM5	95	SM	5

Mechanical Testing

The impact strength of the biocomposites was determined via Izod impact test according to ASTM D256 using CEAST impact testing machine (model 9050). The biocomposite samples were cut by using Makita scroll saw machine (model SJ401) into the form of rectangular shape with dimension of $60 \times 12.7 \times 1.0$ mm^3. They were notched for 1 mm depth by using CEAST notch machine (Notchvis). 0.5 J pendulum was used to conduct the test.Tensile test was carried out based on ASTM D638 using Instron universal testing machine (model 5567) equipped with a 30 kN load cell at room temperature (25ºC). The biocomposite samples were cut into a dumbbell shape (Type V). The initial the crosshead speed and gauge length were fixed to 50 mm and 5 mm min^{-1}, respectively.

Results and discussion

Mechanical Properties

The mechanical properties results of the HDPE/LDPE/cellulose biocomposites with addition of HTAB, SS and SM are displayed in Fig. 2, 3 and 4, respectively. In Fig. 2a and 2b, tensile stress and modulus of the biocomposites has increased from PE/Cell/HTAB4 to PE/Cell/HTAB12 which implied that the addition of HTAB from 4 to 12 wt.% has enhanced the compatibility between the HDPE/LDPE and cellulose [6,7]. However, the tensile stress and modulus of the biocomposites were continuously decreased with addition of 16 and 20 wt.% HTAB. The decrease was occurred as an effect of surfactant oversaturation in the biocomposite systems which caused the HTAB to form aggregates and consequently decreased their mechanical properties as well [3]. On the other hand, in Fig. 2c and 2d, the tensile extension and impact strength were continuously decreased with addition of HTAB. Presumably, this is due to the escalating amount of crystal lattice of HTAB in the biocomposite systems, causing increment in the brittleness of the biocomposites **[3]**. Additionally, the increases of tensile stress and modulus indicated that the HTAB is able to act as a compatibilizing agent to the biocomposites. On top of that, the addition of 12 wt.% HTAB was seen to give optimum compatibilizing effect on the HDPE/LDPE/cellulose biocomposites. This is owing to the fact that the PE/Cell/HTAB12 possessed highest tensile stress and modulus compared to the other biocomposites added with HTAB, which is 3.27 MPa and 293.47 MPa, respectively. The tensile stress of PE/Cell/HTAB12 was improved by 25% while its modulus was improved by 25%.

In Fig. 3a to 3d, the impact strength, tensile extension, tensile stress and modulus have showed decreasing trend after HDPE/LDPE/cellulose biocomposites added with SS. These showed that the applied tensile force and impact energy on the biocomposites were unable to be transferred from HDPE/LDPE to cellulose. The deterioration of mechanical properties is an indication of unimproved interfacial adhesion between the HDPE/LDPE and cellulose. This implied the inability of SS to provide compatibilizing effect on the biocomposite components. Therefore, SS is not suitable to be used as a compatibilizer for the HDPE/LDPE/cellulose biocomposites.

On the other hand, the tensile stress and modulus of SM added HDPE/LDPE/cellulose biocomposites are showed in Fig. 4a and 4b. The addition of SM has caused the tensile stress and modulus of the biocomposites to decrease continuously. This exhibited that the addition of SM has softened the biocomposites and thus, deteriorated their ability to withstand the exerted tensile force. In Fig 4c and 4d, the tensile extension and impact strength of the biocomposite were increased from 1 to 4 wt.% SM,indicating the ability of SM to plasticize the biocomposites. Besides that, the increasing trend of impact strength signified the improvement of impact energy transfer in the biocomposites added with SM. Based on these results, it seemed that the addition of SM has provided plasticizing effect on the HDPE/LDPE/cellulose biocomposites. PE/Cell/SM4 has the highest tensile extension and impact strength, which both are increased by 21% and 25%, respectively. However the tensile extension and impact strength was decrease after the addition of 5 wt.% SM. This is

maybe due to oversaturation of SM in the biocomposite which caused accumulation to occur and thus, forming a mild tough failure concentrator in the biocomposites [3].

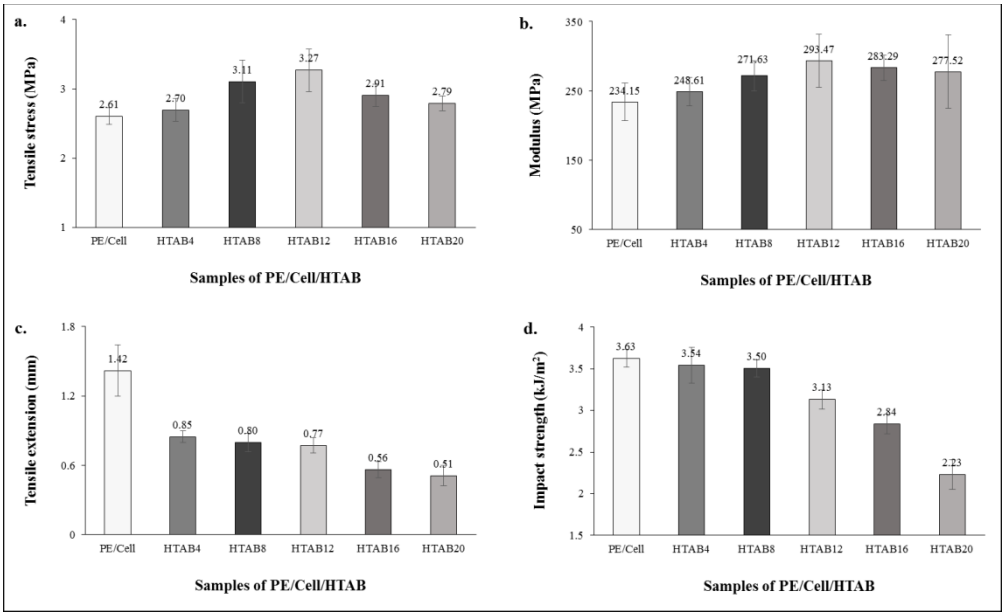

Figure 2. Mechanical properties of HDPE/LDPE/cellulose biocomposites with different weight percentages of HTAB.

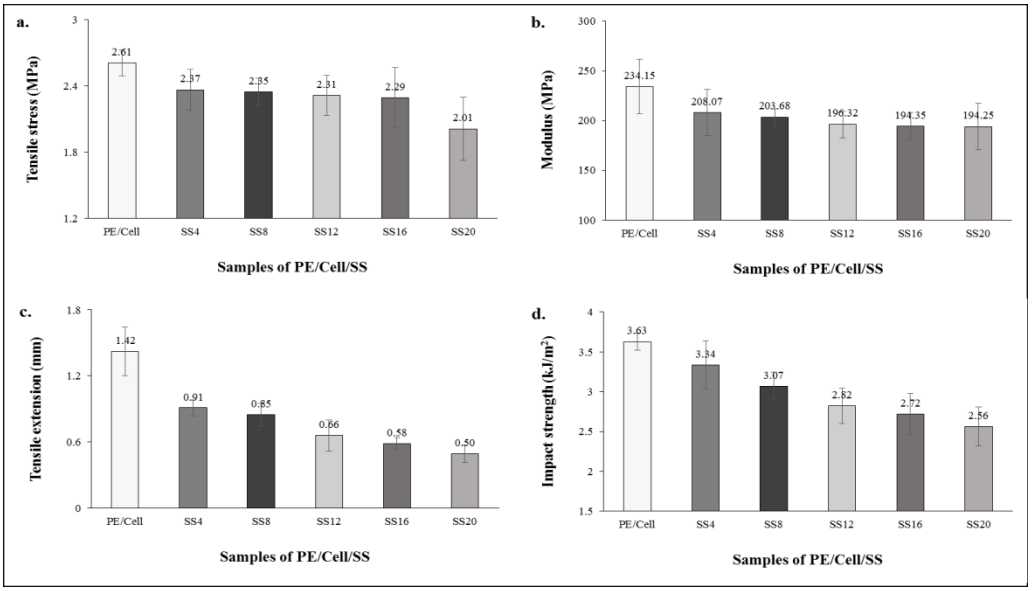

Figure 3. Mechanical properties of HDPE/LDPE/cellulose biocomposites with different weight percentages of SS.

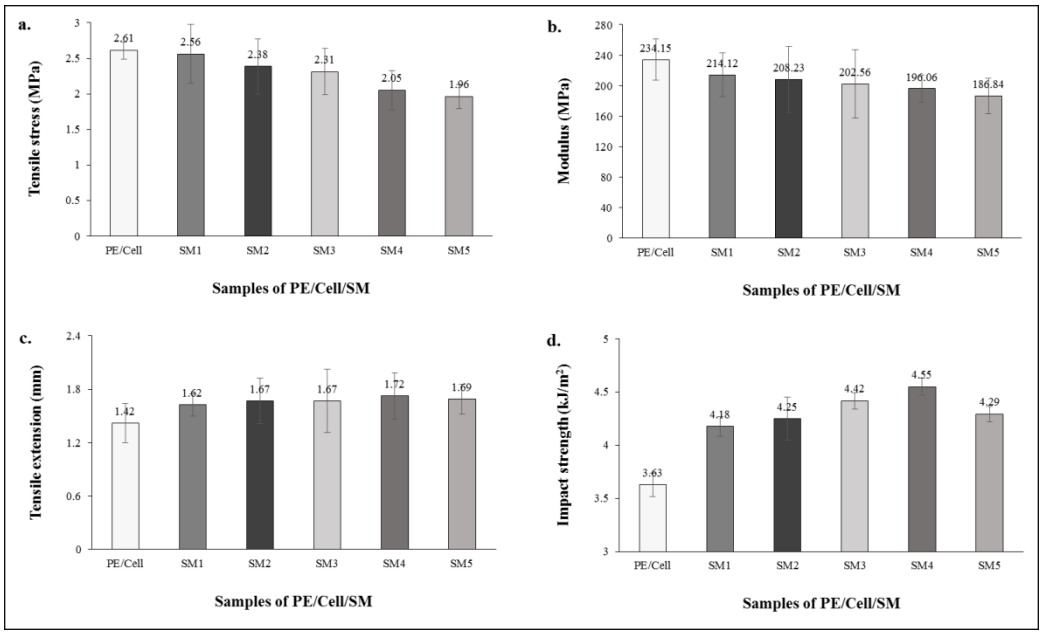

Figure 4. Mechanical properties of HDPE/LDPE/cellulose biocomposites with different weight percentages of SM.

Conclusion

This study showed that the addition of HTAB has improved the tensile stress and modulus, indicating improvement in compatibility between HDPE/LDPE and cellulose. However, the mechanical properties of the biocomposites were deteriorated with addition of SS which implied the inability of the applied tensile and impact force to be transferred from HDPE/LDPE matrix to cellulose filler. Besides that, SM has improved the impact strength and tensile extension of the biocomposites. This exhibited that the addition of SM has plasticized the biocomposites. Referring to the mechanical properties results, the optimum weight percentages of HTAB is 12 wt.% due to provision of the best compatibilizing effect compared to other weight percentage of HTAB. On the other hand, 4 wt.% is the optimum weight percentage of SM as PE/Cell/SM4 showed the highest improvement in impact strength and tensile extension in comparison with other SM added biocomposites. Therefore, the addition of HTAB was found to provide compatibilizing effect whilst SM showed plasticizing effect instead of compatibilizing effecton the biocomposites. However, neither compatibilizing nor plasticizing effect was exhibited by SS.

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References

[1] M. Tajvidi, and A. Takemura, Effect of fiber content and type, compatibilizer, and heating rate on thermogravimetric properties of natural fiber high density polyethylene composites, *Polymer Composites* Vol. 30, 2009, pp. 1226-1233.

[2] N. Rajini, J. T. W. Jappes, P. Jeyaraj, S. Rajakarunakaran, and C. Bennet, Effect of montmorillonite nanoclay on temperature dependence mechanical properties of naturallywoven coconut sheath/polyester composite, *Journal of Reinforced Plastic and Composites* Vol. 32, 2013, pp. 811-822.

[3] A. A. Shamsuri, R. Daik, E. S. Zainudin, and P. M. Tahir,Compatibilization of HDPE/agar biocomposites with eutectic-based ionic liquid containing surfactant,*Journal of Reinforced Plastic and Composites*, Vol. 33, 2014, pp. 440-454.

[4] M. N. Islam and M. S. Islam, Mechanical properties of chemically treated sawdust-reinforced recycled polyethylene composites, *Industrial & Engineering Chemistry Research*, Vol. 50, 2011, pp. 11124-11129.

[5] K. Dzulkefly, H. F. Khoh, F. B. H. Ahmad, S. A. Ahmad, and W. H. Lim, Solvent-free esterification process for the synthesis of glucose bolaform surfactants, *Oriental Journal of Chemistry*, Vol. 26, 2010, pp. 747-752.

[6] A. A. Shamsuri, M. K. A. Azid, A. H. M. Ariff, A. K. Sudari, Influence of surface treatment on tensile properties of low-density polyethylene/cellulose woven biocomposites: a preliminary study, *Polymers*, Vol. 6, 2014, pp. 2345-2356.

[7] P. Skalková, A. Čížová, D. Jochec Mošková, and I. Janigová, LDPE/glucuronoxylan blends: preparation and study of thermal and mechanical properties, *Cellulose Chemistry and Technology*, Vol. 46, 2012, pp. 69-77.

Assessment of Chloride ion Penetration and Water Permeability Properties of RubberisedFibre Mortar

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Abstract. Disposal of worn automobile tyres is one of the many environmental degradation problemsworldwide. In Malaysia more than 50,000 tons of worn tyres are generated annually and on increasing.This paperhighlight the laboratory investigation of incorporation of treated crumb rubber tyre aggregate(TCR) as a partial replacement to fine aggregate (FA) and the inclusion of oil palm fruit fibre (OPFF) asanadditive by the weight of cement to produce rubberised fibre mortar. Durability properties such aschloride ion penetration resistance and water permeability under hydrostatic pressure were measured.Results obtained indicated good practical significance due to the increase in the resistance to chloride ionpenetration and depth of water penetration at 1.0%OPFF for every 10%, 20% and 30% TCRreplacements. This implies that rubberised cement fibre mortar can be used where moderate resistance tochloride ion penetration and low permeability condition is suggested. The product may not besuitablefor liquid containment barriers due to the relatively high pore structure of the material.

Keywords: Treated crumb rubber (TCR), Oil Palm fruit fibre (OPFF), chloride ion penetration resistance, water permeability depth, Rubberised Fibre Mortar, microstructure.

Introduction

In Malaysia more than 50,000 tons of worn automobile tyres are generated annually, and thisfigure has increased over the years with the increase in vehicular traffic. Unfortunately, about 60% of itare unaccounted in standard disposal method causing illegal dumping at dump sites[1]. The discardedtyres can be recycle into many different sizes of rubber particles and use in other industries such asgeotechnical works, road construction, as aggregate in cement based products, fuel in cement kilns andincinerators for production of electricity. One of the rubber particles are refered as

crumb rubber (CR)which have similar sizes with coarse and fine aggregates in concrete. Many researches had shown that
used of these particles as coarse aggregates will not do any improvement on concrete. The use of CR ingranular and powdered form was to minimize the loss in mechanical properties due to the tyre rubberincorporation which has been studied worldwide [2]. Hence this research focus is on fine aggregatesreplacement.Apart from thatMalaysia also produces 18million tons of oil palms per year, which at the sametime create millions of oil palm fruit fibre waste. The oil palm fruit fibre (OPFF) is suitable to be addedin concrete as shrinkage control materials. Hence OPFF was added in this mortar mixes as to improvethe mechanical and shrinkage properties [3]. Both materials OPFF and CR were combined in mortar asadditive and fine aggregate (FA) replacement respectively, to study their effect on the mechanicalproperties.

Experimental programme

The present study concerns with fine aggregates replacement of 10%, 20% and 30% treatedcrumb rubber (TCR) and 0.5% , 1.0% and 1.5% of OPFF addition in cement based matrix. Thespecimens were then subjected at different curing conditions for 28 days before assessment were madeof the durability properties which are resistance to chloride ion penetration and water permeability. Intotal, 192 number of specimens were tested and the results are shown in the following section.Rubberised fibre mortar specimens were produced and chloride ion penetration resistance and waterpermeability tests were conducted in accordance with standard codes of civil engineering laboratorypractice.

Materials

In this study, the mortar mixes were produced using Ordinary Portland composite cement CEMII/B-M (V-L) 32.5R conforming to ASTM C150 [4], stone dust with maximum particle size passing a4.75mm sieve in conformity with ASTM C33 [5], and granulated TRC with particles sizes of 0.15 –2.36mm with compacted density and fineness modulus of 668Kg/m3 and 0.9, respectively. While theOPFF was obtained from Seri Ulu Langat Palm Oil Mill Sdn. Bhd at Dengkil, Malaysia. Inensuring good bonding between granulated TCR and other materials, the earlier was treated with cementto create surface roughness/friction on its surfaces. Cement was choosen because it can play better effectover other chemicals (hydrochloric acid, sodium hydroxide etc) which could be detrimental to themortar/concrete matrix.

Mix proportions

The mortar mixes were made of cement to fine aggregate ratio of 1:2.75 in conformity withhydraulic cement mortar requirements with water to cement ratio of 0.458. [6]. The fine aggregate waspartially replaced with 10%, 20% and 30% of TCR while the OPFF was added at 0.5%, 1.0% and 1.5%by volume of mortar mix. In total, 16 different mortar mixes were prepared of which 96 number of100mm diameter by 50mm thick cylinders and another 96 number of 150mm cubes were produced.

Mixing and curing procedures

In order to prevent balling effect associated with the conventional mixing technique, the drymaterials cement, fine aggregate, and treated TCR aggregate were first mixed for 2 mins in the bowlmixer. Then OPFF and one - third of water were added and further mixed for 2 mins, gradually theremaining water was added and mixing continued for about 5mins until a homogeneous mix wasachieved. After that, 96 numbers of 150 mm cubes and 100mm diameter by 50mm thick cylinders eachwere prepared totaling 192 samples and half was subjected to water ponding (Pw) curing while the otherhalf on water sprinkling (SPW) curing for 28 days. The water ponding curing (Pw) was achieved byimmersing a day old specimen in water at a temperature of 23- 270C with about 100% relative humidity(RH). On the other hand, water sprinkling (SPW) was by covering other day old specimens with aburlap sack under a shade and water sprinkled on it twice daily (maintaining free and sufficientcirculation of air around specimens).

Test methods

The standards for test methods adopted in this study refer to a condition of a tropical temperature rangeof 28 – 300C, except were modifications were required as appropriately stated. The water permeabilityand Chloride ion penetration resistance tests were conducted in accordance with Civil engineeringstandard laboratory practices.

Water permeability depth

Water penetration depth was investigated according to BS 12390-8 [7]. Immediately after demoulding ofthe rubberised mortar cubes , the testing face was roughened and cured by either of the curing method(Pw and SPw) Specimens were then oven dried at 110 ± 50C until a constant mass was attained andrecorded as initial mass in kilogram (Kg) before testing. The specimen was coupled to the apparatus andthe roughened face subjected to a hydrostatic pressure of 0.5MPa for 72hours. Mass of the specimen wastaken immediately after testing before splitting specimen into two halves to record the depth of waterpenetration.

Chloride-ion penetration resistance:
Rapid chloride permeability test (RCPT)

The amount of electrical current passed through a 28days cured 100ı ı Ø ıı 50ıı thick cylindrical specimens were investigated for chloride ion penetration resistance in accordance with ASTM C1202 [8] specification. The PROVE'it Rapid Chloride Permeability Tester (RCPT) of German Instruments (Fig.1)was used for this purpose. During the test, one end of the specimen was immersed in sodium chloride(3.0% NaCl) and the other in sodium hydroxide (0.3 N NaOH) solution to maintain a potentialdifference of 60V dc across the specimen. The total charge passed at 6 hours in coulombs (which isrelated to the resistance of the specimen to chloride ion penetration) was found.

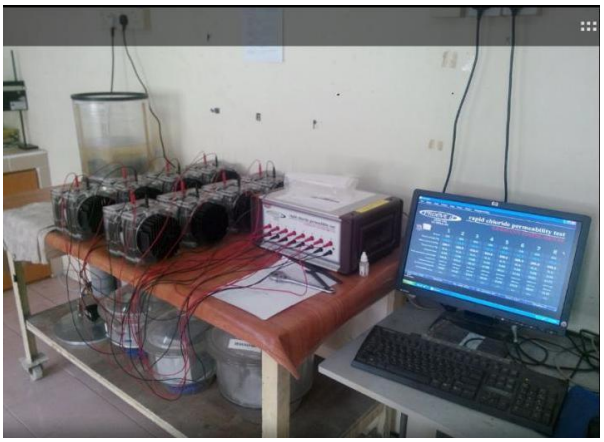

Fig.1.Rapid chloride permeability test (RCPT) setup

Results and discussion

Influence of TCR and OPFF contents in the improvement of chloride ion penetration resistanceis shown in Figure 2. Penetration of the chloride ion was reduced with increase in the OPFF and TCR inthe cement based matrix for specimens cured by Pw and SPW. The reduction in OPFF percentage 0.5%,1.0% and 1.5% (F0.5, F1.0 and F1.5) to TCR 0%, 10%, 20% and 30% (CR0-30) were 11%, 18% and27% respectively. However, at 0% OPFF addition for every treated crumb rubber replacement of0%, 10%, 20% and 30% (F0CR0-30) respectively there was a decrease in chloride ion penetrationresistance with increase in TCR. It is pertinent to note, however the highest percentage increase was 27%at F1.5CR0-30 but the optimum percentage of TCR and OPFF choosen is at the 18% reduction(F1.0CR0-30) because the value of charge passed is within the moderate (2000-4000 coulombs) chloridepermeability class according to ASTM C1202 [8]. Results for specimen cured by SPW (Fig.2b) showed asimilar trend to Fig.2 (a) at F1.0CR0-30 but with the chloride permeability class in the range of high tomoderate (2000->4000coulombs) as per [8].

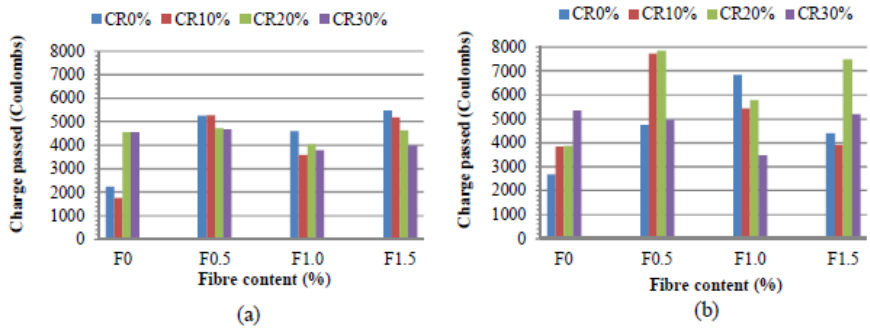

Fig. 2.Chloride ion penetration resistance of specimen cured by (a) water ponding and (b) water sprinkling.

The most effective internal factor in the durability of concrete is its permeability. Somedurability characteristics (corrosion of steel in concrete exposed to aggressive

minerals and oracids, freeze and thaw cycles, carbonation etc) can be reduced or eliminated by controlling thetransport processes “permeability” defined as the property of concrete which measures howfluid flows through concrete under applied hydrostatic preesure.Fig.3(a) shows the result of the permeability depth for Pw cured specimen with anincrease in the permeability value upon increase in the OPFF and TCR aggregate. The valuewas relatively higher at F1.5CR0-30 compared to F0-1.0CR0-30. The optimum decrease in thevalue for permeability depth is at F1.0CR0-20 and classified as medium permeability accordingto DIN 1048 [9]. On the other hand, optimum for SPw specimens (Fig. 3b) is at F0.5CR0-30 andso classified as medium permeability (3-6cm). The increase in permeability depth for samples atdifferent curing methods is due to the reduction in bonding between particles as the degree ofpermeability depends on capillary porosity. Debonding of fibres in the cement matrix alsocreates more pathways for the fluid flow. However, there seem to be capillary discontinuitybased on the results obtained. Typical water depth profile is shown in Fig.4.

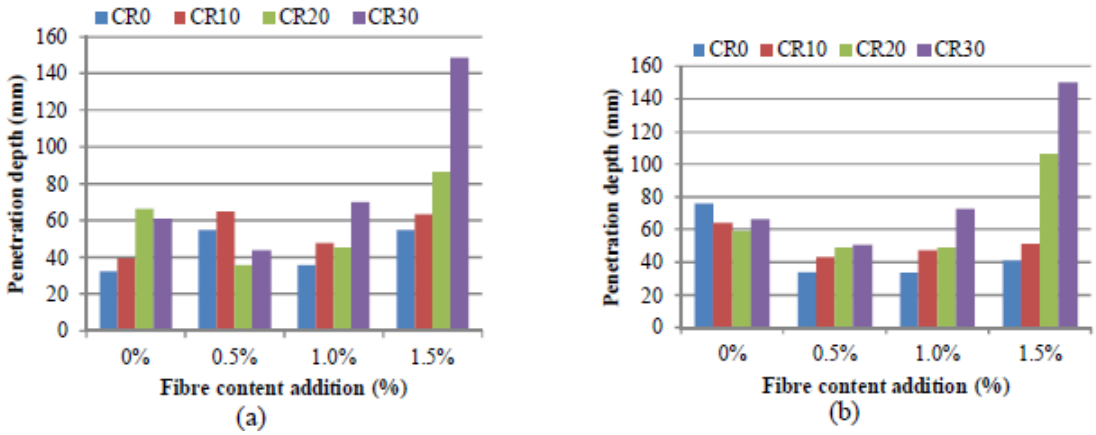

Fig.3.Water penetration depth for specimens cured by (a) water ponding and (b) water sprinkling.

Fig.4.Typical water penetration depth profile of rubberised fibre mortar.

Conclusions

In this study, some durability related properties of rubberised fibre mortar have beeninvestigated. The positive effects in the increase of chloride ion penetration resistance of thecement mortar matrix were observed. This will further reduce the

potential for corrosion ofembedded reinforcement and that is of good practical significance.The increase in permeability depth of the rubberised fibre mortar appeared nonproportionalin both curing conditions compared with control specimens. The primary reasonfor this behaviour cannot be unconnected with the existence of water filled capillaries of thespecimen. This is indeed due to debonding between the TCR particles and cement paste, alsoalong some of the fibres where the interface surface between them (TCR particles, cement pasteand OPFF) serve as the bedding plane and or pathway for the hydrostatic water flow through thecement matrix. Hence, use of TCR aggregate and OPFF increases both chloride ion penetrationand water permeability depth in both curing conditions, but within the moderate and mediumpermeability classes respectively. Hence practically applicable where low chloride ion andpermeability is suggested.

References

[1] S.K. Thiruvangodan, *Waste tyre management in Malaysia*, 2006, Universiti PutraMalaysia.

[2] N.Ali, A. Amos, and M. Roberts. *Use of ground rubber tires in portland cementconcrete*. in *Proceedings of the international conference on concrete*. 2000. London,United Kingdom: Thomas Telford Services Ltd.

[3] M.A. Ismail and H. Hashim. *Palm oil fiber concrete*. in *The 3rd ACF International Conference*. 2008.

[4] B.P. Cement, *Complying with ASTM C150: 1*. Type I. 2.

[5] C33, A., *Standard Specification for Concrete Aggregates*, 2004, ASTM International West Conshohocken, PA.

[6] 109M-05, A.C.C. *Test Method for Compressive strength of Hydraulic Cement Mortars (Using 2-in or [50-mm] Cube Specimens)*. 1994. American Society for Testing and Materials PA, p.

[7] EN, B., *12390-8. Depth of penetration of water under pressure.* British Standards Institution, 2000.

[8] C-05, A., *Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration.* 2005.

[9] Standard, G., *DIN 1048—test methods of concrete impermeability to water: Part 2.* Deutscher Institute Fur Normung, Germany, 1978.

Selecting natural fibers for industrial applications

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Abstract. Natural fiber composites (NFC) have recently emphasized to be potential alternatives for traditional composites in several industrial applications. Such composites show wide desired properties and advantages over traditional ones such as light weights, high specific properties, low cost, ease of manufacturing, recyclability, and degradability characteristics. The final product features of such composites depend on the integrated characteristics of both matrix and fillers properties. Selecting an appropriate natural fiber type to form an NFC is affected by several factors and criteria. Till now, natural fibers are evaluated regarding limited number of criteria. Results demonstrate that better evaluation of natural fibers regarding wide range of criteria will lead to better decisions regarding selecting the suitable NFC for industrial applications and enhance achieving better performance. Such evaluations should consider combined economic and environmental characteristics as well as technical ones. New potential fiber types can be discovered and utilized through better evaluations using combined desired criteria.

Introduction

Proper material selection became crucial process in engineering to achieve both successful sustainable designs and customer satisfaction attributes (AL-Oqla and Sapuan 2013). Implementing new materials as well as bio-composites in a specific industrial sector is limited by several constrains and factors (Dweiri and Al-Oqla 2006, AL-Oqla and Sapuan 2013). Therefore, selecting the most suitable material type for a particular application is a complex matter where proper decisions have to be taken utilizing the pairwise comparisons which is the base of decision making process in different engineering applications (Dweiri and Al-Oqla 2006, AL-Oqla and Hayajneh 2007, Dalalah *et al.* 2010, Al-Widyan and Al-Oqla 2011, Al-Oqla and Omar 2012, Al-Widyan and Al-Oqla 2014).NFC became recently highly valuable type of echo-friendly cheap alternative materials (Sapuan *et al.* 2013). Such materials have been emerged in different applications because of their desired properties (Kalia *et al.* 2011, Pilla 2011, AL-Oqla and Sapuan 2013). Several investigations regarding NFC competitiveness and capabilities as alternative materials had been conducted (Alawar *et al.* 2009, Mir *et al.* 2010, Abdal-hay *et al.* 2012, Sapuan *et al.* 2013). The NFC

finalproducts' performance and properties strongly depend on the properties of their individual constituencies as well as the polymer/filler interfacial characteristics. Based on literature; proper evaluation of NFC composites for industrial applications is not enough discussed regarding wide range of desired criteria. Thence, further comparisons between the NFC constituents (fillers and matrices) are needed in an extensive manner regarding wide range of desired criteria and factors that affect their selection in different applications to end up with proper consistent informative selection decisions.

Consequently, this work aims to elaborate more desired characteristics that designers have to take into consideration regarding selecting NFC materials and their constituents to achieve better performance to their designs. Also, to conduct some pairwise comparisons between different fiber types regarding some selective criteria to emphasize the need for better NFC evaluations regarding further criteria.

Methods

To enhance better selection of NFC materials, criteria affect the selection of natural fibers were suggested, collected from published literature and tabulated. To illustrate the effectiveness of such desired criteria on the selection process, pairwise comparisons between different types of fibers were conducted. Each comparison with respect to each selected single criterion was conducted and interpreted in a separate figure.

Results and Discussion

The complete NFC attributes and capabilities depend on the physical and chemical composition of the inherent material. Therefore, NFC materials should be investigated regarding many different features and properties before being considered to be used in any particular application. Such considerable criteria of selecting the natural fiber composite products can be according to Natural Fiber Properties (NFP), Polymer Base Properties (PBP), Composite Characteristics (CC), and Composite Performance (CP) (AL-Oqla and Sapuan 2013), where different attributes and characteristics have to be investigated and taken into account to reach better evaluations of the NFC materials like physical, mechanical, chemical, biological, and environmental properties as well as toxicity, bio-stability, life cycle, durability, bio-degradability, weather resistance, occupational health and safety others. Criteria that affect selection of natural fibers are shown in Figure 1. Such criteria can be the key driver for the industry to enhance selection the most appropriate NFC materials for a given application through better selection of the composite constituents.

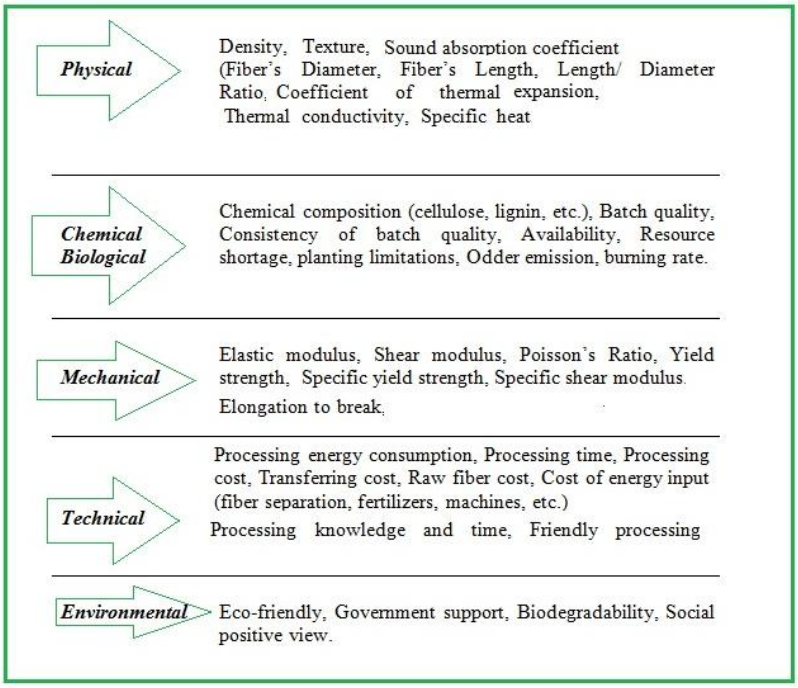

Figure 1: Criteria affect the selection of the natural fibers for NFC materials. (reproduced from (AL-Oqla and Sapuan 2013))

Comparisons between different natural fiber types namely Coir, Date Palm, Hemp, Sisal, Flax and Jute are conducted regarding their specific modulus of elasticity and specific strength as shown in Figure 2. It can be clearly shown that natural fibers vary in their physical and mechanical properties. These comparisons actually combined the fibers' modulus of elasticity and specific strength relative to their densities to give more relative information about different fiber types. It is clear that Flax, Hemp and Jute are the best regarding these criteria (specific modulus and specific strength). But further investigations regarding wider criteria rather than physical and mechanical properties are needed to make more realistic informative decisions regarding the best fiber type. That is; further criteria like cost will change the priority of the previous three fiber types as seen in Figure 3 where the specific strength to the cost ratio comparisons were conducted. It is obvious that Date palm fibers are the best type among all other types if further economic criteria are considered. It can be seen that Date Palm fiber is about four times better than Jute.

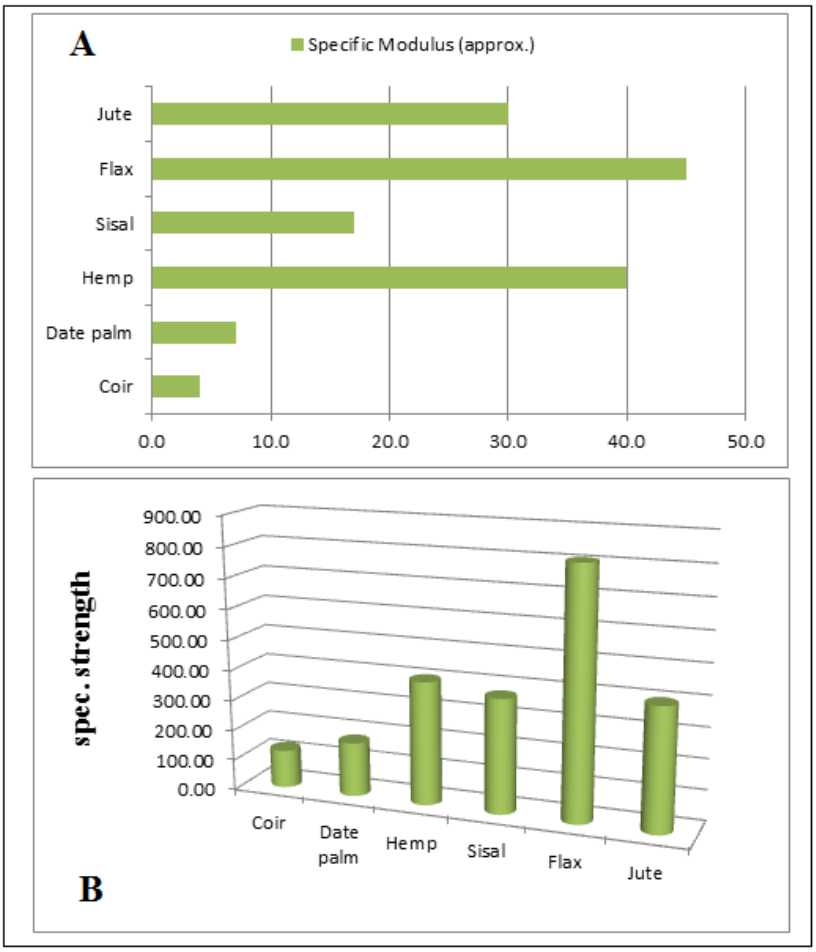

Figure 2: Comparison of different natural fiber types regarding specific strength (A) and specific modulus (B).

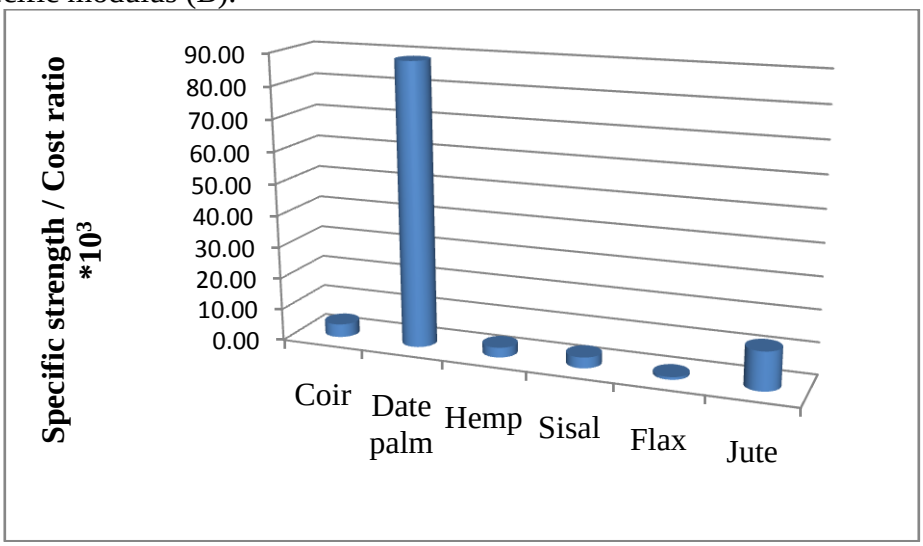

Figure 3: Comparison of different natural fiber types regarding combined specific strength to cost ratio criterion.

Conclusions

Selecting an appropriate natural fiber type to form an NFC is affected by several criteria and considered as a multi-criteria decision making problem. Better evaluations of natural fibers regarding wide range criteria have several advantages. It can lead to better informative decisions regarding selecting the suitable NFC for industrial applications on one hand and enhance achieving better performance on the other. Evaluations of NFC constituents should consider combined economic and environmental characteristics as well as technical ones to achieve more reasonable confident decisions. Moreover, new potential fiber types can be discovered and utilized through better evaluations using wider desired criteria.

References

[1] Abdal-Hay, A., Suardana, N.P.G., Jung, D.Y., Choi, K.-S. & Lim, J.K., 2012. Effect of diameters and alkali treatment on the tensile properties of date palm fiber reinforced epoxy composites. *International Journal of Precision Engineering and Manufacturing,* 13 (7), 1199-1206.

[2] AL-Oqla, F. M., & Hayajneh, M. T., 2007. A design decision-making support model for selecting suitable product color to increase probability. In Design challenge conference: Managing creativity, innovation, and entrepreneurship.

[3] Al-Oqla, F.M. & Omar, A.A., 2012. A decision-making model for selecting the gsm mobile phone antenna in the design phase to increase over all performance. *Progress In Electromagnetics Research C,* 25, 249-269.

[4] Al-Oqla, F.M. & Sapuan, S., 2013. Natural fiber reinforced polymer composites in industrial applications: Feasibility of date palm fibers for sustainable automotive industry. *Journal of Cleaner Production*.

[5] Al-Widyan, M.I. & Al-Oqla, F.M., 2011. Utilization of supplementary energy sources for cooling in hot arid regions via decision-making model. *International Journal of Engineering Research and Applications,* 1 (4), 1610-1622.

[6] Al-Widyan, M.I. & Al-Oqla, F.M., 2014. Selecting the most appropriate corrective actions for energy saving in existing buildings a/c in hot arid regions. *Building Simulation: An International Journal*. DOI 10.1007/s12273-013-0170-3

[7] Alawar, A., Hamed, A.M. & Al-Kaabi, K., 2009. Characterization of treated date palm tree fiber as composite reinforcement. *Composites Part B: Engineering,* 40 (7), 601-606.

[8] Dalalah, D., Al-Oqla, F. & Hayajneh, M., 2010. Application of the analytic hierarchy process (ahp) in multi-criteria analysis of the selection of cranes. *Jordan Journal of Mechanical and Industrial Engineering, JJMIE,* 4 (5), 567-578.

[9] Dweiri, F. & Al-Oqla, F.M., 2006. Material selection using analytical hierarchy process. *International journal of computer applications in technology,* 26 (4), 182-189.

[10] Kalia, S., Kaith, B. & Kaur, I., 2011. *Cellulose fibers: Bio-and nano-polymer composites: Green chemistry and technology* Heidelberg: Springer.

[11] Mir, A., Zitoune, R., Collombet, F. & Bezzazi, B., 2010. Study of mechanical and thermomechanical properties of jute/epoxy composite laminate. *Journal of Reinforced Plastics and Composites,* 29 (11), 1669-1680.

[12] Pilla, S., 2011. *Handbook of bioplastics and biocomposites engineering applications* Massachusetts: Scrivener Publishing.
[13] Sapuan, S., Pua, F.-L., El-Shekeil, Y. & Al-Oqla, F.M., 2013. Mechanical properties of soil buried kenaf fibre reinforced thermoplastic polyurethane composites. *Materials & Design*, 50, 467–470.

Selecting Proper Alternatives of Natural Fiber Composites

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Abstract. Selecting an appropriate reinforcement condition for natural fiber composites can dramatically enhance achieving better low-cost sustainable design possibilities. Several factors affect acquiring such reinforcement conditions which make it a matter of multi-criteria decision making (MCDM) problem. This work was able to build and implement DM models in the field of NFCs to optimize reinforcement conditions for the first time. Here, AHP was utilized to achieve the optimal reinforcement condition of the Date Palm/Epoxy composite to maximize its overall tensile property considering combined evaluation criteria.

Keywords: Polymer-matrix composites; Mechanical properties; NFC selection AHP.

Introduction

It is necessary to select the proper materials as this selection became crucial process in engineering to achieve both successful sustainable designs and customer satisfaction attributes and to contribute achieving successful low cost design that can enhance sustainability as well as customer satisfactions. Therefore, there is a need to develop and assist producing green products with lower prices to be able to widen their usage in order to achieve real sustainable societies. Such implementations of new echo-friendly products require attaining desirable characteristics and properties to be able to achieve their functional requirements. Implementing a specific material type in a particular industry is restricted by several criteria and constrains [1-3], where different performance parameters have to be investigated to ensure the suitability of such material for a particular application. Therefore, the selection of the most appropriate material type for a particular application is deemed as a multi criteria decision making (MCDM) problem where accurate and keen decisions have to be taken to ensure the technical suitability of such material utilizing appropriate decision making tools [1, 4].

Technically speaking, NFC final products' performance and properties strongly depend on the characteristics of their individual constituencies as well as the polymer/filler interfacial adhesion [5-10]. Functional requirements usually determine the needed characteristics of the component made of composite materials. AL-Oqla

and Sapuan [2] introduced extensive criteria that have to be taken into consideration to select the appropriate NFC materials for a specific application. Authors discussed these criteria and classified them into distinguished levels according to the NFC constituents (fiber and matrix) as well as the features of the NFC final composite itself, in addition to both general and specific composite performance levels to enhance evaluating the technical aspects of the NFC in a fairly optimized manner. Accordingly, one of the most potential natural fiber types that can be utilized in different industrial applications are the Date Palm ones [2, 11]. It can be considered as the best regarding several criteria like cost, availability, specific modulus and strength to cost ratio criteria [2]. On the other hand, one of the most suitable Date Palm / polymer matrix composites is the Date Palm/ Epoxy one where good tensile and mechanical properties can be achieved [12, 13]. Unfortunately, the tensile properties of the Date Palm/ Epoxy (like any other natural fiber/ polymer matrix) are dramatically affected by the reinforcement conditions such as fiber diameter, fiber length, and fiber surface treatment [12-14].

Consequently, decision making models have to be utilized and implemented to select the best reinforcement conditions of such composite considering combined evaluation criteria simultaneously which can contribute to its role in different industrial applications to get more economical benefits. This can also guide the evaluation process by determining the initial possible potential alternatives for decision making models that can be used to optimize the reinforcement conditions of such composites.

Methods

Here, the Analytical Hierarchy Process (AHP) was used in determining the optimal refinement condition of the Date Palm/Epoxy composite to achieve maximum tensile properties. The Analytical Hierarch Process (AHP) is one of the most powerful, popular and flexible decision making methods that can achieve best decisions considering both tangible and intangible aspects and attributes. As mentioned previously, AHP method is preferable over the fuzzy-AHP or any combination of fuzzy-MCDM tool particularly when the data is precisely known and when no subjectivity involved in the problem [15, 16]. Furthermore, the AHP was capable to deal with real life complex and uncertain environment more efficiently than fuzzy judgment [17, 18]. That is; the fuzzy AHP has recently a criticism that its arithmetic operation violates the AHP reciprocal and continuity axioms as well as the operational rule of consistency, which make it questionable for decision making problems [18].

Results and Discussion

In order to reach the reasonable optimal reinforcement condition of the Date Palm/ Epoxy that give reasonable high tensile properties, eleven potential reinforcement conditions that have potential tensile properties were considered. These alternatives are combinations of different levels of fiber diameters, fiber lengths, and different NaOH concentration treatments. The NaOH treatments solution was selected because it was reported as one of the most suitable solution types for date palm fiber to

enhance its mechanical properties [12, 19]. The eleven candidate alternatives with respect to the whole evaluation criteria are tabulated in Table 1.

Table 1: Alternatives and evaluation criteria

Criteria / Alternatives	MTS	MSS	EL
C1	0.068	0.043	0.038
C2	0.187	0.058	0.106
C3	0.158	0.052	0.138
C4	0.068	0.138	0.061
C5	0.088	0.138	0.123
C6	0.085	0.143	0.115
C7	0.079	0.138	0.076
C8	0.076	0.083	0.092
C9	0.07	0.083	0.076
C10	0.07	0.069	0.069
C11	0.05	0.055	0.107

[a]MTS: Maximum Tensile Strength. [b]MSS: Maximum Shear Stress. [c]EL: Elongation to Break.

The main combined evaluation criteria were the Maximum Tensile Strength, Maximum Shear Stress and the Elongation to Break. These criteria can dramatically affect the selection of maximum tensile properties of the Date Palm/ Epoxy composite. The values of the considered criteria used in evaluation were deduced from extensive literature considering single fiber pull out technique with similar experimental conditions.

After forming the considered problem in a hierarchy structure, the AHP method was used to determine the weights of the criteria. In this particular stage, another type of questionnaire was constructed and sent to another group of twelve experts worldwide. The final aggregated assignments were finally gained. The final priorities of all candidate alternatives to the main goal (with respect to the whole criteria that maximize the tensile properties of the desired composite simultaneously) are illustrated in Figure 1. The closeness of some alternatives in the final priorities demonstrates that determining the optimal alternative without using a decision making tool is very difficult task.

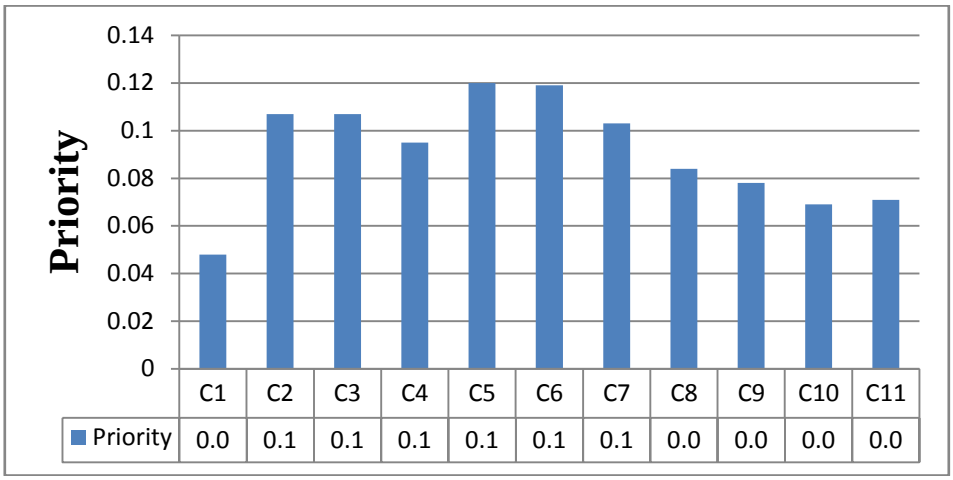

Figure 1: Priorities of all candidate reinforcement conditions with respect to the whole evaluation criteria simultaneously

Conclusions

This study was able to apply MCDM models to optimize the reinforcement conditions of the Date Palm/ Epoxy composite to achieve the best recommended tensile properties. This can enlarge the economic benefit of such composite as well as enhancing its usage in different industrial applications as a cheap ecofriendly alternative type of materials. Implementing DM techniques to select the optimal reinforcement conditions of natural fiber composites were successfully presented for the first time. Therefore, this work can dramatically contribute to the economic production of the Date palm/Epoxy composites as well as assist implementing such techniques to different natural fiber composites to maximize their desired characteristics and hence participate achieving better low-cost sustainable design possibilities.

References

[1] F. Dweiri and F. M. Al-Oqla, *International journal of computer applications in technology*, 26, 182 (2006).

[2] F. M. AL-Oqla and S. Sapuan, *Journal of Cleaner Production*, 66, 347 (2014).

[3] J. Luc Toupe, A. Trokourey, and D. Rodrigue, *Polymer composites*, 35, 730 (2014).

[4] R. Rao and B. Patel, *Materials & Design*, 31, 4738 (2010).

[5] F. M. AL-Oqla and S. M. Sapuan, in "Postgraduate Symposium on Composites Science and Technology 2014 & 4th Postgraduate Seminar on Natural Fibre Composites 2014, 28/01/2014"Ed.^Eds.), Putrajaya, Selangor, Malaysia, Year of Converence.

[6] S. Ojha, G. Raghavendra, and S. Acharya, *Polymer composites*, 35, 180 (2014).

[7] S. M. Sapuan, F.-l. Pua, Y. A. El-Shekeil, and F. M. AL-Oqla, *Materials & Design*, 50, 467 (2013).

[8] D. Nurwaha, W. Han, and X. Wang, *Journal of the Textile Institute*, 104, 419 (2013).
[9] H. Yu and C. Yu, *Journal of the Textile Institute*, 101, 452 (2010).
[10] X. Deng, Y. Hu, Y. Deng, and S. Mahadevan, *Expert Systems with Applications*, 41, 156 (2014).
[11] F. M. AL-Oqla, M. S. Sapuan, M. R. Ishak, and N. A. Aziz, *BioResources*, 9, 4608 (2014).
[12] A. Shalwan and B. Yousif, *Materials & Design*, 53, 928 (2014).
[13] T. Alsaeed, B. Yousif, and H. Ku, *Materials & Design*, 43, 177 (2012).
[14] A. Alawar, A. M. Hamed, and K. Al-Kaabi, *Composites Part B: Engineering*, 40, 601 (2009).
[15] R. V. Rao, "Decision making in the manufacturing environment: using graph theory and fuzzy multiple attribute decision making methods", Springer, 2013.
[16] T. L. Saaty and J. S. Shang, *European journal of operational research*, 214, 703 (2011).
[17] T. L. Saaty and L. T. Tran, *Mathematical and Computer Modelling*, 46, 962 (2007).
[18] K. Zhü, *European journal of operational research*, 236, 209 (2014).
[19] A. Abdal-hay, N. P. G. Suardana, D. Y. Jung, K.-S. Choi, and J. K. Lim, *International Journal of Precision Engineering and Manufacturing*, 13, 1199 (2012).

Application of Halpin-Tsai Equation to Kenaf Reinforced Biopolymer Composites

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Abstract. This paper demonstrated the utilization of theoretical modeling method for the determination of natural fiber reinforced biopolymer composites elastic modulus. The biocomposites utilized the combination of kenaf bast fiber as the reinforcement material and sugar palm starch (SPS) biopolymer as the matrix material. The elastic modulus prediction was performed using Halpin-Tsai equation for varying total fiber content condition. Modeling results showed that final kenaf reinforced SPS biocomposites elastic modulus increases as the total kenaf fiber contents were increased, in both longitudinal and transverse direction. Obtained results also indicated that significant improvement in longitudinal moduli was achieved using higher fiber contents compared to the transverse moduli for that kenaf reinforced SPS biocomposites. The Halpin-Tsai equation implemented in this study also proved its suitability for preliminary biocomposites design purpose in order to gain quick and cost effective information prior to experimental biocomposites characterization approach.

Keywords: Theoretical Analysis, Biocomposites, Kenaf-Sugar Palm Starch, Halpin-Tsai Method.

Introduction

In recent years, biocomposites have made significant progress in many aspects involving materials, processes and applications due to the higher awareness towards using better sustainable resources by consumers [1]. On the other hand, biocomposites also offers cost and lightweight advantages compared to synthetic composites. Kenaf natural fiber is among the most used type of reinforcement for biocomposites fabrication which is contributed to its high specific strength and modulus as well as low cost comparable to other commodity type of natural fibers such as hemp, flax and jute [2]. In addition, the emergence of new matrix materials made from natural resources such as starch to replace petroleum based polymers have also contributed to high potential of using biocomposites materials where fully biodegradable and renewable composites can be manufactured [3]. Among the promising type of

biopolymer matrix is sugar palm starch (SPS) made from sugar palm tree which have high raw material availability especially in Malaysia.

Until now, very limited research has been reported on the application of the SPS for biocomposites application. One notable effort was reported by Sahari et al [4] whereby the SPS biopolymer was used as the matrix material in combination with sugar palm fiber as the reinforcement material to formulate fully biodegradable biocomposites. Through literature review, it was also found that the application of kenaf with SPS biopolymer have yet been reported in literature despite the advantage offered kenaf. Hence, the gap found have motivated the authors to explore the potential of producing kenaf reinforced SPS biocomposites and characterized the mechanical properties of this new class of biocomposite material. In this research, the elastic modulus of kenaf reinforced SPS biocomposites was determined using theoretical modeling approach. Halpin-Tsai micromechanical model was applied in assessing the final biocomposites properties at varying fiber loading for both longitudinal and transverse conditions.

Materials and Methods

Materials

In the kenaf reinforced SPS biocomposites, the kenaf bast fiber length was selected at 2 mm and the total fiber contents were varied from 0 wt% to 50 wt%. Apart from that, SPS biopolymer was prepared using 70 wt% sugar palm starch and 30 wt% glycerol as plastisizer. All material properties used in the analysis were obtained from literature review. Table 1 summarized the physical and mechanical property of the individual constituents for the biocomposites.

Table 1: Kenaf bast fiber and SPS biopolymer material properties [5]–[7]

Material	Density, ρ (g/cm^3)	Tensile modulus, E (GPa)	Fiber diameter (μm)
Kenaf (bast)	1.45	53	12-36
Sugar palm starch (at 30 wt% glycerol)	1.40	2.5	NA

Methods

The elastic modulus of single fiber/matrix composite system, E using Halpin-Tsai equation is calculated using Equation (1)

$$E = E_m[(1+\zeta\eta V_f)/(1-\eta V_f)] \quad (1)$$

where η is the efficiency factor and calculated using equation (2)

$$\eta = [(E_f/E_m)-1]/[(E_f/E_m)+\zeta] \quad (2)$$

where ζ is the shape fitting factor of the lamina/laminate. E_f and E_m are the fiber modulus and matrix modulus respectively.

Summary of the shape fitting factor, ζ values corresponding to the different type of moduli is shown in Table 1.

Table 1: Shape fitting factor, ζ values [8]

Composite modulus, E	Fiber modulus, E_f	Matrix modulus, E_m	ζ	Remarks
E_{11}	E_f	E_m	$2(L/d)$ or $2(L/t)$*	Longitudinal modulus
E_{22}	E_f	E_m	2	Transverse modulus

*Note: L/d or L/t = fiber aspect ratio where L= fiber length, d= fiber diameter and t=fiber thickness

Results and Discussion

Based on Halpin-Tsai equation, theoretical modeling results on the effect of fiber loadingsfor kenaf reinforced SPS biocomposites elastic modulus is shown in Figure 1. It is observed that the biocomposites elastic modulus property was increased as the fiber loadings increased, for both longitudinal and transverse directions. Significant improvement in longitudinal moduli wasalso predicted using higher fiber contents compared to the transverse moduli for that kenaf reinforced SPS biocomposites.

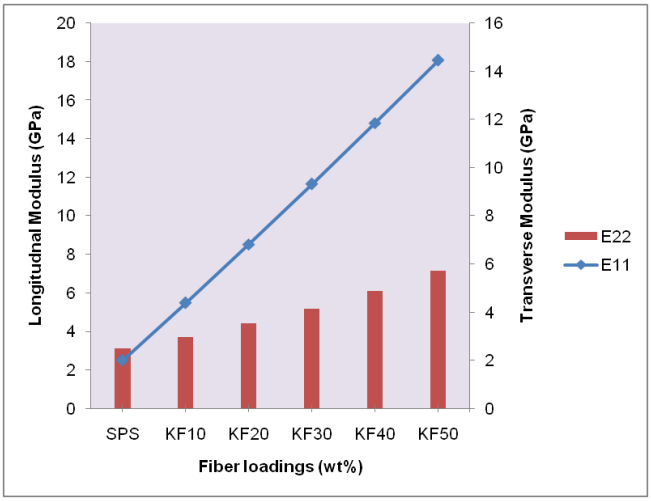

Figure 1: Effect of fiber loading on kenaf reinforced SPS biocomposites elastic modulus

Conclusions

In conclusion,theoretical modeling results using Halpin-Tsai equation showed that final kenaf reinforced SPS biocomposites elastic modulus increases as the total kenaf fiber contents were increased, in both longitudinal and transverse direction. Significant improvement in longitudinal moduli wasalso achieved using higher fiber

contents compared to the transverse moduli for that kenaf reinforced SPS biocomposites. The Halpin-Tsai equation implemented in this study also demonstrated its suitability for preliminary biocomposites design purpose in order to gain quick and cost effective information prior to experimental biocomposites characterization approach.

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References

[1] S. M. Sapuan and M. R. Mansor, Concurrent engineering approach in the development of composite products: A review,*Materials and Deign*, Volume 58, 2014, pp. 161–167.

[2] M. R. Mansor, S. M. Sapuan, E. S. Zainudin, A. A. Nuraini, and A. Hambali, Hybrid natural and glass fibers reinforced polymer composites material selection using Analytical Hierarchy Process for automotive brake lever design, *Materials and Design*, Volume 51, 2013, pp. 484–492.

[3] O. Faruk, A. K. Bledzki, H.-P. Fink, and M. Sain, Biocomposites reinforced with natural fibers: 2000–2010,*Progress in Polymer Science*, Volume 37, No. 11, 2012, pp. 1552–1596.

[4] J. Sahari, S. M. Sapuan, E. S. Zainudin, and M. A. Maleque, Mechanical and thermal properties of environmentally friendly composites derived from sugar palm tree, *Materials and Design*, Volume 49, 2013, pp. 285–289.

[5] J. Sahari, S. M. Sapuan, E. S. Zainudin, and M. A. Maleque, A new approach to use Arenga Pinnata as sustainable biopolymer: Effects of plasticizers on physical properties, *Procedia Chemistry*, Volume 4, 2012, pp. 254–259.

[6] H. M. Akil, M. F. Omar, A. A. M. Mazuki, S. Safiee, Z. A. M. Ishak, and A. Abu Bakar, Kenaf fiber reinforced composites: A review, *Materials and Design*, Volume 32, No. 8–9, 2011, pp. 4107–4121.

[7] J. Sahari, S. M. Sapuan, E. S. Zainudin, and M. A. Maleque, Thermo-mechanical behaviors of thermoplastic starch derived from sugar palm tree (Arenga pinnata),*Carbohydrate Polymers*, Volume 92, 2013, pp. 1711–1716.

[8] J. C. Halpin and J. L. Kardos, The Halpin-Tsai Equations: A review,*Polymer Engineering and Science*, Volume 16, No. 5, 1976, pp. 344–352.

Study on linear relation between energy used by internal mixer screw and torque or time of compounding kenaf core polypropylene composites

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Abstract. Area under torque-time graph produced by internal mixer shows energy used or work done by the mixing screws. Through equation manipulation, energy used is proportional to torque and time. Thisstudy looks intorelation between energy and torque and also between energy and time by comparing either same time or same torque of several processes. The mixing process were carried out by melting polypropylene, insertion and diffusion of kenaf core fibre into polypropylene to produce kenaf core polypropylene with 40% fibre loading. Analysis of data began with insertion of fibre until diffusion stage. Moreover, second analysis only in diffusion stage was done to reduce various variables fibre insertion stage. It is concluded, linearly proportional relation for energy used by internal mixer screw only be produce when processes are compared base on same torque value.

Introduction

Internal mixer commonly used to prepare thermoplastic composites. In most cases, rotational speed and temperature of mixing chamber has a fixed value as determine by the user. The processing output was recorded in form of graph torque verses time and stock temperature verses time. From torque-time graph, by using the definition of power for rotational object (power is product of torque and angular speed) and power as rate of energy or work done, area under the graph is work done or energy used by internal mixer screws.

Work done/Energy used = constant x torque x time = constant x area under the graph

Work done is proportional to mixing time and torque.

Comparison between mixing processes were made by using time or torque as constant i.e. comparing processes by using same mixing time or same torque. Thisstudy looks into relation between work done and torque and also between work done and mixing time. Blending or mixing behaviour of polymers or composites were studied through torque internal mixer has been done by various researchers [1-4]. Insertion of fibre into the internal mixer was ignored by most researchers because the inconsistency of fibre insertion. Through experience, smooth and constant insertion of natural fibre into

internal mixer is almost impossible to achieve. Small opening or entranceofmixing chamber and hydrophilic behaviour of low density of natural fibre produce clogging and bridging during fibre insertion. Moreover, in some occasions, vapour was produced and expelled from mixing chamber through entrance. Steady flow of fibre can be achieved by inserting small amount of fibre one at a time.All this situations caused torque variations in torque-time graph. On the other hand, without above situations (after material loading); under constant rotational speed and temperature, variations of torque can be used as indirect measure of molar mass variation [1].Therefore most of the studies startto analyse their data after material loading.

Methodology

Kenaf core fibre size less than 40 micrometer was used in this study. Kenaf core fibre were heat treated at temperature 103,165, 175, 185, 195 and 205°C in normal atmosphere. Polypropylene, brand Titan Pro grade 6331 with melt flow index of 14, was used as a matrix for composite. This study compound 16g of kenaf core and 24g polypropylene into kenaf core polypropylene composite with 40% fibre loading. The compounding process was carried out using Brabender internal mixer. Rotational speed and temperature for compounding process are 60 rpm and 175°C respectively.The compounding process involving few stages, first stages is polymer melting, then fibre insertion and finally diffusion stage. In first stage, polypropylene was inserted into mixing chamber until it was homogenously melted as indicated by constant torque. In second stage, kenaf core fibre was added into the chamber. External force was used ensure continuous flow of material into the entrance to break bridging and clogging form at the entrance. In final stage, after all the fibre was inserted, 5kg load were used to close the entrance until the compounding process is finished. The process was repeated for all treated fibre and one untreated fibre with 2 replications. Since raw data of the process not provided by the software, the produced graphsof compounding process was digitized using Web Plot Digitizer [5]. Data were analysed from fibre insertion stage to either fixed time or torque to study the relation between energy used and torque and also time. Second study look into data relation during decreasing torque in diffusion stage (after fibre insertion stage).

Results and discussion

Relation between energy used and torque (by fixing time) is shown by Figure 1. Data for each mixing time, the data were clustered on torque axis and energy required axis. For higher mixing time, data were clustered on upper left of the graph. This is due to fixed time was chosen at diffusion stage where torque are in declining stage therefore longer time recorded less torque.

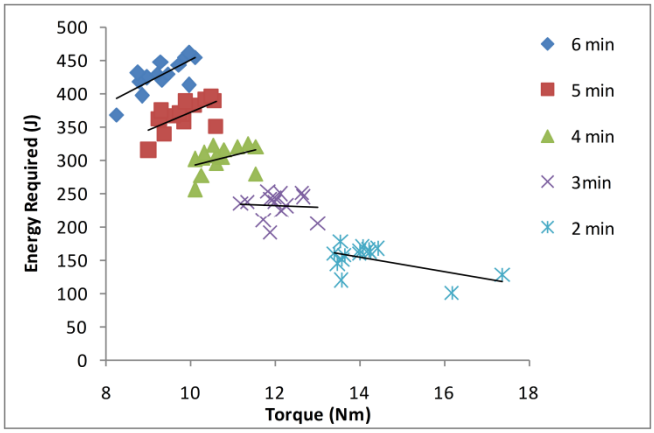


Figure 1. Relation between energy used and torque

By analysing data in each cluster using linear regression as shown by Table 1;for different mixing time,gradients of each equation is greatly varied and does not have strong relation between energy required and torque as shown by low value of R^2, coefficient of determination.

Table 1. Linear regression for energy used vs torque (fix time)

Fixed time	Regression equation	R^2
2	y = -10.76x + 303.6	0.32
3	y = -2.223x + 258.7	0.003
4	y = 15.89x + 132.8	0.162
5	y = 27.18x + 99.68	0.401
6	y = 33.25x + 117.5	0.569

Relation between energy used and time (by fixingtorque) is shown by Figure 2. Contrary to fix mixing time, higher fix torque would produce a cluster of data on upper right side of energy required vs time graph. Moreover, data in a clustertend to be formed in straight line.

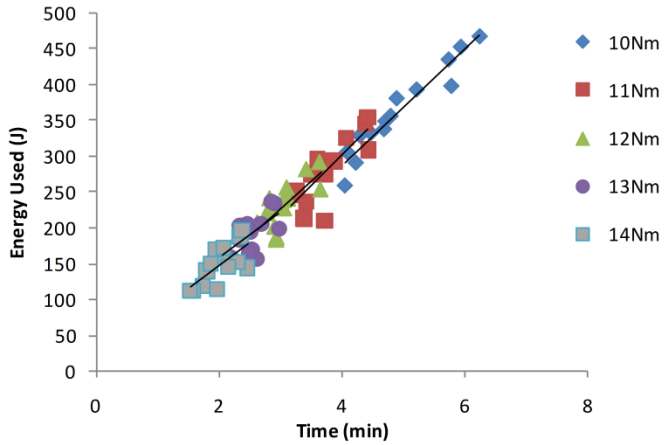


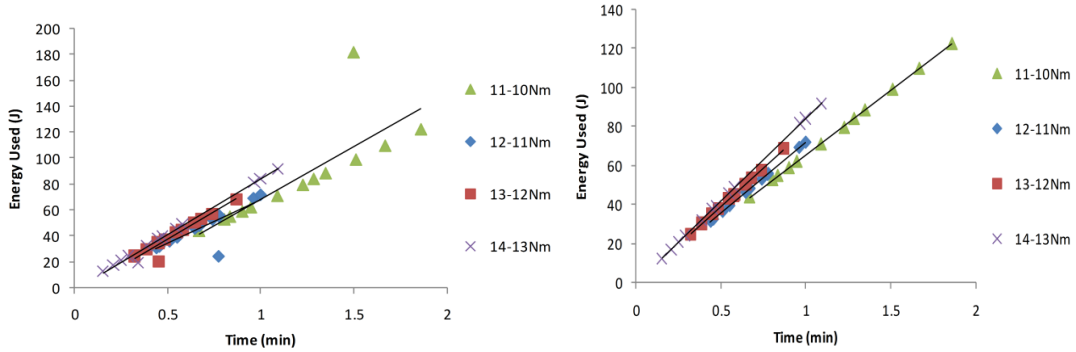
Figure 2. Relation between energy used and time

Data in each cluster have strong linear regression relation between energy required and time as shown in Table 2. The R^2 valuesare closer to 1 which indicate the equation derived from the data can be use to predict the amount of energy used to compound composite which include fibre insertion and diffusion stage. R^2 values of each cluster shows lower descending torque produce better relation for energy used and time (duration of mixing).

Table 2.Linear regression for energy used vs time (fix torque)

Fixed Torque	Regression Equation	R^2
10	y = 81.56x - 38.74	0.926
11	y = 85.26x - 39.89	0.653
12	y = 77.85x - 5.856	0.545
13	y = 64.62x + 28.37	0.414
14	y = 66.19x + 15.79	0.5

The above results included fibre insertion stage, which influenced by various factors, and diffusion stage. For study of interaction between fibre and matrix, fibre insertion stage should be excluded. Data were analysed when torque is reduced by 1 Nm in diffusion stage. Results of analysed data were shown by Figure 3afor raw data and Figure 3b) for data without outlier.



a) b)

Figure 3. Relation between energy used vs time of descending torque a) raw data b) without outliers.

It is noted that outlier for torque range 11-10, 12-11, 13-12Nm, was contribute by single data from second replication of heat treated fibre at temperature 175°C. Theoretically, only interaction between fibre and matrix exist during diffusion stage. Presence of outliers indicates other factor has influenced the movement of screws even at diffusion stage. Most probably the factors are contributed by variation of natural fibres. Equation and R^2 values for both figures are shown by Table 3.

Table 3 Equations and R^2 values of relation between energy used vs time of descending torque

Torque reduction	Equation (raw data)	R^2	Equation (without outlier)	R^2
11-10Nm	y = 81.41x - 12.57	0.655	y = 65.95x - 0.380	0.999
12-11Nm	y = 65.99x + 1.991	0.653	y = 72.72x - 0.451	0.999
13-12Nm	y = 83.5x - 3.837	0.917	y = 79.22x - 0.500	0.999
14-13Nm	y = 86.06x - 1.491	0.990	y = 84.71x - 0.113	0.999

After eliminating the outlier, equationsfor relation between energy used and time has shown that gradient decreases for lower segment of torque reduction. The relation is very strong that R^2 value is almost 1.

Conclusions

By manipulating power equation, energy used is proportional to time and torque. However, to compare data of several processes, linearly proportional relation only produced when the processes are compared on the base of same torque value.

References

[1] W. R Waldman and M.A.D.Paoli,Influence of compatibilizer on blends degradation during processing. *Polímeros*, 2013, 23, pp.7-12.

[2] D.O. Castro, A. Ruvolo-Filho, E. Frollini, Materials prepared from biopolyethylene and curauafibers: Composites from biomass, *Polymer Testing*,31,2012,pp 880-888

[3] G.P. Baeza, A.C.Genix, C. Degrandcourt,L.Petitjean, J.Gummel, M. Couty, and J. Oberdisse,Multiscale filler structure in simplified industrial nanocomposite silica/SBR systems studied by SAXS and TEM. *Macromolecules*, 2012, 46(1), pp.317-329.

[4] A. R. M. Costa, T.G. Almeida, S.M. Silva, L.H. Carvalho, E.L.Canedo, Chain extension in poly(butylene-adipate-terephthalate). Inline testing in a laboratory internal mixer.*Polymer Testing*, 2015, (42),pp. 115-121.

[5] AnkitRohatgi; WebPlotDigitizer v3.6; January 2015; http://arohatgi.info/WebPlotDigitizer.

Flexural Properties of Random Orientated Kenaf Mat Reinforced Epoxy Fabricated via Vacuum Infusion

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Abstract. In this study, the random orientated kenaf mat was used as a reinforcement material and epoxy as the resin. The composites were fabricated via vacuum infusion in which the resin was impregnated into the kenaf mat by producing a vacuum pressure to absorb the resin. From the composites made, flexural tests were performed and from the results, it is understood that neat epoxy had a higher flexural strength than kenaf reinforced composite at 147% when compared to the strongest kenaf reinforced composite of 50 vol% kenaf. However, from flexural modulus results, 50 vol% kenaf reinforced epoxy was the strongest at 150% stronger than the weakest that is of neat epoxy.

Keyword: flexural properties, flexural strength, flexural modulus, kenaf reinforced epoxy, vacuum infusion

Introduction

Fibers from natural source are gaining importance in the field of reinforcing materials to replace petroleum based synthetic fibers. The positive traits of natural fibers as reinforcing materials include renewability, low density, high specific properties, nonabrasive and lack of residues when burnt while the negative traits of natural fibers include porosity that leads to fiber swelling, low heat resistance and variable properties according to cultivation soil and climate [1]. Apart from these properties, kenaf (*Hibiscus cannabinus L.*) is used in this work due to its ecologically friendly properties. Kenaf can absorbs the nitrogen and phosphorus that are present in the soil [2]. These minerals also help to increase cumulative weed weight, crop height, stem diameter and fiber yield. Furthermore, kenaf has a significantly high ability to accumulate carbon dioxide and turn it into oxygen. Its photosynthesis speed is at least three times higher than that of usual plants, and it can absorb carbon dioxide 1.4 times its own weight [3].

There are multiple ways to fabricate a composite reinforced with natural fiber such as hand lay-up and vacuum bagging. Amongst them there is the vacuum infusion process, also known as resin infusion or vacuum assisted resin transfer molding. It is a

closed mold process in which the resin is introduced to its reinforcement material by applying a vacuum. The reinforcing material itself is compressed due to the vacuum and only one side of the mold is solid reducing the initial investment cost. As vacuum infusion is a closed mold process, the operator is less exposed to volatile components that might be emitted in the work atmosphere such as styrene vapour that causes detrimental effects to the workers [4]. Further good qualities of the vacuum infusion process are high fiber content, low void content, increased mechanical strength for the same thickness, fume free and the ability to allow the manufacturing of large, highly integrated structures [5]. However, there are drawbacks to vacuum infusion process such as the variation in thickness of the part, product of the non-uniform compaction pressure and the limited ability to achieve high fiber content [6].

Methodology

Materials

The resin used in this work is epoxy of Epoxamite® 100 and 103 SLOW Hardener. The resin to hardener ratio is 100:28.4 by weight, the pot life is 55 min and the curing time is 20-24 h. Kenaf mat with random orientated fibers was used as reinforcing material for the epoxy.

Fabrication

According to ASTM D790 the size of the sample for flexural test is 125mm×12.7mm×3mm. Thus, to produce 10 samples of composite per plate, the size of the plate (150mm×150mm×3mm). As preparation, kenaf mat was cut into 150 mm×150mm in size and weighed. Afterward, the weighed kenaf was laid on top of one another to accommodate the total weight needed for fabrication process and compressed in between two metal plate. The compressed mat was put on top of a glass table and preparation for vacuum infusion was made.

The preparation of vacuum infusion started with the placement of material from bottom to top: kenaf mat, peel ply, resin mesh and vacuum bag. Peel ply was used to remove excess resin ontop of the mat, resin mesh was used to increase the speed of resin flow and vacuum bag act as a mold that shapes the composite. Two tubes were used as an inlet to infuse the resin and as an outlet to be connected tovacmobile 20/2 vacuum system. The spiral tubes were placed at the inlet and outlet to spread the resin throughout the mat and silicon tape was used to stick and seal the vacuum bag onto the glass table and to prevent leakage of gas during the resin infusion process. Before the resin was infused, gas leakage was checked and sealed upon discovery.

After the preparation was completed, epoxy is mixed with hardener and infused into the kenaf mat. Plates with kenaf loading of 20, 30, 40, 50 and 60 Volume Percent (vol%) were made. For each loading, 2 plates were made and from each plate, 10 samples were obtained. Separately, 2 plates of neat epoxy were made by pouring adequately stirred epoxy and hardener mixture into a mold of 180mm×120mm. From each neat epoxy plates, 9 samples were obtained.

Flexural Test

Each sample was objected to 3-point bending flexural test under ASTM D790 using Instron® 3365 Dual Column Tabletop Testing System. The rate of crosshead motion was the thickness of the sample divided by 10.

Results and Discussion

The flexural strength of each samples are obtained and the results are plotted in Fig. 1. As can be seen from Fig 1, the neat epoxy (0 vol% kenaf's loading) had a higher flexural strength compared to kenaf reinforced epoxy at 76.6 MPa. However, with the increment of kenaf, the strength of the composites increased gradually with the optimum strength of kenaf reinforced epoxy at 52.2 MPa (50 vol% kenaf's loading).When flexural modulus of each samples are plotted as illustrated by Fig. 2, the neat epoxy exhibited the lowest modulus of 2.37 GPa while kenaf reinforced epoxy at loading rate of 50 vol% provided highest modulus of 3.56 GPa. Again, 50 vol% of kenaf's loading indicates itself as the strongest of kenaf reinforced epoxy.

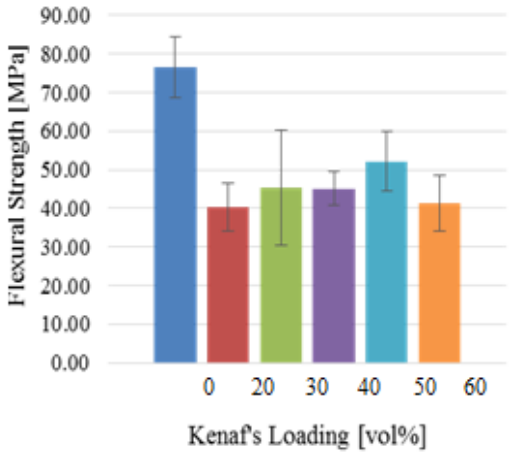

Fig. 1. Flexural strength for each kenaf's loading

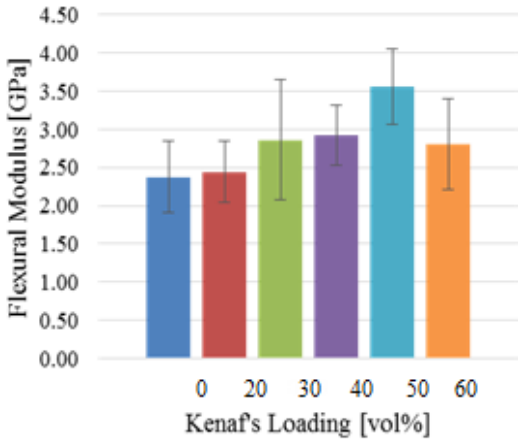

Fig. 2. Flexural modulus for each kenaf's loading

From Fig. 1 and Fig. 2 it can be concluded that while kenaf reinforced epoxy can withstand higher mechanical stress before being deformed, the composite itself could not withstand a high degree of deformation before fracturing when compared to neat epoxy. This can be explained by the higher flexural modulus as the flexural modulus is the ratio of stress and strain (deformation) and low flexural strengthof kenaf reinforced composite when compared to neat epoxy.

Conclusions

As a conclusion, random orientated kenaf mat reinforced epoxy was successfully fabricated via vacuum infusion. The neat epoxy exhibited highest flexural strength of 76.6 MPa and lowest flexural modulus of 2.37 GPa. While kenaf reinforced epoxy generally produced a composite of lower flexural strength than neat epoxy, composite

with 50 vol% kenaf had the highest flexural strength of 52.2 MPa and overall highest flexural modulus of 3.56 GPa.

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References

[1] E. Rodríguez, R. Petrucci, D. Puglia, J.M. Kenny, and A. Vazquez, Characterization of composites based on natural and glass fibers obtained by vacuum infusion,*Journal of Composite Materials*, Vol 39(3), 2005, pp. 265-282.
[2] H. Akil, M.F. Omar, A.A.M. Mazuki, S.Z.A.M. Safiee, Z.A.M. Ishak, and A.A. Bakar,Kenaf fiber reinforced composites: a review,*Materials & Design*, Vol 32(8),2011, pp. 4107-4121.
[3] S. Serizawa, K. Inoue, andM. Iji, Kenaf-fiber-reinforced poly (lactic acid) used for electronic products,*Journal of Applied Polymer Science*, Vol 100(1), 2006, pp. 618-624.
[4] C. Williams, J. Summerscales, and S. Grove, Resin infusion under flexible tooling (RIFT): a review,*Composites Part A: Applied Science and Manufacturing*, Vol 27(7), 1996, pp. 517-524.
[5] A. Hammami, and B.R. Gebart,Analysis of the vacuum infusion molding process,*Polymer Composites*, Vol 21(1),2000, pp. 28-40.
[6] B. Yenilmez, M. Senan, and E.M. Sozer,Variation of part thickness and compaction pressure in vacuum infusion process,*Composites Science and Technology*, Vol 69(11),2009, pp. 1710-1719.

The effect of alkalization and silane coupling agent treatment on the water absorption of roselle fibre reinforced vinyl ester composites

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Abstract. In this study, the effect of water absorptions of different surface treatment roselle fiber reinforced vinyl ester composites prepared by wet hand lay-up method was studied. The water absorption tests were conducted by immersing the composite samples into distilled water at room temperature for a period of 10 days. From the results, it can be seen that the water absorption behavior of composite was found to follow a non-Fickian behavior. The maximum water absorption percent were determined from the obtained water absorption curves. The results show that non treatment samples absorb more water absorption percentage compared to the treated samples. The samples treated with silane coupling agent shows the lowest percentage of water absorption. This can be concluded that the surface treatment can reduces water absorption of roselle fibre reinforced vinyl ester.

Keywords: Roselle fibre, vinyl ester, water absorption, alkalization, silane coupling agent.

Introduction

Natural fibres such as hemp, kenaf, jute, sisal, banana, flax, and oil palm have been in considerable demand in recent years due to their eco-friendly and renewable nature [1]. Recently, in line with raising environmental concerns, scientists and researchers are now replacing synthetic fibres with natural fibres as the main component in composites [2-7]. The advantages of natural fibres include low cost, good mechanical properties, abundant availability, material renewability, biodegradability, non-abrasive nature, and ease of recycling as compared to synthetic fibres [8-9]. These reasons have attracted material engineers to use natural fibres as

reinforcing filler in polymer composites to reduce uses of timber or forest resources and explore under-utilized natural fibres. Natural fibres are widely used in automotive[10] and construction engineering [11]. Natural fibres can be found in southeast Asian countries such as Malaysia, Indonesia, and Thailand [8].

Natural fibre such as Roselle (Hibiscus sabdariffa) are found in abundance in nature and cultivated in Borneo, Guyana, Malaysia, Sri Lanka, Togo, Indonesia and Tanzania. Roselle is one of the plants found to be suitable to be used to produce natural fibres.The scientific name for Roselle is *Hibiscus sabdariffa*L. and it is from Malvacea family. Roselle belongs to the hibiscus family and is found abundantly in tropical area. They are commonly used as an infusion and to produce bast fibre. There are various use of roselle. The fruit is commonly used in medical [12][13] and food industry [14][15] while the fibre is used as a textile [16] and reinforcement material for polymer composites [17]. However, very limited studies have been done on the application of Roselle fibres and its composites[18].Roselle stem is red in colour as illustrated in Figure 1 in water retting process. In Malaysia, after a year, Roselle plant will be cut, and it will become a waste. This is because the quality of roselle fruit is not good after a year. In order to use this plant efficiently, the fibre can be used as a reinforcement material for polymer composite.

Fig. 1Water retting process

Although roselle fibres have some advantages over synthetic fibre especially in term of material cost, is still weakness on the fibre matrix interface. Crucial issues in using natural fibres includes poor adhesion between fibre and matrix, water absorption and high moisture content which leads to dimension instability of the fibers that can cause micro cracking of the composite and degradation of mechanical properties. The performance and quality of the composites product highly depends on fibre matrix adhesion and stress from the matrix to the fiber Researchers have reported work on modification of the roselle fibre to improve the fibre/matrix interfacial bonding for fabrication of polymer composites for different applications [18-22]. Also a few researchers have review papers covering the chemical and mechanical properties of roselle fibre in polymer composites [17][21].

Alkalization are common surface treatment methods for natural fibres. Many studies on the effect of alkalization have been performed, which indicate positive results on the mechanical properties of the composites [23-26].This treatment affects the chemical composition of the fibres, which can reduce their water absorption. This result might be due to removal of the lignin and hemicelluloses component, wax and oil of fibre [27-29]. Furthermore, the hydrophilic nature of the fibre is transformed into a more hydrophobic nature by the alkali treatment [28].The alkali treatment also reduces the fibre diameter, thus increasing the aspect ratio. The increase of aspect ratio roughens the surface, which further improves the mechanical properties of the fibre, resulting in better bonding between the fibre and matrix [31]. After treatment, the roselle fibre clearly developed a rough surface and the impurities were removed, which leads to an improvement of the mechanical properties of the composites as shown in Figure 2. Physically, the presence of a rough surface provides a mechanical lock between the fibre surface and the matrix, which may enhance the interfacial bonding between them [32]. Alkali treatment of natural fibre is illustrated in scheme 1 [33].

$$Fibre - OH + NaOH \rightarrow Fibre - O^{-}Na^{+} + H_2O$$

Scheme 1: Alkali treatment of natural fibre[33].

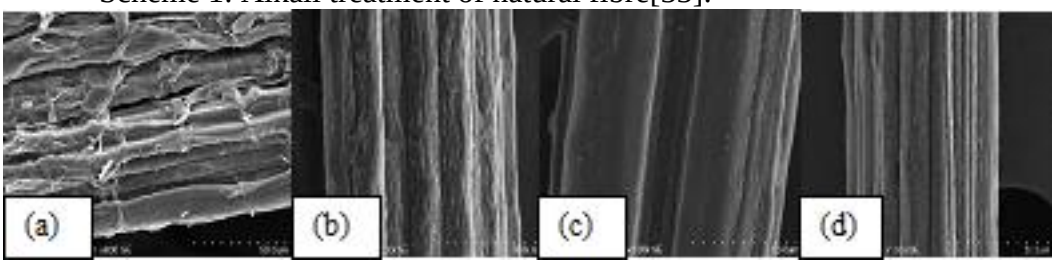

Fig.2 SEM Micrograph of (a) untreated roselle fibre, (b) 3% of NaOH treated of roselle fibre, (c) 6% of NaOH treated of roselle fibre, (d) 9% of NaOH treated of roselle fibre [34]

Further effective chemical treatment of natural fibres involves immersion in a silane coupling agent. Many studies have been performed on this method, and an extensive review was performed by [35-36]. Before immersion into this chemical, pre-treatment by NaOH is required to enable a more effective reaction with the chemical. Finally, treatment will improve the interface and bonding between the fibre matrix surface [33]. In this study, alkalization and silane coupling agent treatment have been used due to the low price and it's efficiency. Different percentages of concentration of NaOH (3%, 6%, 9%) and silane coupling agent treatment have been conducted to study the effect on the chemical properties (FTiR), physical (water absorption), mechanical (Tensile and impact test), morphology (scanning electron microscope).

Methodology

Materials

Vinyl ester (VE) obtained from Polymer Technology Pte. Ltd. Singapore was used in this study. The density, heat distortion temperature (HDT), viscosity and glass transition temperature of VE are 1.6g/cc, 120^0C, 400cps, 104.44 – 143.33^0C respectively. Methyl ethyl ketone proxide (MEKP) was used as a hardener. Roselle plants have been collected from Selangor, Malaysia. Roselle fibres were extracted by using water retting process for 14 days. The retted stem of the roselle plant was washed in running water and fibres were removed manually. Next, fibres were cleaned, and dried in the sunlight. Finally, the fibres were grinded and segregate it by using sieve machine (100 - 425μm) for the composites samples.

Chemical treatment

In this study, the fibres were prepared in 4 different types of treatment. Roselle fibres were immersed in the three different concentrations of NaOH for 2 hours at room temperature in a basin. 3%, 6% and 9% of concentrations have been chosen for the chemical treatment. For silane coupling agent treatment, the fibres of 6% NaOH was further immersed in silane solutions for 24 hours. After the chemical treatment of roselle fibre, the fibre were thoroughly washed with running water and dried in oven at 104 ^{0}C to eliminate the moisture effect of the fibres for 48 hours.

Composites samples

Wet hand lay-up process was used for sample preparation of roselle fibre reinforced vinyl ester composites. A rectangle mold made by aluminum sheet with a certain dimension was used for the composites samples. The composites sample was prepared by mixed 5%wt of roselle fibre and 92.5 %wt of VE. First, Roselle fibre was gradually added into vinyl ester composites and stirred using mechanical stirrer at 100 to 250 rpm till the mixing is uniformly distributed. Then, 2.5 %wt of Methyl ethyl ketone peroxide (MEKP) was added to composites mixing for curing. Finally, the mixture of roselle fibre and VE resin was poured into the aluminum mold and cured in for 24 hours at room temperature. The samples were cut from cured composites into a certain dimension as per ASTM for tensile test and impact test.

Water Absorption Test

In this study, the percentages of water absorption of roselle fibre were conducted by using ASTM D570-98[37]. The test was conducted by immersing the composites samples into the distilled water for 10 days. Water absorption was determined by the weight difference. In this study, the percentage weight gain (water uptake) of the composite specimens was measured at different time intervals (2, 8, 24–240 hr), totally 10 days. Before the test, composite specimens were dried in an oven at 80^0C in order to eliminate the effect of moisture content. This process was repeated until no mass change could be recorded. After immersion in water, composite samples were taken out from the water and all surface water was removed with a clean dry cloth or

tissue paper. Percentage of water absorption was determined by using eq.1. Three samples were prepared and average of percentage water absorption was calculated. The samples were weighed as M_0 first before being immersed in fresh water. After immersion, the samples were then weighed again as M_1.

Water absorption (%) $= \frac{M_1 - M_0}{M_0} x\ 100$ (1)

Water absorption

Although natural fibres have many advantages relative to their use as a reinforcement material such as being environmental friendly and having relatively similar properties as synthetic fibre, there are still flaws in natural fibre. The hydrophilic behavior of natural fibre makes it difficult to have a good adhesion between fibre/matrix and contribute to high water absorption of natural fibre; and this will weaken the composites product in application [38]. However, this problem can be overcome with surface treatment of natural fibre [35][39]. In this studies, the roselle fibre non-treated and treated have been investigated to evaluate their effect to reduce water uptake of the roselle fibre reinforced vinyl ester. From the published literature, it can be concluded that the influence of chemical treatment to water absorption on the natural fiber reinforced composites are important limitations for the outdoor application of such materials as the natural fbre absorb more water compared to the synthetic fibre[37]. The effect of this absorbed water content is to degrade mechanical properties[40], [41]. Figure 3 shows the percentages of water absorption of roselle fibre reinforced vinyl ester with different chemical treatment which are, 3, 6, 9%, and silane coupling agent. Generally, the treated fibre reduces the percentages of water absorption of the composites samples. It can be seen that the water absorption percent increased with immersion time, reaching a certain value at a saturation level, beyond which no more water content was absorbed and the water content absorbed by composite specimens remained constant. The roselle fibre treated with silane coupling agent gives the lowest value of water absorption while without treated fibre gives the highest percentage of the water absorption. Initially the water absorption of the composites samples is fluctuated and keeps on increased until it reached the saturation point. Without treatment and 6%NaOH treated reach the saturation point at 180 hours and the maximum water uptake is 2.2% and 1.75% respectively. It noticed that after 24 hours immersion, without treatment fibre gives the highest percentages of water absorption followed by 3, 6, 9% NaOH and the lowest is silane coupling agent with the value of 0.83, 0.68, 0.65, 0.54, and 0.48 respectively. The most of the water was absorbed through the sides of composite specimens because the fibers in composites are completely embedded in the resin matrix[37]. Roselle fibers are hydrophilic, so they can store more water than vinyl ester resin.

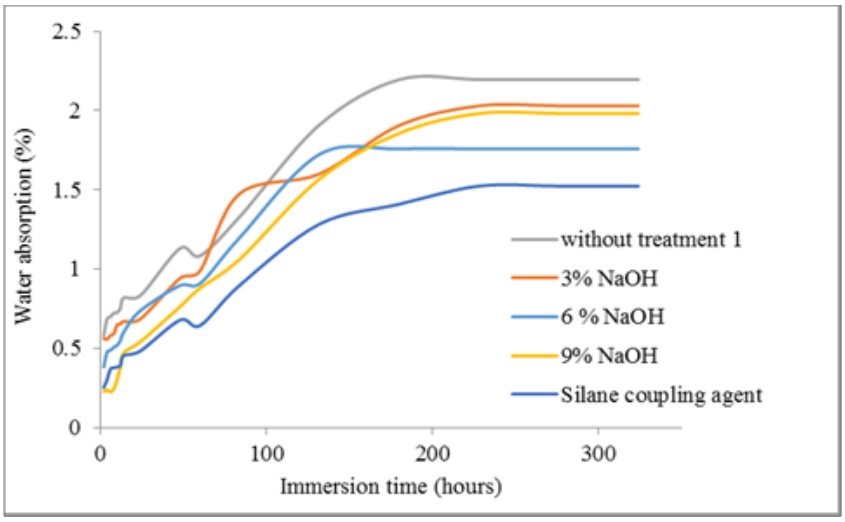

Figure 3. Percentages of water absorption versus immersion time of different treatment of 5% roselle fibre reinforced vinyl ester.

Conclusion

Roselle fibers have recently become attractive to material engineers and researches as a potential reinforcement for fiber reinforced polymer composites. However, the water absorption of these fibers gives serious concern, especially for their potential of outdoor applications. In this present work, the water absorption behavior of Roselle fibre reinforce vinyl ester composites with different chemical treatment were studied. The results show an improvement where the treatment roselle fibre reduced the water absorption of the samples. Silane coupling agent is the best method to reduce the water uptake of the composites where it gives the lowest percentage of the water absorption.

References

[1] H. P. S. A. Khalil, M. Jawaid, A. Hassan, M. T. Paridah, and A. Zaidon, "Oil Palm Biomass Fibres and Recent Advancement in Oil Palm Biomass Fibres Based Hybrid Biocomposites," in *Composites and Their Applications*, 2012, pp. 187–220.

[2] K. Begum and M. A. Islam, "Natural Fiber as a substitute to Synthetic Fiber in Polymer Composites : A Review," *Res. J. Eng. Sci.*, vol. 2, no. 3, pp. 46–53, 2013.

[3] T. Sathishkumar, P. Navaneethakrishnan, S. Shankar, R. Rajasekar, and N. Rajini, "Characterization of natural fiber and composites - A review," *J. Reinf. Plast. Compos.*, vol. 32, no. 19, pp. 1457–1476, Jul. 2013.

[4] V. V. Cholachagudda, U. P. A., and Ramalingaiah, "Mechanical characterisation of coir and rice husk reinforced," *Int. J. Innov. Res. Sci. Eng. Technol.*, vol. 2, no. 8, pp. 3779–3786, 2013.

[5] S. M. Reem, M. Ansari, and M. Saleh, “A Study On Mechanical , Thermal and Morphological Properties Of Natural Fibre / Epoxy Composite,” *J. Purity, Util. React. Environ.*, vol. 1, no. 5, pp. 267–296, 2012.

[6] S. L. Favaro, “Chemical, morphological and mechanical analysis of sisal fiber-reinforced recycled high-density polyethylene composites,” *eXPRESS Polym. Lett.*, vol. 4, no. 8, pp. 465–473, Jul. 2010.

[7] U. Nirmal, N. Singh, J. Hashim, S. T. W. Lau, and N. Jamil, “On the effect of different polymer matrix and fibre treatment on single fibre pullout test using betelnut fibres,” *Mater. Des.*, vol. 32, no. 5, pp. 2717–2726, 2011.

[8] M. R. Ishak, S. M. Sapuan, Z. Leman, M. Z. a Rahman, U. M. K. Anwar, and J. P. Siregar, “Sugar palm (Arenga pinnata): Its fibres, polymers and composites.,” *Carbohydr. Polym.*, vol. 91, no. 2, pp. 699–710, Jan. 2013.

[9] M. Jawaid and H. P. S. Abdul Khalil, “Cellulosic/synthetic fibre reinforced polymer hybrid composites: A review,” *Carbohydr. Polym.*, vol. 86, no. 1, pp. 1–18, Aug. 2011.

[10] F. M. AL-Oqla and S. M. Sapuan, “Natural fiber reinforced polymer composites in industrial applications: feasibility of date palm fibers for sustainable automotive industry,” *J. Clean. Prod.*, vol. 66, pp. 347–354, Mar. 2014.

[11] D. B. Dittenber and H. V. S. GangaRao, “Critical review of recent publications on use of natural composites in infrastructure,” *Compos. Part A Appl. Sci. Manuf.*, vol. 43, no. 8, pp. 1419–1429, Aug. 2012.

[12] N. D. Tori Hudson, “A Research Review on the use of Hibiscus Sabdariffa,” *Better Medicine - National Network of Holistic Practitioner Communities*, 2011. .

[13] A. Mungole and A. Chaturvedi, “Hibiscus Sabdariffa L a Rich Source of Secondary Metabolisme,” *Int. J. Pharm. Sci. Rev. Res.*, vol. 6, no. 1, pp. 83–87, 2011.

[14] F. Grace, “Investigation the suitability of Hibiscus Sabdariffa calyx extract as colouring agent for paediatric syrups,” 2008.

[15] W. Wilson, “Discover the many uses of the Roselle plant,” *NParks*, 2009. [Online]. Available: http://mygreenspace.nparks.gov.sg/discover-the-many-uses-of-the-roselle-plant/.

[16] V. A. Managooli, “Dyeing Mesta (Hibiscus sabdariffa) Fibre with Natural Colourant,” 2009.

[17] M. Thiruchitrambalam, A. Athijayamani, and S. Sathiyamurthy, “A Review on the Natural Fiber- Reinforced Polymer Composites for the Development of Roselle Fiber-Reinforced Polyester Composite,” *J. Nat. Fibers*, vol. 7, pp. 307–323, 2010.

[18] P. Ramu and G. V. R. Sakthivel, “Preparation and Characterization of Roselle Fibre Polymer Reinforced Composites,” *Int. Sci. Res. Journals*, 2013.

[19] B. Kaith and A. Chauhan, “Synthesis , Characterization and Evaluation of the Transformations in Hibiscus,” *E- J. Chem.*, vol. 5, pp. 980–986, 2008.

[20] A. Chauhan and B. Kaith, “Versatile Roselle Graft-Copolymers : XRD Studies and Their Mechanical Evaluation After Use as Reinforcement in Composites,” *J. th Chil. Chem. Soc.*, vol. 3, pp. 1262–1266, 2012.

[21] A. Chauhan and B. Kaith, “Accreditation of Novel Roselle Grafted Fiber Reinforced Bio-Composites,” *J. Eng. Fiber. Fabr.*, vol. 7, no. 2, pp. 66–75, 2012.

[22] A. Chauhan and B. Kaith, “Development and Evaluation of Novel Roselle Graft Copolymer,” *Malaysia Polym. J.*, vol. 6, no. 2, pp. 176–188, 2011.

[23] A. Abdal-hay, N. P. G. Suardana, D. Y. Jung, K.-S. Choi, and J. K. Lim, “Effect of Diameters and Alkali Treatment on theTensile Properties of Date Palm Fiber Reinforced Epoxy Composites,” *Int. J. Precis. Eng. Manuf.*, vol. 13, no. 7, pp. 1199–1206, Jul. 2012.

[24] H. Abral, M. F. Gafar, H. Andriyanto, S. M. Sapuan, and M. R. Ishak, “Alkali Treatment of Screw Pine (Pandanus Odoratissimus) Fibers and Its Effect on Unsaturated Polyester Composites,” *Polym. Plast. Technol. Eng.*, vol. 51, no. 1, pp. 12–18, Jan. 2012.

[25] S. S. Mullick, “Fabrication and Characterisation of Alkali Treated Natural Fibre Reinforced Polymer,” National Institute of Technology Rourkela, 2012.

[26] J. Rout, M. Misra, S. S. Tripathy, S. K. Nayak, and A. K. Mohanty, “The influence of fibre treatment on the performance of coir-polyester composites,” *Compos. Sci. Technol.*, vol. 61, no. 9, pp. 1303–1310, Jul. 2001.

[27] M. Jannah, M. Mariatti, A. Abu Bakar, and H. P. S. Abdul Khalil, “Effect of Chemical Surface Modifications on the Properties of Woven Banana-Reinforced Unsaturated Polyester Composites,” *J. Reinf. Plast. Compos.*, vol. 28, no. 12, pp. 1519–1532, Jul. 2008.

[28] D. Sampathkumar, R. Punyamurthy, B. Bennehalli, and S. C. Venkateshappa, “Effect of Esterification on moisture Absorptionof Single Areca Fiber,” *Int. J. Agric. Sci.*, vol. 4, no. 4, pp. 227–229, 2012.

[29] L. Y. Mwaikambo and M. P. Ansell, “Chemical Modification of Hemp , Sisal , Jute , and Kapok Fibers by Alkalization,” *J. Appl. Polym. Sci.*, vol. 84, pp. 2222–2234, 2002.

[30] P. R. D. Sampathkumar, C. V. Srinivaa, and B. Bennehalli, “Effect of alkali treatment on water absorption of single cellulosic abaca fiber,” *Bioresources*, vol. 7, pp. 3515–3524, 2012.

[31] H. S. Lee, D. Cho, and S. O. Han, “Effect of natural fiber surface treatments on the interfacial and mechanical properties of henequen/polypropylene biocomposites,” *Macromol. Res.*, vol. 16, no. 5, pp. 411–417, Jul. 2008.

[32] D. Bachtiar, S. M. Sapuan, and M. M. Hamdan, “The Influence of Alkaline Surface Fibre Treatment on the Impact Properties of Sugar Palm Fibre-Reinforced Epoxy Composites,” *Polym. Plast. Technol. Eng.*, vol. 48, no. 4, pp. 379–383, Apr. 2009.

[33] S. Kalia, B. S. Kaith, and I. Kaur, “Pretreatments of Natural Fibers and their Application as Reinforcing Material in Polymer Composites — A Review,” *Polym. Eng. Sci.*, vol. 49, pp. 1253–1272, 2009.

[34] R. Nadlene, S. M. Sapuan, M. Jawaid, and M. R. Ishak, “Mercerization Effect on Morphology and Tensile Properties of roselle fibre,” vol. 755, pp. 955–959, 2015.

[35] Y. Xie, C. A. S. Hill, Z. Xiao, H. Militz, and C. Mai, “Composites : Part A Silane coupling agents used for natural fiber / polymer composites : A review,” *Compos. Part A*, vol. 41, no. 7, pp. 806–819, 2010.

[36] X. Li, L. G. Tabil, and S. Panigrahi, “Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review,” *J. Polym. Environ.*, vol. 15, no. 1, pp. 25–33, Jan. 2007.

[37] N. M. Barkoula, B. Alcock, N. O. Cabrera, and T. Peijs, “Effect of Various Water Immersions on Mechanical Properties of Roselle Fiber–Vinyl Ester Composites,” *Polym. Compos.*, pp. 1–9, 2014.

[38] C. W. Nguong, S. N. B. Lee, and D. Sujan, “A Review on Natural Fibre Reinforced Polymer Composites,” in *World Academy of Science, Engineering and Technology*, 2013, vol. 73, pp. 1123–1130.

[39] S. H. Aziz and M. P. Ansell, “The effect of alkalization and fibre alignment on the mechanical and thermal properties of kenaf and hemp bast fibre composites: Part 1 – polyester resin matrix,” *Compos. Sci. Technol.*, vol. 64, no. 9, pp. 1219–1230, Jul. 2004.

[40] I. Aji, E. Zainudin, K. Abdan, S. Sapuan, and M. Khairul, “Mechanical properties and water absorption behavior of hybridized kenaf/pineapple leaf fibre-reinforced high-density polyethylene composite,” *J. Compos. Mater.*, vol. 47, no. 8, pp. 979–990, May 2012.

[41] Z. N. Azwa, B. F. Yousif, A. C. Manalo, and W. Karunasena, “A review on the degradability of polymeric composites based on natural fibres,” *Mater. Des.*, vol. 47, pp. 424–442, May 2013.

Optimization of pultruded kenaf composites process

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Abstract. During pultrusion process, there are some factorsthat are affected to composite profiles such as pulling speed, filler ratio, curing temperature, etc. The proper parameters setting reduce defect and increase the quality of pultruded composites profile. The objective of this paper is to optimize the pultruded kenaf composites process parameters in order to increase the tensile properties of the kenaf pultruded composite profiles. Besides, the best combination and the most contribution pultruded kenaf composite parameters also been revealed through analysis of variance (ANOVA).

Keywords: pultrusion process, pulling speed, filler ratio, gelation temperature, curing temperature, analysis of variance, kenaf pultruded.

Introduction

Pultrusion process is combination of pulling and impregnation process of fibre and matrix through the resin bath and cured in the heated die to produce composite profiles [1]. Pultrusion process technology has ability to produce continuous profile such as rectangular, round tube, hollow square, bar and beam without stop even during cutting process time. Pultrusion technology have been reported improves composite properties because the composites are forced and cured and hence are tightly bonded to each other [2]. The advantage of pultrusion process is produced with high fibre ratio which is increase the stiffness of the composites. Nosby et al, has reported that pultrusion process has an ability to produce high fibre concentration up to 70% fibre volume [3]. In this research, the study are focused on the improving the process parameter of the kenaf pultruded composites via the analysis of variance (ANOVA). The best parameter sets and the percentage of the contribution for each parameter also been presented.

Materials and Methods

Kenaf yarn origin from Bangladesh with average 1000 tax are used with 60% volume fraction were used as the reinforcement while vinyl ester resin from Swancor 901 pultrusion grade as a polymer matrix were purchased from FormalchemSdn Bhd. Vinyl ester swancor 901 was blended with 1.5% benzoyl peroxide (BPO) and 0.6% tertiary butyl per benzoate (TBPB) catalyst for 5 minutes. Parametersthat used in this study are filler ratio, gelation temperature, curing temperature, and pultruded profile

pulling speed. The selected pultrusion processing parameter for pultruded kenaf reinforced vinyl ester composite are inserted into orthogonal arrays table. The table is set at $L_9(34)$ orthogonal arrays which is contain 9 specimen of pultruded kenaf reinforced vinyl ester composite sample with 4 different pultrusion processing parameter and 3 level of parameter values. Micro size calcium carbonate ($CaCO_3$) is used in the pultruded composites as pultrusion process parameter. The resin was blend with three different type of formulation with different $CaCO_3$filler ratio; 10%, 30%, 50% of weight ratio which is set as the level of Parameter 1. Parameter 2 (gelation temperature) is to study the parameter of optimal gelation temperature of the pultruded composites which normally located between in enter tap of the heated die and the centre of the mould. At this temperature section is set as lower temperature zone just to form the pultruded composites at 80, 100 and 120°C. Parameter 3 is to study appropriated curing temperature where the pultruded kenaf changes from gelation to solidity condition. At this section, the temperature is set at the higher than gelation temperature at 140, 160 and 180°C.

Results and Discussion

All parameter level were set base on the factory setting for pultruded glass fibre composite **[4]**. The results of the flexural modulus for sample 1 to sample 9 shown in Table 1. In this experiment "the biggest the best" of tensile moduli value is used, therefore, signal to noise (S/N) of tensile modulus(TM) for each sample was calculated based on equation(1)

S/N=-10log(MSD)

$$\text{MSD} = \sum_{i=1}^{n} \left(\frac{1}{yi}\right)^2 / n \qquad (1)$$

Table 1: Tensile modulus and signal to noise(S/N) result for pultruded kenaf composite.

Filler Loading (%)	Gel Temp (°C)	Cure Temp (°C)	Pulling Speed (meter/minute)	Result	S/N
				Tensile Modulus	
10	80	140	0.2	7371.2	77.35075367
10	100	160	0.4	7903.2	77.95607755
10	120	180	0.6	8016.2	78.07937522
30	80	160	0.6	10637.4	80.53674973
30	100	180	0.2	8902.4	78.99013061
30	120	140	0.4	8019.6	78.08307955
50	80	180	0.4	12295.5	81.79494277
50	100	140	0.6	9107.4	79.18788643
50	120	160	0.2	9322.3	79.39050721

The effect of factors at different levels has been determined to find which level that have the highest effect of factors. Table 2 shows the most effect of factors at different level for pultruded kenaf reinforced vinyl ester composite. The highest value signal to noise for filler loading shown at level 3 which is set t 50% of the filler ratio. The highest signal to noise value for parameter of gelation temperature at level 2, curing temperature at level 3 and pulling speed at level 2.

Table 2: The effect of factors at different levels for pultruded kenaf reinforced vinyl ester composite.

Factors	Level 1	Level 2	Level 3
Filler Loading	77.7954	79.20332	80.12445
Gelation Temp	79.89415	78.71136	78.51765
Curing Temp	78.20724	79.29444	79.62148
Pulling Speed	78.57713	79.27803	79.268

Table 3: The effect of factors for the optimization of pultruded kenaf reinforced vinyl ester composite.

Factors	Level	Parameter Setting	PC%
Filler Loading	Level 3	50%	48%
Gelation Temp	Level 1	80°C	25%
Curing Temp	Level 3	180°C	20%
Pulling Speed	Level 2	0.4m/m	7%
Predicted Tensile Modulus			12295.53

The percentage of the parameter contribution is listed in the Table 3 and the filler loading has been found as the most contribute parameter for pultruded kenaf composites with 48%. Gelation Temperature become second in the most contributes parameter followed by curing temperature. The percentage of contribution parameter of pulling speeds at below 7%. The sample was run using the parameter setting given through ANOVA. The tensile test result of the kenaf pultruded specimen using the best combination parameter is given the value of 11754MPa which is less than 5% from the predicted value.

Conclusions

The parameters optimization via analysis of variance (ANOVA), the results revealed which parameters of pultruded kenaf composites process contribute most.Filler loading have shown the highest per cent of contribution in the process with 48%. The study also revealed the best combination of the pultruded kenaf composites with the setting for pulling speed at 0.4m/min, gelation temperature at 80°C, Curing temperature 180°C, and filler loading at 50% of the weight. The tensile modulus result of experiment and predicted value using ANOVA has been compared and the error is less than 5%.

References

[1] X. Peng, M. Fan, J. Hartley, and M. Al-Zubaidy, "Properties of natural fiber composites made by pultrusion process" *Journal of Composite Materials.*, vol. 46, 2011, pp. 237-246.

[2] K. Chandrashekhara, S. Sundararaman, V. Flanigan, and S. Kapila, "Affordable composites using renewable materials," *Material Science Engineering A*, vol. 412, 2005, pp. 2-6.

[3] N. Nosbi, H. Md Akil, Z.A. Mohd Ishak, and A. Abu Bakar. "Degradation of compressive properties of pultruded kenaf fiber reinforced composites after immersion in various solutions." *Materials & Design*, vol. 31, 2010, pp. 4960-64.

[4] R. Senawi, Pultrusion Procedure (Innovative Pultrusion Sdn Bhd), Senawang, 2012.

The Influence Of Oyster Shells As Natural Calcium Carbonate Filler In Hydraulic Lime Mortars For Use In High Temperature & Humidity Climatic Conditions

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Abstract. The widespread adoption of alternative binders is playing an increasing role in carbon dioxide (CO_2) abatement in green construction and the repair of traditionally built structures. Natural Hydraulic Lime (NHL) has better environmental credentials than Ordinary Portland Cement (OPC) due in part to its lower calcination temperature and its ability to absorb CO_2 during carbonation. Whilst exhibiting relatively favourable environmental credentials, NHL is more sensitive to climatic conditions during the setting and hardening process and this is especially pronounced in high humidity climates. Hydraulic lime relies upon a series of complex hydration and carbonation reactions to achieve its full set. It is well understood that high humidity environments create favourable conditions for the development of the products of hydration, but the carbonation reactions reduce or become dormant due to high sustained moisture contents. Anecdotal evidence suggests that the addition of $CaCO_3$ into hydraulic lime mortars can enhance the carbonation reaction due to 'seeding' of the crystal architecture. This research assesses the influence of $CaCO_3$ modification of NHL mortars subject to high humidity environments and investigates the subsequent effect on early development of various physical properties. Two types of micro calcite have been used in this research namely; precipitated calcium carbonate (99.99% pure) and oyster shells. Physical testing will be undertaken to investigate the micro-structural properties and moisture handling characteristic of the hydraulic lime mortars before and after the modification. The aim of the research is to encourage the set processes in relatively unfavourable environmental conditions.

Keywords: calcite, hydraulic lime, mortar, seeding

Introduction

Variations in environmental conditions, such as relative humidity and temperature, influence the performance of lime and cement mortars alike. Although lime mortar interacts with its environment during curing process, the environment also plays a major role in the evolution of products of hydration and other mineral compounds (Dotter, 2010). The efficacy of formation plays a significant role in determining strength and longer term durability and thereby resistance to deterioration characteristics. Climatic conditions cause stress to building through temperature variations (heat and cold), intensity of solar radiation, atmospheric gases, humidity,

rainfall and wind (Tanabe, 2008). Rapid dehydration of water in a freshly placed hydraulic mortar is well understood to lead to partial hydration. On the contrary, a hydraulic lime mortar placed in a high humidity environment will fail to carbonate and therefore not attain its full set characteristics (Allen, 2003). Making the usage of lime binder is unpopular in several part of the world. The wide scale adoption of alternative binders (i.e. NHL) can only be achieved if suitable set characteristic can be attained out (Pavia, 2008, Ball *et al* ., 2009, Ball *et al*., 2010, El-Turki *et al*., 2007) . Uptake of NHL in extreme climate such as arid and tropical regions would be surmised to be slow due to the climatic conditions prohibiting carbonation and hydration respectively benefits and therefore use.

Theoretically, plain mortar exposed to elevated temperature shows an accelerated precipitation of hydration products hydration (Desai *et al*., 2011). This phenomenon is responsible for the observed early strength development. However, this fast hydration in the initial stage leads to a more non-uniform distribution of the hydration products. This causes non-homogeneity in the microstructure that significantly reduces the long term strength.With the purpose of modifying and/or improving some of the properties of the mortars, traditionally they have been mixed (together with the basic components), some different products or additional constituents. Taking similar theory from cement hydration, the presence of some mineral admixtures in the mixture could modify the kinetic of hydration, reduce the heat evolution and produce additional calcium silicate hydrates (C-S-H) (Ezziane *et al.*, 2007). Inclusions of calcite crystals, for example in the form of crushed of seashells, can promote crystal growth during hydration process thus helping to develop strength. This can be achieved by process called "seeding"; accelerating carbonation by seeding calcium carbonate ($CaCO_3$) crystal growth on its physically similar (freshly) broken surfaces (Forsyth, 2008). The presence of this material aids carbonation as it is a porous particulate and the presence of carbonate in a mortar assists the carbonation of lime by "seeding" calcite crystal growth. As a porous particulate, accelerating carbonation by being permeable to carbonic acid; CO_2 can pass through the binder and the aggregate to reach more calcium hydroxide ($Ca(OH)_2$) deeper in the mortar, more quickly. Ezziane *et. al*. (2007) later concluded the seeding process can influence the mortar hydration and minimize disorders caused by the temperature rise according to the characteristics of these admixtures and their replacement rates. Lawrence (2006) stated that the seeding of lime mortars with 6% finely ground calcite has been shown to improve the rate of carbonation.

Research Objectives

The aim of the research is to undertake modification of naturally hydraulic lime mortars and investigate the subsequent effect on early strength development; in order aid in the specification of mortars high temperature and high humidity environment. Research objectives are;

a. to investigate the effects of mortar seeding on physical properties using laboratory testing techniques i.e. flexural and compressive strength

b. to investigate how mortar seeding additives affect the moisture handling characteristics e.g. capillary adsorption and vapour permeability.

Experimental Work

Specimen Preparation and Curing

Natural Hydraulic Mortar Class 3.5 (Moderately NHL 3.5) has been used to prepare the mortar. The product is compliant with European normative (British Standards, 2010). The binder/aggregate ratio (B/Ag) prepared was 1:3 by volume. This ratio is chosen because of the gauged mixes of cementatious material related to the sand content as the theory is that the voids of empty space between the sand particles account for a 1/3 of the volume of the sand. Volume proportions of compounds were then converted in weight to avoid measurement imprecision on mixing process. The sand was prepared by isolating 6% of the total sand weight in range of 250-300 μm to be seeded by the equivalent weight of seeding material ground to fineness similar to the sand replaced.

In this work, twotypes of micro calcite were procured to be used in this research; (i) precipitated calcium carbonate and (ii) oyster shells. Precipitated calcium carbonate (99.99% pure) was used as point of reference to ensure consistency. Oyster shells composed nearly 98% of $CaCO_3$ although the exact percentage would vary based on several factors such as the species, molecular impurities in the shell, the number and frequency of proteins embedded in the shell (Cocks, 2009).

The water:lime (W:L) ratio used to prepare all mortar pastes was 1:1.40. A normal consistency and a good workability (165 ± 3 mm) as required by criteria set out in the BS EN 459-2: 2010 measured by the flow table test were achieved by using this water amount. The mortar was prepared by 'dry mixing' methodology undertaken by Allen (2003) and Ball *et al*. (2009). Aggregates and lime were blended for 5 min for homogeneity. Water was then added and mixed for 5 min at low speed, and finally for 1 min at high speed. The mixtures then placed into lightly oiled with a proprietary mould release agent 160 mm x 40 mm x 40 mm mould using an automatic jolting table to compact them and remove any air bubbles and voids (British Standards, 2010b). The specimens then left in the curing cabinet under controlled environment of 33°C with 90% Relative Humidity (RH) using TAS Environmental Cabinet until the testing day.

Physical and Chemical Characteristics Investigations

A range of tests were established to determine the physical and chemical characteristics of the materials. Control specimens were used to provide a baseline for pre deteriorated properties. Specimens that had been subjected to accelerated deterioration were tested at various intervals (as previously outlined) adopting the following techniques:

a. **Carbonation Depth** (**Phenolphthalein staining test)**: Carbonation is of fundamental importance in making mortars harder and therefore more durable (Lanasa *et al.*, 2004) through examination of the diffusivity of CO_2 in mortar and normally

involves an increase in mass caused by the transformation of portlandite into calcite (Dheilly *et al.*, 2002). The freshly broken specimen (without size modification) will be used to check the carbonation depth in the specimen. This can be done by spraying a freshly exposed surface of the mortar specimen with 1% phenolphthalein solution (Lo and Lee, 2002). The solution is a colourless acid/base indicator, which turns pink when the pH is above 9, denoting the presence of $Ca(OH)_2$. It indicates the boundary at which the carbonated front meets the un-carbonated mortar, where mortar is alkaline. The $Ca(OH)_2$ will turn into pink while the carbonated portion is uncoloured.

b. **Sorptivity testing**: The sorptivity of the mortar specimens were evaluated using the 'sharp front theory' established by Hall and Hoff (2002). The sorptivity tests were carried out on parallelepiped specimens, after drying in a drying oven, at a temperature of 100 ± 10ºC were used. The preparation of specimens also included water impermeability of their lateral faces, reducing the effect of water evaporation. The test started with the registration of specimens' weight and afterwards, they were placed in a recipient in contact with a height of water capable to submerge them about 5 mm. After a predefined period of time, the specimens were removed from the recipient to proceed to weight registration. Before the weight, the specimens' superficial water was removed with a wet cloth as shown Figure 1. Immediately after the weight, the specimens were replaced in the recipient till reach the following time. The procedure was repeated, consecutively, at various times such as 1 minute, 3 minutes, 5 minutes, 10 minutes, 15 minutes and 30 minutes until the last reading.

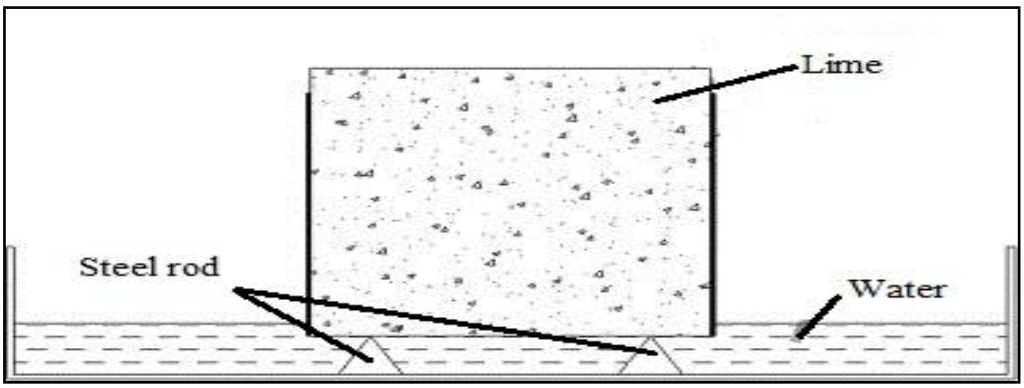

Figure 1: Sorptivity experiment set-up

c. **Flexural and Compressive Strength**: Each specimen type was tested in a Lloyds Universal Testing Machine (Model M5K) at specific intervals. This enabled an assessment of the influence of loss of $Ca(OH)_2$ on the physical characteristics of the mortar specimens.

Results and discussions

Carbonation Depth

The carbonation is a chemical reaction whose main reactant, in cementitious systems, is the portlandite. The extent and rate (kinetic) of the process are also affected by physical parameters of the masses (porosity/permeability) and by the practical curing and exposure conditions (e.g. CO_2 concentration, humidity, temperature, etc.) (Pacheco Torgal *et al.*, 2012).

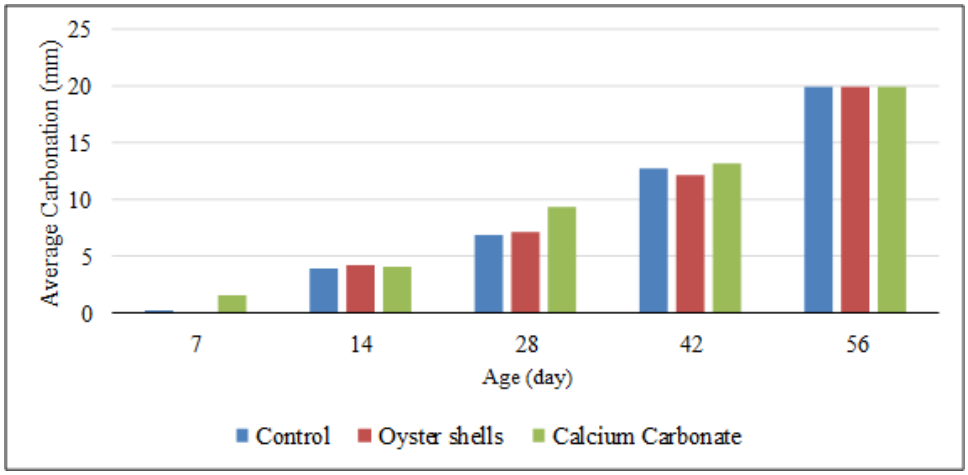

Figure 2: Phenolphthalein Test Results

It was observed during the experimental run that all the specimens were fully carbonated after 42 days as shown in Figure 2. From the data obtained, it can be seen that seeded specimens carbonate quicker than control specimens although not significant indicating the more permeable mortars carbonate quicker.

In principle, a lower occurrence of the carbonation phenomenon should be expected in mixtures that have or generate more portlandite i.e. higher hydraulic set (Forster, 2004).However, this theory cannot be entirely established in this study due to the absence of X-ray Diffraction (XRD) and Environmental Scanning Electron Microscope (ESEM) tests.

Sorptivity Test

Because of small initial surface tension and buoyancy effects, the relationship between cumulative water absorption (g/m^2) and square root of exposure time ($t^{0.5}$) shows deviation from linearity during first few minutes. Thus, for the calculation of sorptivity coefficient, only the section of the curves for exposure period from 1 minute to 30 minutes, where the curves were consistently linear, was used for the calculation of sorptivity. The sorptivity coefficient (k), was obtained by using the following expression (Dias, 2000):

$$\frac{W}{A} = k\sqrt{t}$$

Where W= the amount of water adsorbed in (g); A= the cross section of specimen that was in contact with water (m^2); t = time (min); k = the sorptivity coefficient of the specimen ($g/m^2/min^{0.5}$).

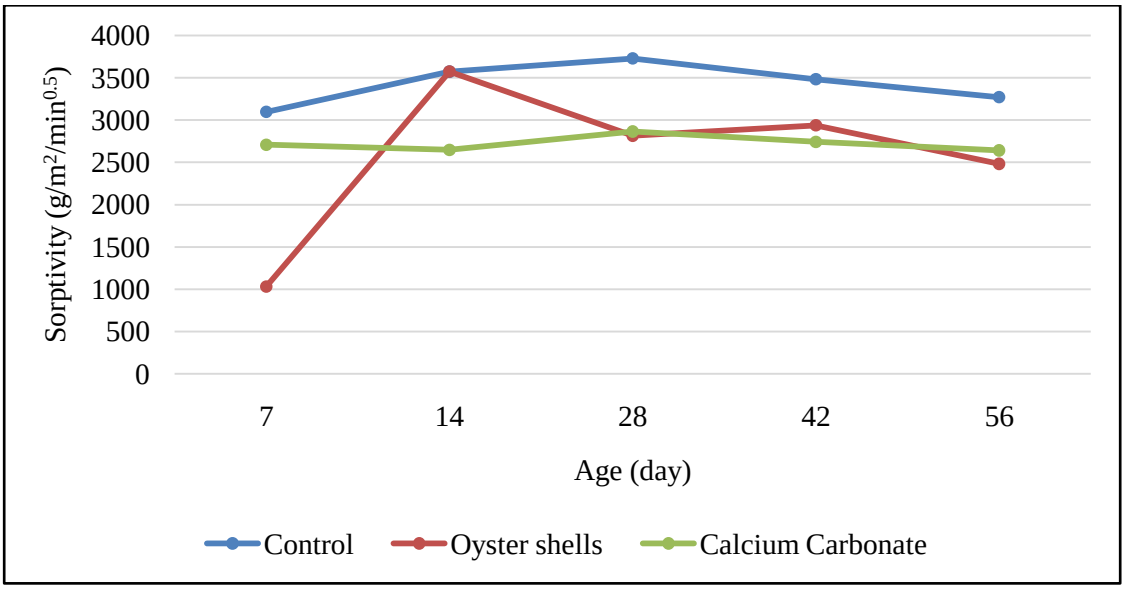

Figure 3: Sorptivity Coefficient

The seeding method used in this work, conferred a better performance to the plain lime mortar (control specimen) appraised through to the water absorption by capillary represented by the sorptivity coefficient; one of the parameters used to foresee durability. Analyzing the evolution of the water absorption by capillary of mixtures from the age of 7 days until 28 days, it is noticed with the evolution of hydraulic lime hydration, a modification on the microstructure, the calcite materials fill the capillary pores better than plain mortar. Since the calcite materials used were finer than the aggregate replaced, pores in bulk paste or in the interfaces between aggregates and lime binder is filled by this calcite addition hence, the capillary pores are reduced.

Flexural and Compressive Strength

The experiment results indicate a clear difference between the specimens subjected to plain mortar (control) and calcite specimens (Figure 4 and 5).

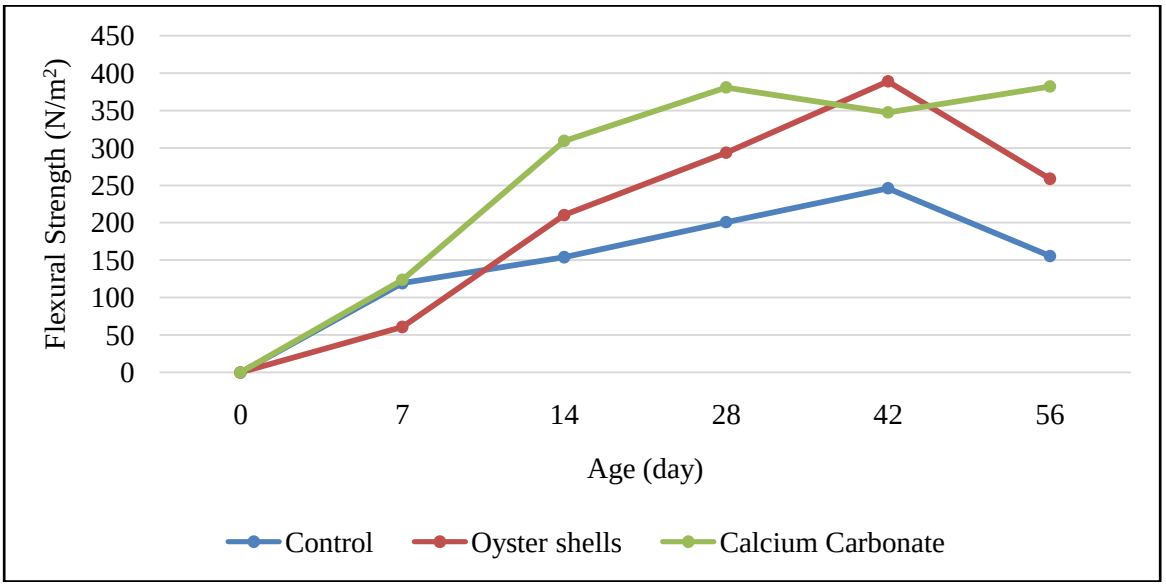

Figure 4: Flexural Strength

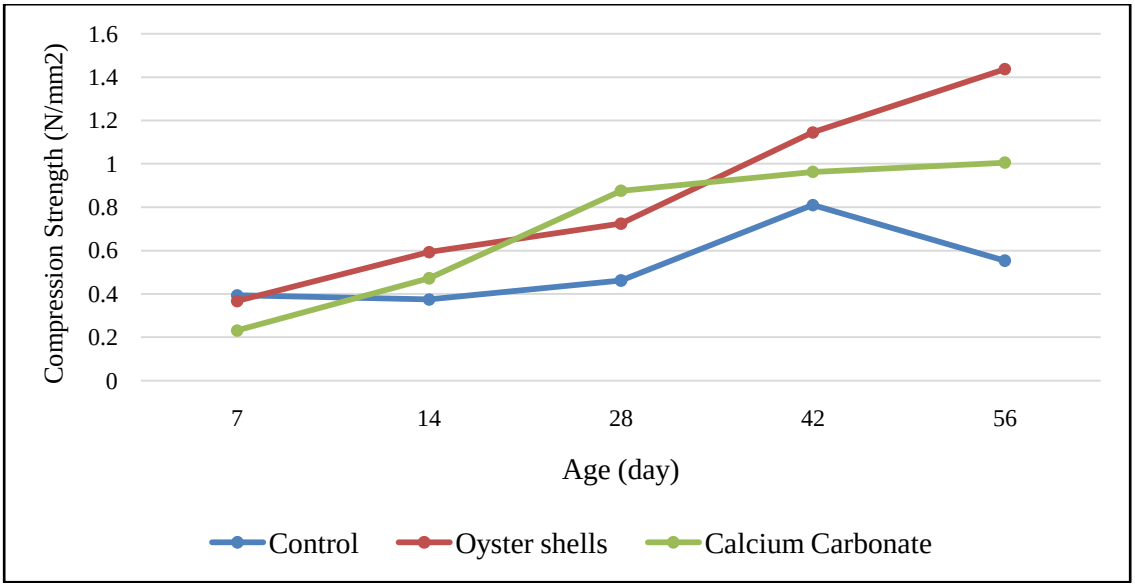

Figure 5: Compressive Strength

Figure 5 shows the variation of the compressive strength development of calcite mortars with curing time and temperature of 33°C and 90% RH. For control specimen, the inconsistency of gaining and loss of strength is at 14, 28, 42 and 56 days were very noticeable. However, the presence of the calcite in the mixtures reduces the negative effect of the temperature rise, and the longer the specimens are exposed the lesser the effect would be. After rapid strength gain for the first 7 days (steep gradient), the calcite mortar undergoes a plateau value of its ultimate strength and gaining strength again after 42 days. These results are confirmed by K. Ezziane *et al.* (2007).

Conclusion

This study has allowed to valorise the usage of calcite and to analyse its behaviour under elevated temperatures and high humidity. As been stated before, mortars exposed to high temperature will generates rapid compressive strength at early age but a decrease at late age. When a part of the cement is substituted by the calcite, the experimental results show an increase of the activation energy. This explains the slowness of its hydration and its beneficial effect in hot climate.Under elevated temperatures, the fast formed hydrates cause a great porosity and a loss of ultimate strength as reported by several authors. This phenomenon improves with seeding method; inclusions of calcite crystals by replacing 6% of aggregates weight with calcite. In addition to the volume percentage or weight percentage pores in the mass, their shape and interconnectivity will be also relevant in determining the permeability of the material and therefore the carbonation extent.

References

[1] Allen, G. 2003. *Hydraulic lime mortar for stone, brick and block masonry,* Shaftesbury, Donhead.

[2] Ball, R. J., El-turki, A. & Allen, G. C. Influence Of Carbonation On The Load Dependant Deformation Of Hydraulic Lime Mortars. 11th International Conference on Non-conventional Materials and Technologies (NOCMAT 2009), 2009 Bath, UK.

[3] Ball, R. J., El-Turki, A. & Allen, G. C. 2010. Influence of carbonation on the load dependent deformation of hydrauliclime mortars. *Materials Science and Engineering: A,* 528.

[4] British Standards, I. 2010a. BS EN 459-1: Building Lime Part 1. Definitions, specifications and conformity criteria. UK: British Standards.

[5] British Standards, I. 2010b. BS EN 459-2: 2010 Building lime. Test Methods. UK: BSI.

[6] Cocks, F. H. 2009. *Energy Demand and Climate Change: Issues and Resolutions,* Durham, NC USA, Wiley-VCH. Copyright.

[7] Desai, S. N., Patel, S. & Patil, H. H. 2011. Elevated Temperature Curing of Concrete with Industrial Wastes. *International Journal of Advanced Engineering Technology,* 2.

[8] Dheilly, R. M., Tudo, J., Sebai BI, Y. & Quéneudec, M. 2002. Influence of storage conditions on the carbonation of powdered $Ca(OH)_2$. *Construction and Building Materials,* 16.

[9] Dias, W. P. S. 2000. Reduction Of Concrete Sorptivity With Age Through Carbonation. *Cement and Concrete Research,* 30.

[10] Dotter, K. R. 2010. Historic Lime Mortars: Potential of Local Climate on the Evolution of Binder Morphology and Compostion. *In:* Smith, B. J., Robin, C. & Gomez-Heras, M. (eds.) *Limestone in the Built Environment: Present-day Challenges for the Preservation of the Past.* Bath, UK: The Geological Society Publishing House.

[11] El-Turki, A., Ball, R. J. & Allen, G. C. 2007. The influence of relative humidity on structural and chemical changes during carbonation of hydraulic lime. *Cement and Concrete Research,* 37.
[12] Ezziane, K., Bougara, A., Kadri, A., Khelafi, H. & Kadri, E. 2007. Compressive strength of mortar containing natural pozzolan under various curing temperature. *Cement & Concrete Composites,* 29.
[13] Forster, A. M. 2004. *How hydraulic lime binders work : hydraulicity for beginners and the hydraulic lime family,* Edinburgh, Love Your Building Publishing.
[14] Forsyth, M. 2008. Materials and skills for historic building conservation. Oxford,UK: The Blackwell Publishing.
[15] Hall, C. & HOFF, W. D. 2002. *Water Transport in Brick, Stone and Concrete,* London, Taylor & Francis.
[16] Lanasa, J., Pe´rez bernalb, J. L., Bellob, M. A. & Alvarez galindo, J. I. 2004. Mechanical properties of natural hydraulic lime-based mortars. *Cement and Concrete Research,* 34.
[17] Lawrence, R. M. H. 2006. *A study of carbonation in non-hydraulic lime mortars.* Doctor of Philosophy, University of Bath.
[18] Lo, Y. & Lee, H. M. 2002. Curing effects on carbonation of concrete using a phenolphthalein indicator and Fourier-transform infra-red spectroscopy. *Building and Environment,* 37.
[19] Nesse, W. D. 2000. *Introduction To Mineralogy,* New York, USA, Oxford University Press, Inc.
[20] Pacheco Torgal, F., Miraldo, S., Labrincha, J. A. & De Brito, J. 2012. An overview on concrete carbonation in the context of eco-efficient construction: Evaluation, use of SCMs and/or RAC. *Construction and Building Materials,* 36.
[21] Pavia, H. S. 2008. A study of the workability of natural hydraulic lime mortars and its influence on strength. *Materials and Structures,* 41.
[22] Sickels-Taves, L. B. & Sheehan, M. S. 2002. Specifying Historic Materials: The Use of Lime. *In:* Throop, D. & Klinger, R. E. (eds.) *Masonry: Opportunities For The 21st Century.* Salt Lake City, Utah, USA: ASTM.
[23] Tanabe, T.-A. 2008. Influence of Local Climate Conditions on the Carbonation Rate of Concrete. *In:* Tanabe, T., Sakata, K., Mihashi, H., Sato, R., Maekawa, K. & Nakamura, H. (eds.) *Creep, Shrinkage and Durability Mechanics of Concrete and Concrete Structure.* London, UK: Taylor & Francis Group.
[24] Wilkinson, S. 2009. *A Study of the Moisture Buffering Potential of Hemp in Combination with Lime and Clay-Based Binders* MSc Architecture: Advanced Environmental and Energy Studies, University of East London.

Effects of Carbon Nanotubes Addition in Sn-5Sb/Cu Solder Joint

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Abstract: The influence of multi-walled carbon nanotubes (MWCNT) on the melting temperature and interfacial IMC layer of the Sn-5Sb/Cu joints with three different weight percentages (wt.%) of MWCNTs was investigated. The melting temperature of composite solders was observed to slightly decrease when compared with the plain solder due to high surface free energy of MWCNTs in the solder matrix. Respective solder-copper joints were subjected to isothermal aging to investigate the evolution and growth of the intermetallic compound (IMC) layer. The IMC results revealed that the as reflowed samples showed a marginal IMC suppression in the composite solders with respect to the plain solder. While results for the IMC suppression became appreciable in the composite solders after 500h of isothermal aging.

Introduction

For the production of good and dependable interconnects, the intermetallic compound (IMC) stratum formed between the solder alloy and substrate must possess desirable characteristics. Naturally, the brittle nature of IMC increases as the layer thickness increases thereby enhancing the propensity to generate defects such as cracks or coarseness of microstructural grains. Laurila et al. [1] indicated that a way to influence the interfacial reactions and the resulting product layers in a given system is to alloy either metallization (conductor) or solders with small amounts of additional elements. In recent times, nano-sized particles have been employed as reinforcement materials in lead free solders due to their effectiveness in restricting grain boundary sliding through uniform distribution of the particles at the grain boundaries [2].In the studies of Xu et al. [3], the authors revealed that the subjection of both solder samples to 336 h aging process under a continuous current density of 1.2×10^4 A/cm^2 resulted in formation of less thick overall IMCs (Cu_6Sn_5 and Cu_3Sn) in composite solders than those of the plain solder. The current study therefore attempts to offer a promising candidate in the form of a composite solder with MWCNTs reinforcement to upgrade the intrinsic properties of the conventional Sn-5Sb lead free solder as well as produce a candidate that can decently operate in a robust environment.

Experimental procedures

Multi walled carbon nanotubes (MWCNTs) as shown in Fig. 1, were utilized in this study. The MWCNTs have a diameter range of 15-20nm and length of 0.5-2µm. The Sn-5Sb, Sn-5Sb-0.01CNT and Sn-5Sb-0.1CNT solder samples were prepared by the ball milling process at 800rpm for 6h. The milled powders were cold compacted using a pressure of 80MPa to form a 20mm solder tablet. Differential scanning calorimetry (DSC) was used to study the melting properties of the solder samples. The samples were heated till 600°C at 10°C/min heating rate under a protective nitrogen environment. Solder samples were reflowed on a pure (99.99%) Cu substrate with a thin layer flux application. Thereafter, some of the samples were subjected to solid-state aging for 500 hours.

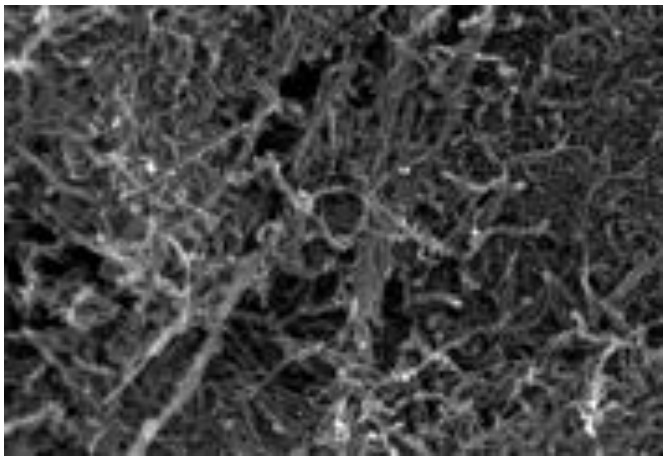

Fig. 1 SEM image of MWCNTs

Results and discussion

Melting temperature analysis

A major cardinal property to be considered in the packaging of electronic hardware is indisputably the melting temperature of the interconnection materials. Table 1 presents the values of the melting point (T_m), solidus temperature (T_{end}), liquidus temperature (T_{onset}) and pasty range of both the plain solder and composite solders as retrieved from the DSC scans.It can be observed that, with the 0.1wt% MWCNTs addition to the solder matrix, both the onset temperature (T_{onset}) and melting temperature (T_m) results of the composite solder exhibited the lowest value amidst the other samples. It is noteworthy that the melting temperature of the 0.1wt% MWCNTs reinforced solder sample dropped by2.72°C from 241.31°C for the plain solder to 238.59°C. As it was observed in the literature [4,5], the marginal decrease in the melting point of the composite solders could be linked with the higher surface free energy potentials of MWCNTs in the solder matrix which in other words have tendencies of exciting an increase in the surface instability of the composite solders.

Table 1 Thermal parameters for plain and composite solders from DSC scans.

Sample	Peak Temperature. T_m (°C)	Solidus Temperature T_{end} (°C)	Liquidus Temperature T_{onset} (°C)	Pasty Range
Sn-5Sb	241.31	253.42	231.80	21.62
Sn-5Sb-0.01CNT	240.45	253.00	231.36	20.64
Sn-5Sb-0.05CNT	240.95	253.39	231.98	21.41
Sn-5Sb-0.1CNT	238.59	250.33	230.97	19.36

Interfacial IMC formation

The representative optical micrographs for the intermetallic evolution results of as reflow and 500h aged samples are presented in Fig 2. As exhibited in the micrographs of the respective aging time, an appreciable suppression in the IMC growth has been detected in the composite solders with the Sn-5Sb-0.05CNT solder sample having the least IMC growth for the subjected conditions

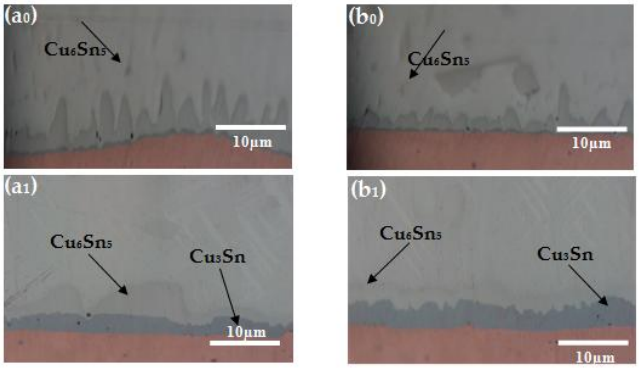

Fig 2. Optical micrographs of interfacial IMC layer of (a) Sn-5Sb (b) Sn-5Sb-0.05CNT solder joint samples. Subscripts 0 and 1 represents the as reflow and 500h (Aging 170°C)

Figure 3 illustrates the average total IMC thickness results versus the plain and composite solders solder samples. From the plots, it can be deduced that the composite solders exhibited a considerable degree of suppression when compared with the plain solder. A minimal retardation in the total intermetallics was observed in the composite solders with respect to the plain solder for the reflowed samples, where IMC thicknesses of 2.60µm and 3.04µm were recorded for the Sn-5Sb-0.05CNT and Sn-5Sb solder samples respectively.For the 500h isothermally aged samples, the overall IMC thicknesses for the composite solder samples showed a remarkable decrease with the Sn-5Sb-0.05CNT sample having a value of 6.73µm which indicates 0.96µm reduction in the value of the plain solder. Hence, it is proposed in this study that the high potentials of the Sn-Sb alloy in dissolving the Cu substrate was cushioned by the CNTs distribution in the solder matrix which serves as inhibitors that impedes the diffusion of Sn atoms which is pivotal in the Cu_3Sn IMC formation. This finding is in line with the investigations of [6,7].

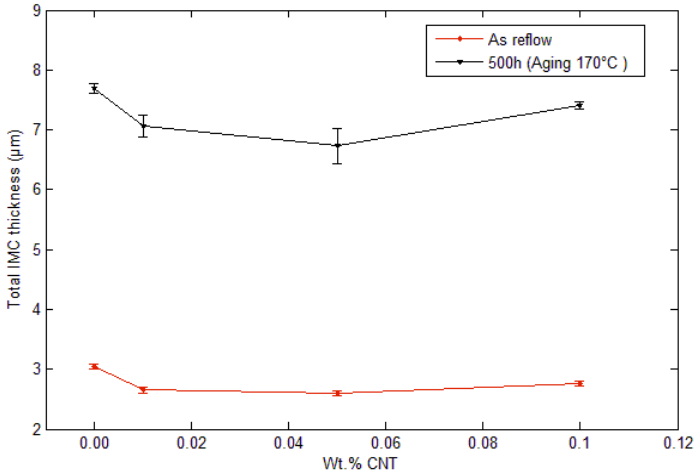

Fig 3. Total IMC thickness layer against the plain and composite solders subjected to as reflow and aging conditions

Conclusion

The Sn-5Sb/Cu joints, with varying wt.% of carbon nanotubes were developed through the powder metallurgy route. The melting properties and interfacial IMC layers for the plain and composite solders were investigated. The melting temperatures of composite solders were observed to slightly decrease due to high surface free energy of MWCNTs in the solder matrix. More so, the interfacial IMC thickness of plain and composite solder joints where compared. It was noted that the as reflowed samples showed a marginal IMC suppression in the composite solders with respect to the plain solder. However, the IMC suppression became remarkable in the composite solders after 500h of isothermal aging.

References

[1] Laurila, T., Hurtig, J., Vuorinen, V., & Kivilahti, J. K. (2009).*Microelectronics Reliability*, *49*(3), 242–247.

[2] Shen, J., & Chan, Y. C. (2009). *Microelectronics Reliability*, *49*(3), 223–234.

[3] Xu, S., Cheong, Y., Zhang, K., & Yung, K. C. (2014). *Journal of Alloys and Compounds*, *595*, 92–102.

[4] Kumar, K. M., Kripesh, V., & Tay, A. a. O. (2008). *Journal of Alloys and Compounds*, *450*(1-2), 229–237.

[5] Niranjani, V. L., Rao, B. S. S. C., Singh, V., & Kamat, S. V. (2011).*Materials Science &Eng A*, *529*,257-264.

[6] Nai, S. M. L., Wei, J., & Gupta, M. (2009). *Journal of Alloys and Compounds*, *473*(1-2), 100–106.

[7] Yang, Z., Zhou, W., & Wu, P. (2014). *Materials Science & Engineering A*, *590*, 295–300.

Study on Lead Free Composite Solders (Bi- 205Ag) as a Replacement for Classic Solder at High Temperature Application

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Abstract. These days the electronic gadgets are nearly everywhere. The electronic parts of these gadgets are assembled and connected to each other by soldering technology. The classic soldering alloys contains high level of lead. The development of lead-free solders has become an important task for material scientists due to health and environmental concerns regarding the high toxicity of lead. Adding carbon nanotube or graphene to the lead free soldering alloys may improve the metallurgical behavior of these alloys.

Keywords: lead free soldering alloys, CNT, Graphehe

Introduction

Lead– Tin (PbSn) solders, for metal interconnections, were first used about 2000 years ago. Currently, the usage of solders has become indispensable in the interconnection and packaging of virtually all electronic devices and circuits. Pb-bearing solders and especially the eutectic or near-eutectic SnPb alloys have been used extensively in the assembly of modern electronic circuits [1]. Research into the science and engineering of soldering has taken a major change in direction ever since the mandatory requirements for Pb-free imports introduced by countries such as Japan and some other in the European Union [2-3]. These compulsory requirements are due to the fact that Pb (and Pb-containing compounds), as cited by the Environmental Protection Agency (EPA) of the US, is one of the top 17 chemicals posing the greatest threat to human beings and the environment [4-5]. In the electronics industry, the Pb generated by the disposal of electronic assemblies is considered as hazardous to the environment. In Japan, the legislation prohibiting Pb from being sent to landfills and other waste disposal sites is already in place. In the US, legislations in limiting the use of Pb have been introduced in both the Senate and the House of Representatives. The legislation includes (a) H.R. 2922, The Lead Based Paint Hazard Abatement Act of

1991; (b) S. 391, the Lead Exposure Reduction Act of 1991, and (c) H.R. 3554, the Lead Exposure Act of 1992 [6,7]. Although these bills have not been passed yet, it is likely that some forms of bills will be passed in the future. The European Union has officially designated 1 July 2006 as the date when the Directive on the Restriction of Hazardous Substances in Electrical and Electronic Equipment will require ‘‘the use of lead, mercury, cadmium, hexavalent chromium, and halogenated flame retardants’’ be phased out [4]. This requirement applies to the manufacturing of both domestic and export products. Electronics manufacturers now have two choices: either attaining 100% recycling of Pb, or using Pb-free solder alloys [8-9].

High-temperature solders are widely used as die-attach solders in power semiconductor packaging[10]. The application of powerelectronics has been extended to a variety of automotive, aerospace and energy production industries [11]. With the miniaturization drive and increasing power of power electronics, hightemperature operations have become a serious issue. As a response to the growing demand for high-temperature operations, next generation power semiconductors such as SiC and GaN and packaging materials such as AlN and Si_3N_4 have been developed for high-temperature applications. Thus, high-temperature solders are indispensable for power semiconductor packagings. High-lead content solders are currently being used as high-temperature solders [12]. Despite numerous studies on lead-free solders in the recent years, development of high-temperature lead-free solder alloys alternatives [13]. The reason is the volume of high-lead content solders used is very little in comparison with the volume of the eutectic Pb-Sn solder alloy. However, with the advent of high-temperature applications, the usage of high-lead content solders is sharply increasing [14]. Thus, the establishment of high-temperature lead-free solders or other interconnection technologies has been an urgent priority in the electronics industries [12].

Bi based alloys

Bi is the least toxic of the heavy metals and with a melting point of 270 ^{0}C is a natural choice for high-temperature lead-free solder alternatives [15]. The development of Bi based alloys for Pb-free high-temperature soldering is severely hindered by the poor thermal and electrical conductivity possessed by the Bi rich phases which is critical for the solder alloy. The thermal conductivity of Bi (k = 8 W/m K) is much lower when compared to the existing high-lead content solders like Pb–5Sn (k = 35W/m K) [16]. It has been well documented that the addition of Ag to Bi would slightly improve the thermal and electrical conductivity of the alloy [15,17,18]. The Bi–Ag system has a eutectic melting point of 262 ^{O}C corresponding to 2.5 wt% Ag. However, further increasing or decreasing the Ag content from the eutectic point entails an increase of only the liquidus temperature and not the solidus temperature. The microstructure of the Bi–Ag consists of Ag rich dendrites dispersed on the matrix (Bi) phase [15]. Coupled growth of the eutectic phases has not been reported since the eutectic point lies on the region with a very high Bi content. Alloying small amounts of Ag cannot drastically improve the thermal and electrical conductivity of the alloy. Bi–2.5Ag alloy also possesses a higher wetting angle of 39^O when compared to the existing

high-lead content solders like Pb–5Sn (16^{O}) on a Cu substrate [18]. Bi–2.5Ag is the only possible candidate alloy in addition of being close to the required solidification criterion; it is also close in hardness to the existing high-lead content solders [15,18]. Despite this advantage it cannot be used as an alternative since high-temperature solders are currently being employed in applications that generate high amounts of heat and it demands a high heat conductivity of the solder.

Mechanical and Physical Properties

The melting point should be low enough to avoid thermal damage to the assembly being soldered and high enough for the solder joint to bear the operating temperatures [9]. Although several commercial and experimental Sn-based Pb-free solder alloys exist, none meets all the above requirements, if the melting point of the candidate alloy is required to be very close to that of PbSn. Nevertheless, they also need to possess the desirable material properties, reliability and manufacturability. Knowing this, and coupled with a tight timetable to meet legislative requirements, industries are prepared to get round the problems by providing supporting processing equipment and components to meet the increase in the processing temperature. It has been reported that Japanese companies have provided a strong driving force for Pb-free manufacturing and accelerated the useage of Pb-free solders [19].

Modern electronic assemblies are made up of materials with a wide range of thermal expansion coefficients. When an electronic device is in operation, it normally generates heat. As these materials are held together, the physical constraint provides internal thermal stress and strain during operation. A prime objective in terms of material selection for electronic assembly is to provide the least amount of thermal mismatch. However, in practice, this cannot be fully achieved; therefore thermal stresses in an assembly, including the interconnection materials, are unavoidable. It is therefore important for solder alloys, as prime interconnection materials, to be able to withstand the required thermal stresses. It is also noted that during the operation, the absolute temperature is very close to the absolute melting temperature of the solder. Thus, the solder material is normally subjected to creep during operation [9]. With adequate design, PbSn solder is capable of withstanding normal operating stress, strain, and creep deformation. Needless to say, Pb-free solder alloys should have adequate mechanical strength such as tensile, compressive and creep resistance. By adding Carbon nanotube or Graphene due to theirs electric conductivity and mechanical property the feature of alloys can be improve [20, 21, 22, 23]. The research which is done by Lui et al , X. D., indicate that adding Graphene improved the UTS of the composite and the ductility of composite decreased with the addition Graphene.

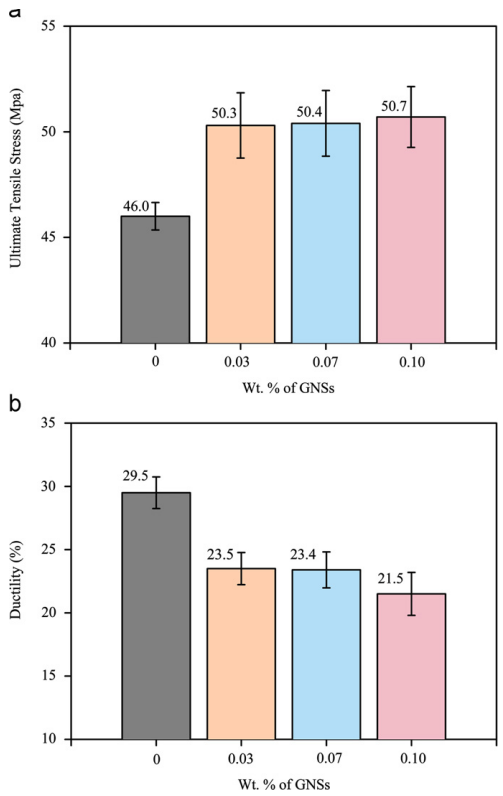

Fig 1. (a) UTS and (b) ductility of the composite solders as a function of weight percent of the added [24].

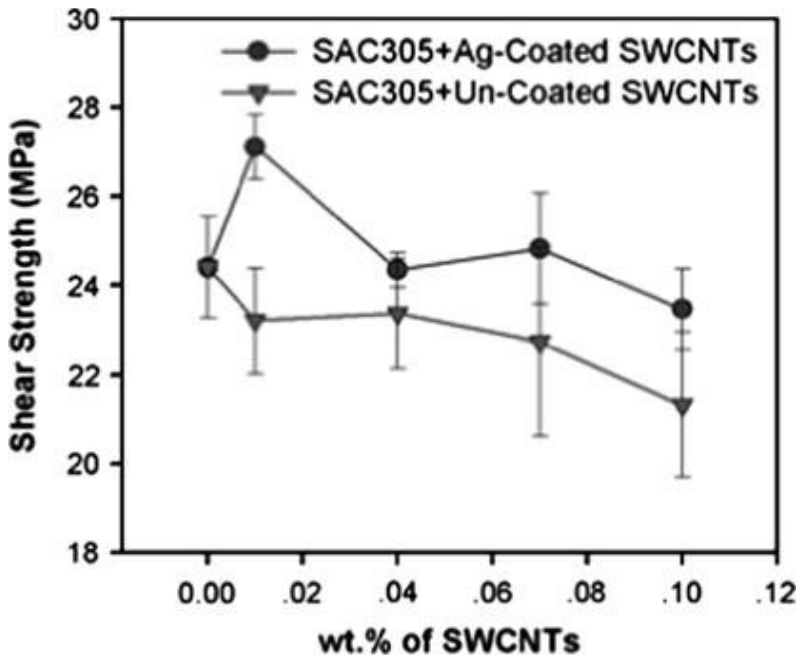

Fig 2. Shear strength of composite solder joints as a function of wt% SWCNTs [25]

Chantramanee et al. find that A shear strength improvement of 11 % was observed with the addition of only 0.01 wt% Ag-coated SWCNTs in the composite solder—this was the smallest loading in the experimental design, and optimizing the loading for shear strength might give even larger improvements. The Ag-coated SWCNTs had a clustering tendency observed at the higher loadings that may have reduced [25]

Summary

Soldering is a metallurgical process for joining metal parts which uses molten filler metal to wet the surfaces of joint that provide the conductive path in order to connect one circuit element to another. Tin – lead alloys are widely used as solder in the industry especially electronic industry. The common Tin-lead [(95-5)% or (90-10)%] alloys are normally used for high temperature soldering application. Bi alloy base lead free soldering alloys are one of the alternatives for replacing the classic solder due to its mechanical and physical behavior. Besides, it is less harmful to nature compared to lead. The metallurgical properties such as mechanical strength of lead free alloys composite at high-temperature applications (260 ^{o}C) which reinforced by adding Graphene or carbon nanotube can be improved regarding the properties of Graphene and CNT. The mechanical strength of Graphene and CNT help the matrix to show better properties.

References

[1] Tu, K.N. and K. Zeng, *Tin-lead (SnPb) solder reaction in flip chip technology.* Materials Science and Engineering: R: Reports, 2001. 34(1): p. 1-58.

[2] Shangguan, D. (Ed.). (2005). Lead-free solder interconnect reliability. ASM international.

[3] Ganesan, S., & Pecht, M. G. (Eds.). (2006). Lead-free electronics. John Wiley & Sons.

[4] Chen, X., et al., *Study on the properties of Sn–9Zn-Cr lead-free solder.* Journal of Alloys and Compounds, 2008. 460(1): p. 478-484.

[5] Subramanian, K. V. (2007). Lead-Free Electronic Solders: A Special Issue of the Journalof Materials Science: Materials in Electronics (Vol. 11). Springer Science & Business Media.

[6] Matsugi, K., et al., *Long Time Stability of Pb-Free Sn-9 Zn Elements for AC-Low Voltage Fuse Performance.* Materials Transactions, 2011. 52(4): p. 753-758.

[7] Bath, J. (2007). *Lead-free soldering*. Springer Science & Business Media.

[8] Suganuma, K. (2003). *Lead-Free Soldering in Electronics: Science, Technology, and Environmental Impact*. CRC Press.

[9] Henshall, G., Bath, J., & Handwerker, C. A. (Eds.). (2011). *Lead-free solder process development*. John Wiley & Sons.

[10] Wu, C., et al., *Properties of lead-free solder alloys with rare earth element additions.* Materials Science and Engineering: R: Reports, 2004. 44(1): p. 1-44.

[11] Chidambaram, V., J. Hattel, and J. Hald, *High-temperature lead-free solder alternatives.* Microelectronic Engineering, 2010.
[12] Kim, S.J., et al., *Characteristics of Zn-Al-Cu Alloys for High Temperature Solder Application.* Materials Transactions, 2008. 49(7): p. 1531-1536.
[13] Yamada, Y., et al., *Pb-free high temperature solders for power device packaging.* Microelectronics and reliability, 2006. 46(9-11): p. 1932-1937.
[14] Abtew, M. and G. Selvaduray, *Lead-free solders in microelectronics.* Materials Science and Engineering: R: Reports, 2000. 27(5-6): p. 95-141.
[15] Suganuma, K., S.J. Kim, and K.S. Kim, *High-temperature lead-free solders: Properties and possibilities.* JOM Journal of the Minerals, Metals and Materials Society, 2009. 61(1): p. 64-71.
[16] M. Rettenmayr, P. Lambracht, B. Kempf, M. Graff, Adv. Eng. Mater. 7 (10) (2005) 965–969.
[17] J.N. Lalena, N.F. Dean, M.W. Weiser, Electron. Mater. 31 (11) (2002) 1244–1249.
[18] Y. Yamada, Y. Takaku, Y. Yagi, Y. Nishibe, I. Ohnuma, Y. Sutou, R. Kainuma, K. Ishida, Microelectron. Reliab. 46 (9–11) (2006) 1932–1937.
[19] Y. Shi, W. Fang, Z. Xia, Y. Lei, F. Guo, X. Li, Mater. Sci: Mater. Electron. 21 (9)(2010) 875–881.
[20] Tu, K. and K. Zeng. *Reliability issues of Pb-free solder joints in electronic packaging technology*.2002: IEEE.
[21] Zeng, G., McDonald, S., & Nogita, K. (2012). Development of high-temperature solders: Review. Microelectronics Reliability, 52(7), 1306-1322.
[22] Chidambaram, V., Hattel, J., & Hald, J. (2011). High-temperature lead-free solder alternatives. Microelectronic Engineering, 88(6), 981-989.
[23] Nai, S. M. L., Wei, J., & Gupta, M. (2006). Improving the performance of lead-free solder reinforced with multi-walled carbon nanotubes. Materials Science and Engineering: A, 423(1), 166-169.
[24] Liu, X. D., Han, Y. D., Jing, H. Y., Wei, J., & Xu, L. Y. (2013). Effect of graphene nanosheets reinforcement on the performance of Sn瑕Ag瑕Cu lead-free solder. Materials Science and Engineering: A, 562, 25-32.
[25] Lau, A. K. T., & Hui, D. (2002). The revolutionary creation of new advanced materials—carbon nanotube composites. Composites Part B: Engineering, 33(4), 263-277.

Porous bioceramics in bone implants, methods: review

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Abstract: Because of the evolution in biomaterials and its ability to meet the need, especially in themedical field and in particular in the implants of the damaged parts of the body or those lost its continuityin the work because of the factors which exposed to the human body. In the present short review, somemethods that are used to form porosity in bioceramic materials have been studied.

Key words: bioceramics, porosity, bone implants.

Introduction

The synthetic or natural materials that are appropriate for entrance into living tissue, especially aspart of a medical device are now called biomaterials [2]. There are many types of biomaterials.Bioceramics one of these types that are specially designed for reconstruction and repair of damaged ordiseased parts, osteoporosis and fracture of the body such as hydro -xyapatite and tri calciumphosphates[3, 4]. Three basic types of bioceramics known presently are the bio restorable ceramics thatactively participate in the metabolic processes of an organism such as calcium phosphate ceramics, the bioactive ceramics which form direct chemical bonds with bone or even with the soft tissue of a livingorganism such as bio glass and glass ceramics [5, 6] the inert bioceramic that has high strength such asand Zirconia [7] and carbon [6, 8, 9]. The porous ceramic manufacturing processes have been subject tomuch consideration in order to meet the wide range of property demands of specific applications. Inaddition, providing the structure for reinforcement in a ceramic composite facilitates the tissue growth ina bio scaffold, insulating properties at high temperature and filter impurities are the main functions of thepores [10].In recent years, several researchers have reviewed the topic of porous materials, Shuilin Wua etal, have overviewed the design of ideal bio mimetic porous scaffolds [7], E.C. Hammel, et al haveoverviewed applications of porous ceramics developing [10], Min Wang have overviewed

compositescaffolds for bone tissue engineering[11], while Darmawati Mohamad Yunos,et al. have representedthe optimal solution and the scaffolds of choice for bone regeneration strategies.[1] and Molly M. Stevenshave overviewed biomaterials for bonetissue engineering[12] . In the present study, some routs that havebeen used to manufacture the porosity in bio ceramic bone implants were reviewed.

Porosity techniques in bio-ceramics

Choosing porous ceramic materials for tissue engineering scaffolds is a practical option for the repair ofdamaged bone [10]. Ceramic, polymers, metals and composites are used to synthesize artificial boneimplants and are widely used for bone regeneration and reconstruction. These synthesized scaffolds canexhibit satisfactory mechanical properties and biocompatibility [10, 13]. Another important function ofthe scaffold provides the framework for the cells when tissues are formed [14, 15]. In particular, it hasbeen reported that the size increases in bone growth with increasing pore size [16, 17]. Many porousceramic scaffolds produced to date have exhibited strength in the range of 10–30MPa [7]. It is generallyagreed that there should be porous network of interconnected pores having a diameter in the range of 50 to 1000 mm [18].In addition ,to produce porous bioceramics, many techniques have been developed which can be divided into those employing synthetic processing routes and natural resources [10].Synthetic bioceramics have been widely used as bone substitutes and offer a good alternative to natural bone grafts . In contrast, the using of bio ceramic those natural resources (such as coral) in bone grafts implants is limited because of certain drawbacks such as biological variability, viral or bacterial supply difficulty, and contamination risks[5]. Several synthetic techniques have been developed for the fabrication of porous bioceramics such as, replication of polymer foams , freeze casting, gel-casting foaming[19]and foaming with employment of several pore-creating additives[20, 21] for example, (producing porous hydroxyapatite- based materials and fabrication of porous calcium phosphate bioceramics [5, 21].

Sacrificial template method

This method use sacrificial material or pore former to act as a placeholder within the slurry or ceramic powder .when the green body is formed, the pore former is removed to leave behind empty pores . Freeze casting is one particular method ,utilizes growing ice crystals in a ceramic slurry to form the pores in a ceramic body [10].In the other words, the technique involves the preparation of a biphasic composite comprising a homogeneously dispersed sacrificial phase in a continuous matrix of glass or ceramic particles as shown in fig.1[3]. The sacrificial phase is ultimately extracted from the partially consolidated matrix to generate pores within the microstructure. The removal of the sacrificial phase does not lead to flaws in the structure as is the case of positive replica methods. Therefore, the mechanical strength of the structures made by the sacrificial template method is usually higher than that of scaffolds fabricated by the replica method; however, porosity and pore interconnectivity are substantially lower. For example, hydroxyapatite porous bodies produced from wax and starch particles,

PMMA (polymethyl methacrylate) particles, as well as naphatane and sucrose as sacrificial materials have been made by this method [1].

Direct foaming method

Air is incorporated into a ceramic suspension , which is then set in order to create a structure of air bubbles in the direct foaming method approach as shown in fig .1 [10]. In most cases, to produce a high strength porous ceramic, the consolidated foams are afterwards sintered at high temperatures and the most critical process is stabilization of air bubbles in the initial suspension. The stability of the air bubbles can be achieved by various surfactants and particle stabilizers. The foam structures prior to solidification are important because they influence the microstructure, pore size , total porosity, and wall thickness of the final product. The average pore size can change from 10 to 300 μm, whereas the porosity of foams produced by this method typically varies between 40% and 93%. Sol–gel derived bioactive glass scaffolds and calcium phosphate and hydroxyapatite scaffolds obtained by gel-casting setting process are examples of scaffolds produced from the direct foaming technique [1]. In addition , gas foaming/particulate leaching method have been used to manufacture the highly porous scaffolds it is include mixing, pressing, using high pressure (e.g CO_2 gas), leached in liquid (e.g distilled water) [22].

Foam replication method (Polymer sponge method)

Foam replication method is the standard method for producing Alumina, Titania, zirconia and Bio glassfoams. The scaffolds produced by the replica method using polymer sponges as the synthetic templates[1,23]. Fig.1 shows the typical structure of a Bio glass- based glass–ceramic scaffold fabricated by thefoam replica method .

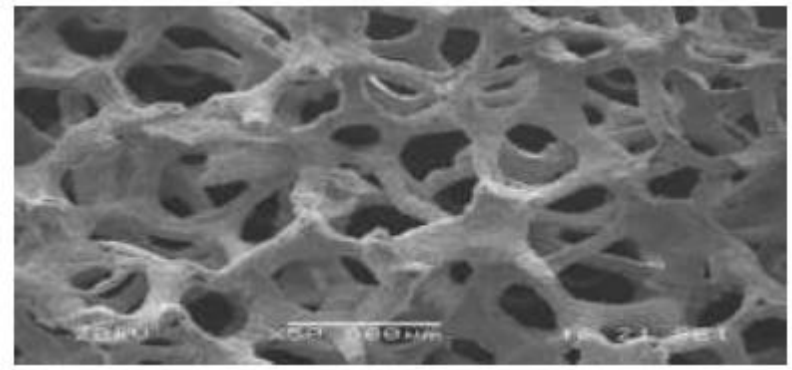

Fig.1 Macroporous structure of a Bioglass_based glass–ceramic scaffold fabricated by the foam replica methodusing PU sponge as sacrificial template [1]

To form macro porous ceramics by the replica method approach, can be used the natural (such as coraland wood) and synthetic templates (such as polymer foams and typically polyurethane) that give adesired macrostructure. In the beginning, the template is soaked into a ceramic suspension until the strutsare homogeneously coated with the ceramic material. In addition, to avoid dripping by thixotropic effects, the

coating should be viscous enough. Colloidal silica, clays, carboxymethyl cellulose and polyethyleneoxide in combination with conventional dispersants can be used as a thickening additives [1, 23].Moreover, in order to prevent cracking of the struts during the subsequent heat-treatment process, thebinders and plasticizers are added to the initial suspension. After that, the polymer template ceramiccoatedis dried and burnt out through careful heating between 300 and 800 C˚ and finally densified bysintering in an appropriate atmosphere at temperatures between 1,000 to 1,500 C ˚, depending on thematerial. Highly porous ceramics can be produced reaching open and interconnected porosity levels in therange 40–95% with sizes of pores between 200 μm and 3 mm. The tendency to produce a hole in thecentre of each strut resulting from the removal of the polymer skeleton on heating is one possibledisadvantage of this method. In spite of the negative affect of hole presence on the mechanical propertiesof the foams, the approach of filling the hole with a polymer, leads to improved mechanical behaviourexploiting the interaction between the ceramic and polymer phases [1].For example, the calciumphosphate porous ceramic manufactured by the technique of polycarbonate polyurethane sponge and timetracking sponge coating stents resulted in 90-65% porosity and has the compressive strength in rangefrom 0.3 to 3.3MPa [16].

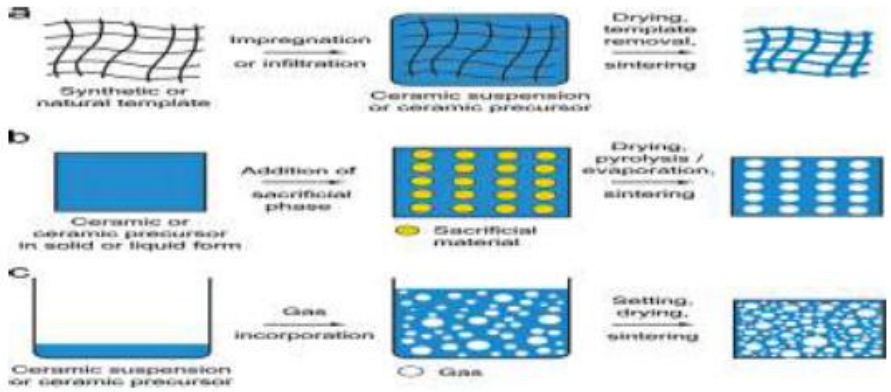

Fig.2 Typical processing methods for the production of macro porous ceramics :(a)replica technique;(b)sacrificial template technique and (c) direct foaming technique [10].

Freeze casting

By removing the frozen vehicle network via sublimation in this method, three-dimensionallyinterconnected pore channels are readily formed. To date, camphene and water have been successfullyadopted as the vehicle materials. Of these two materials, camphene can be frozen and easily sublimednear room-temperature, offering more flexibility in the process[24]. For example, Eun-Jung Leeet al. have been fabricated highly porous hydroxyapatite (HA) bioceramics using the camphene-based freezecasting method as shown in fig.3.

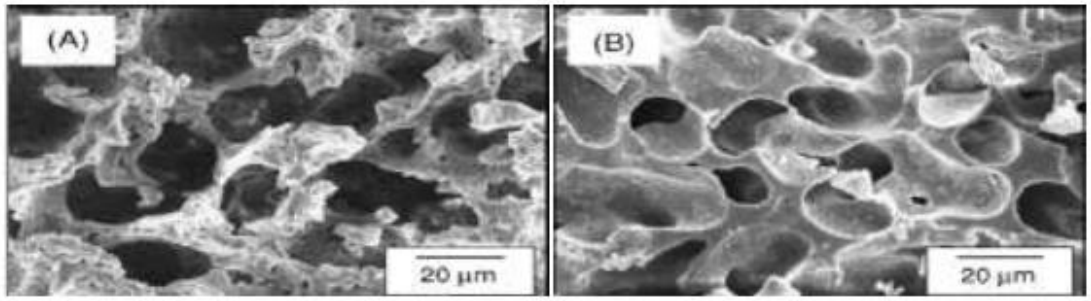

Fig. 3. SEM micrographs of the porous HA bioceramics with initial HA contents of (A) 10 vol.%, (B) 15vol.%, and .[24]

Gel-casting foaming

The gelling by in situ polymerization of concentrated ceramic slurry suspended in a monomeric solutionis called the gel casting process. In brief,the mix of bioceramics that dispersed in a monomer solution ispoured into a mold.The monomer is polymerized to freeze the particles and to form gel-like bondingphase (binders) .The bioceramics is remove from the mold while still wet, dried, heated to burn out theorganic additive and sintered at the final step[25, 26]. Compared with the slip casting process,thismethod can produce very uniform green bodies with high strength by forming organic networkencapsulating the ceramic particles. For example, S. Padilla el al, used the gel-casting method to preparehydroxyapatite (HA) pieces,the mechanical properties of the green bodies were higher than obtained byother methods[26]. In addition,there are many new routes to form macro porous bioceramics. In brief,one of these routes have been used by F. Stergioudia et al to form porosity (manufacturing of open-cellporous calcium phosphate). The manufacturing process consists of four stages: mixing, compaction,dissolution and sintering as shown in fig 4. The macro pore sizes, varying from 200 to 800 µ m, andporosities between 60–75% ensuring high interconnectivity of pores can be obtained by this process [5].

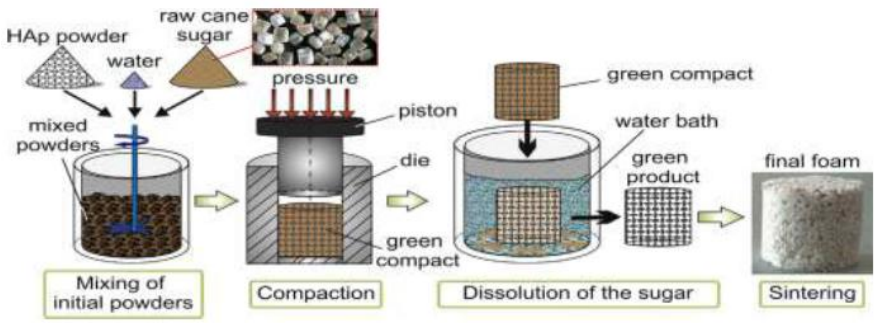

Fig. 4. Schematic presentation of the production method for manufacturing of open-cell porous calcium phosphate using crystalline raw cane sugar as a space holder material.[5]

Conclusion

In this brief review, some routes of manufacturing the porosity in bio ceramic materials have beenpresented because of the importance of these materials in the medical field, especially in bone implants.Some of the ways were conventional methods for example, sacrificial template method, direct foamingmethod and some of the methods were modern methods for example, freeze casting and gel-castingfoaming. The porosity of foams produced by this method typically varies between 40% and 93%, whereasthe average pore size can change from 10 to 300 μm. By using the polymer sponge method, highly porousceramics can be produced in the range of 40–95% with sizes of pores between 200 μm and 3 mm. For new routes for example, manufacturing of open-cell porous calcium phosphate, the macropore sizes varying from 200 to 800 μm and porosities between 60–75% ensuring high interconnectivity of pores obtained by this process.

Acknowledgements

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References

[1] D. M. Yunos, O. Bretcanu, and A. R. Boccaccini, "Polymer-bioceramic composites for tissueengineering scaffolds," *Journal of Materials Science,* vol. 43, no. 13, pp. 4433-4442, 2008.

[2] J. Chevalier, and L. Gremillard, "Ceramics for medical applications: A picture for the next 20years," *Journal of the European Ceramic Society*, vol. 29, no. 7, pp. 1245-1255, 2009.

[3] J. R. Woodard, A. J. Hilldore, S. K. Lan *et al.*, "The mechanical properties and osteoconductivityof hydroxyapatite bone scaffolds with multi-scale porosity," *Biomaterials*, vol. 28, no. 1, pp. 45-54, Jan,2007

[4] A. F. Khan, M. Saleem, A. Afzal *et al.*, "Bioactive behavior of silicon substituted calciumphosphate based bioceramics for bone regeneration," *Mater Sci Eng C Mater Biol Appl*, vol. 35, pp. 245-52, Feb 1, 2014.

[5] F. Stergioudi, A. Choleridis, E. Paulidou *et al.*, "Novel production and characterization of porouscalcium phosphate suitable for bone tissue engineering applications," *Ceramics International,* vol. 41, no.3, pp. 3822-3832, 2015

[6] S. R. Paital, and N. B. Dahotre, "Calcium phosphate coatings for bio-implant applications:materials *R: Reports,* vol.66, no. 1, pp. 1-70, 2009.

[7] S. Wu, X. Liu, K. W. K. Yeung *et al.*, "Biomimetic porous scaffolds for bone tissue engineering,"*Materials Science and Engineering: R: Reports,* vol. 80, pp. 1-36, 2014.

[8] T.Thamaraiselvi, and S. Rajeswari, "Biological evaluation of bioceramic materials-a review,"*Carbon*, vol. 24, no. 31, pp. 172, 2004.

[9] B. Caruta, *Ceramics and composite materials: new research*: Nova Publishers, 2006.

[10] E. C. Hammel, O. L. R. Ighodaro, and O. I. Okoli, "Processing and properties of advanced porous ceramics: An application based review," *Ceramics International*, vol. 40, no. 10, pp. 15351-15370, 2014.
[11] M. Wang, "Composite scaffolds for bone tissue engineering," *Am J Biochem Biotechnol*, vol. 2,no. 2, pp. 80-84, 2006.
[12] M. M. Stevens, "Biomaterials for bone tissue engineering," *Materials Today*, vol. 11, no. 5, pp.18-25, 2008.
[13] H. J. Haugen, M. Monjo, M. Rubert *et al.*, "Porous ceramic titanium dioxide scaffolds promotebone formation in rabbit peri-implant cortical defect model," *Acta Biomater*, vol. 9, no. 2, pp. 5390-9,Feb, 2013.
[14] Y. Song, Y. Ju, Y. Morita *et al.*, "Surface functionalization of nanoporous alumina with bonemorphogenetic protein 2 for inducing osteogenic differentiation of mesenchymal stem cells," *Mater SciEng C Mater Biol Appl*, vol. 37, pp. 120-6, Apr 1, 2014.
[15] G. Fostad, B. Hafell, A. Førde *et al.*, "Loadable TiO2 scaffolds—A correlation study betweenprocessing parameters, micro CT analysis and mechanical strength," *Journal of the European CeramicSociety*, vol. 29, no. 13, pp. 2773-2781, 2009.
[16] Y.-K. Jun, W. H. Kim, O.-K. Kweon *et al.*, "The fabrication and biochemical evaluation ofalumina reinforced calcium phosphate porous implants," *Biomaterials*, vol. 24, no. 21, pp. 3731-3739,2003.
[17] V. Komlev, and S. Barinov, " Novel bioactive hydroxyapatite-Base ceramics and gelatinimpregnatedcomposites.," *Ceramic and composite materials : new research* pp. 133, 2006.
[18] A. Butscher, M. Bohner, S. Hofmann *et al.*, "Structural and material approaches to bone tissueengineering in powder-based three-dimensional printing," *Acta Biomater*, vol. 7, no. 3, pp. 907-20, Mar,2011.
[19] Q. Chen, F. Baino, S. Spriano *et al.*, "Modelling of the strength–porosity relationship in glassceramicfoam scaffolds for bone repair," *Journal of the European Ceramic Society*, vol. 34, no. 11, pp.2663-2673, 2014.
[20] B. Jiang, X. Hu, and Z. Huang, "Porous bio-ceramic coating on zirconia formed through freezedrying,"*Materials Letters*, vol. 109, pp. 66-69, 2013.
[21] C. Zhou, P. Xie, Y. Chen *et al.*, "Synthesis, sintering and characterization of porous nanostructuredCaP bioceramics prepared by a two-step sintering method," *Ceramics International*, vol. 41,no. 3, pp. 4696-4705, 2015.
[22] D. G. Morales-Hernandez, D. C. Genetos, D. M. Working *et al.*, "Ceramic identity contributes tomechanical properties and osteoblast behavior on macroporous composite scaffolds," *J Funct Biomater*,vol. 3, no. 2, pp. 382-97, 2012.
[23] H. Haugen, J. Will, A. Köhler *et al.*, "Ceramic TiO2-foams: characterisation of a potentialscaffold," *Journal of the European Ceramic Society*, vol. 24, no. 4, pp. 661-668, 2004.
[24] E.-J. Lee, Y.-H. Koh, B.-H. Yoon *et al.*, "Highly porous hydroxyapatite bioceramics withinterconnected pore channels using camphene-based freeze casting," *Materials letters*, vol. 61, no. 11, pp.2270-2273, 2007.

[25] M. N. Rahaman, *Ceramic Processing and Sintering*: Taylor & Francis, 2003
[26] S. Padilla, M. Vallet-Regı, M. Ginebra *et al.*, "Processing and mechanical properties ofhydroxyapatite pieces obtained by the gelcasting method," *Journal of the European Ceramic Society*, vol.25, no. 4, pp. 375-383, 2005.

Plasticizing and Anti-Plasticizing Effect of Different Plasticizerson Tensile Properties of Sugar Palm Starch Films

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Abstract. The effect of different plasticizer type (glycerol (G), sorbitol (S) and glycerol/sorbitol (GS)) and concentration (15, 30 and 45, w/w %) on the tensile properties of biodegradable sugar palm starch films were evaluated. Regardless of plasticizer type, the tensile strength of plasticized SPS films decreasedwhereas, their flexibility and extensibility increased as the plasticizer concentrations were adjusted. However, the percent elongation of G and GS-plasticized films significantly reduced at higher plasticizer concentration of 45% (w/w) due to antiplasticization effect of plasticizers. GS-films with 30% (w/w) plasticizer content gave the best tensile properties.

Keywords: Sugar palm starch, Glycerol, Sorbitol, Plasticization, Antiplasticization

Introduction

Sugar palm (arenga pinnata) is known to be a versatile tree and indigenous to tropical countries, most especially in South-East Asia [1]. The tree is popular for its numerous economic uses. They provide significant products such as palm sap, fresh palm juices, palm neera, brown sugar, vinegar, palm wine, bio-ethanol and black fibers as raw materials for furniture making,brooms,handicraft, house construction, water resistant shipping ropes and other uses [2], [3]. Starch can also be obtained from sugar palm trunk, preferably when the tree no more produces sugar and fruits [4]. Sugar palm starch has been traditionally used as raw materials for glue substances [1]. However, it has not yet received much attention required for developing its potentiality as an industrial starch biopolymer.On the basis of biodegradability, availability, renewability, non-toxicity and affordability, starch is one of the most promising among all the potential biopolymer based packaging materials [6] . The use of starch in packaging promotes sustainability and addresses the negative impact non-

biodegradable plastics pose on the environment [10]. Thus, starch has attracted great deal of interest as potential alternative to conventional plastics for packaging applications.

Native starch based materials are reported in many investigations to be very brittle with many surface cracks and difficult to handle. However, these drawbacks can be resolve by the addition of plasticizers to pure starch to improve their workability and suppress film brittleness [11]. The ultimate role of plasticizers is to enhance the flexibility and processibility of starch by reducing the strong intermolecular interactions between starch molecules [12]. The most common plasticizer utilize for starch based films are polyols such as glycerol and sorbitol, amongst many others. Several researches have been conducted regarding the effect of polyols (most especially glycerol and sorbitol) on the properties of films based on various starch origin [13]–[16]. Nonetheless, there is very little work done on sugar palm starch based films. Hence, the aim of this current study is to investigate the effect of using different plasticizers (glycerol, sorbitol or their combination) on the tensile properties of sugar palm starch films.

Materials and Methods

Materials

The native sugar palm starch utilized in the study was extracted from the core of a sugar palm tree at Jempol, Negeri Sembilan (Malaysia). The starch extraction method was adapted from Sahari et al. [1] with slight modifications. Glycerol and sorbitol were used as film plasticizers and were supplied by LGC Scientific (Selangor, Malaysia). Distilled water served as the solvent for preparing filmogenic solutions.

Methods

Film preparation

Pure sugar palm starch was weighed and dissolved in distilled water to obtain film forming suspension (8 w/w % starch concentration). The film forming suspension was heated at 95 ± 2°C for 15 mins under continuous stirring before adding 30 % of plasticizer. The plasticized solution was later allowed to cool down to 40 °C. Film forming solution was casted on petri-dishes (10 cm diameter) and let it dry for 24 h.

Tensile properties

The mechanical properties of the films were tested using a standard method D882-02 (ASTM, 2002). Tensile strength, young's modulus and strains at break were determined by using a Texture Analyzer TA-XT2 (Universal Testing Machine) with a load cell of 30 kg. Films were cut in form of strips with dimension of 10 mm × 70 mm. The strips were clamped between two tensile grips and the initial gauge length was set at 30 mm. Films were pulled using a crosshead speed of 2 mm/min. During the stretching, force (N) and deformation (mm) were recorded. Measurements were carried out on 10 different specimens. The mechanical properties were calculated as average value from the obtained results.

Results and Discussion

Tensile strength of SPS plasticized films

The effect of plasticizer type and concentration on the tensile strength of SPS films are shown in Fig. 1. The presence of plasticizer at lower concentration of 15 % demonstrated high tensile strength value of 28.35 MPa for S-plasticized films, 15.82 MPa for GS-plasticized films and 9.59 MPa for G-plasticized films. The possible reason for the high tensile strength at low plasticizer concentration is the domination of strong hydrogen bonds produced by starch – starch intermolecular interaction over starch – plasticizer attraction. However, the addition of plasticizers from 15 to 45 % caused significant reduction in the tensile strength of films, regardless of plasticizer type. The tensile strength of G-plasticized films notably decreased from 9.59 to 1.67 MPa and that of S-plasticized films dropped from 28.35 to 5.84 MPa as plasticizer concentration increased from 15 to 45 %. In the case of GS-plasticized films, tensile strength reduction was observed from 15.82 to 3.99 MPa at the same range of plasticizer concentration. The decrease in tensile strength of starch based films as plasticizer concentration increases were reported by numerous authors [17], [18]. This phenomenon can be explained through the role of plasticizers in diminishing the strong intra-molecular attraction between the starch chains and promoting the formation of hydrogen bonds between plasticizers and starch molecules. Thus, reduces the tensile strength of SPS plasticized films by subsequently weakening the hydrogen bonds between starch chains [10].

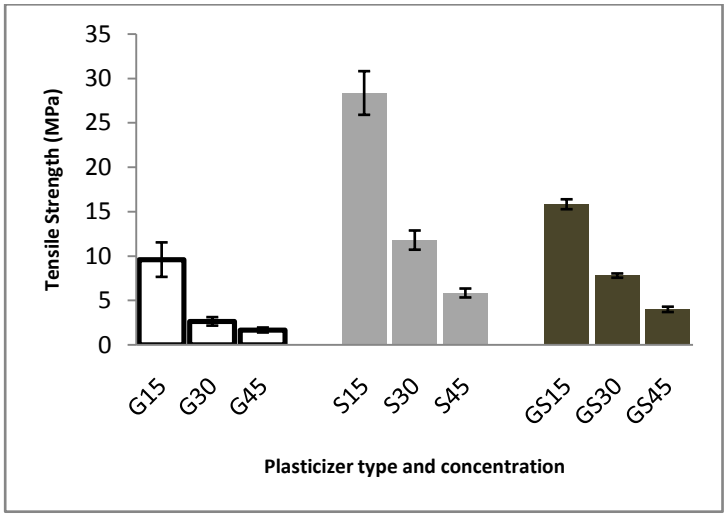

Fig. 1. Effect of plasticizer type and concentration on the tensile strength of SPS films

As the glycerol concention increased from 15 to 45 %, G-plasticized films showed the highest reduction in tensile strength as compared to S- and GS-plasticized films. These results revealed that glycerol has higher efficiency in plasticizing SPS films than sorbitol. Razavi et al. [19] and Musat et al. [10] reported similar observation that glycerol induces greater tensile strength reduction compared to other polyols. This tendency can be ascribed to the smaller molar mass of glycerol (92.0928 g/mol; sorbitol 182 g/mol) which facilitate easy interaction between glycerol – starch

molecular chains. Tapia-Blácido et al. [20] also testified that glycerol is a more effective plasticizer for most edible films.

Fig 1 explicitly manifested that S-plasticized films demonstrated superior tensile strength over their counterparts at constant plasticizer concentration. For example, the tensile strength values of S-plasticized films are 28.35, 11.79 and 5.84 MPa compared to 9.59, 2.64 and 1.67 MPa for G-plasticized films at 15, 30 and 45 % plasticizer concentration, respectively. These results are consistent with the findings of Tapia-Blácido et al. [20] and Tapia-Blácido et al. [11], who reported that sorbitol plasticized flour films are more resistant to break than films plasticized with glycerol at constant proportion. Interestingly, GS-plasticized films exhibited tensile strength between individual G- and S-plasticized films. For example, the tensile strength value of GS30 is 7.79 MPa, which is between the tensile strength of 2.64 MPa for G30 and 11.79 MPa for S30 films. This is because GS-plasticized films are combination of glycerol and sorbitol plasticizers. Therefore, the resulting films (GS-plasticized films) improve the low tensile strength of G-plasticized films and compromise the high tensile strength of S-plasticized films.

Elongation at break of SPS plasticized films

Elongation at break is the extendibility of film length from initial length to the point of break. Moraes et al. [21] defined elongation at break (E%) as the ability of films to deform before finally breaking. This parameter (E%) helps to determine the flexibility and stretchability of films. The desired flexibility of biopackaging films depends on their intended application and subsequent transportation, handling and storage of packaged foods.The effect of plasticizer concentration (15 – 30 %) on the elongation of SPS plasticized films has an inverse behaviour (see Fig 2) compared with their correspondent tensile strength. As anticipated, the increase of plasticizer concentration from 15 to 30 % registered considerable increase in film elongation: 26.52 – 61.63 % for G-plasticized films, 5.38 – 34.5 % for S-plasticized films and 15.1 – 46.65 % for GS-plasticized films. Similar film elongation behaviour has been reported by Kurt and Kahyaoglu [22], Suppakul et al. [23], Muscat et al. [10], Talja et al. [25] and Mali et al. [14]. The observed increase in film elongation is because plasticizers decrease the intermolecular bonds between amylose, amylopectin and amylose – amylopectin of the starch matrix and thus, substitute them with hydrogen bonds formed between plasticizer and starch molecules. Such disruption and reconstruction of starch molecular chains reduce the rigidity and promotes flexibility of films by allowing more chain mobility. Zavareze et al. [26] reported that the elongation of polymeric materials depends on the mobility of their moleculer chains.

The results presented in Fig 2 verified that film elongation increased as the concentration of plasticizers was adjusted. However, the elongation of G- and GS-plasticized films significantly decreased from 61.63 to 28.39% and 46 to 34.27 %, respectively, when plasticizer content increased from 30 to 45 %. Sahari et al. [27] also reported a dramatic drop in the elongation of plasticized sugar palm starch when 40 % glycerol was used. This occurence can be explained through the anti-plasticization behaviour of plasticized starch films. Suppakul et al. [23] observed

antiplasticization effect on the elongation at break of cassava flour films at 50 % sorbitol concentration. However, such antiplasticization behaviour was not observed even in S45 films.

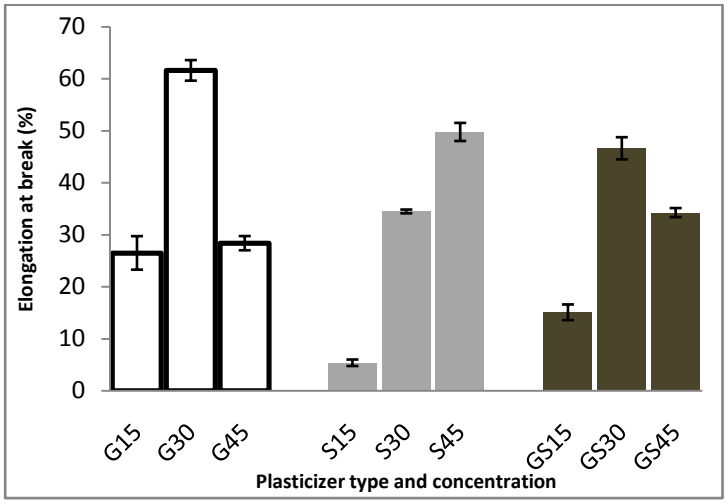

Fig. 2. Effect of plasticizer type and concentration on the tensile strength of SPS films

Conclusions

The different plasticizers utilized in the film-forming solution have remarkable effects on the mechanical and thermal properties of the resulting films. In all the plasticized SPS films, the tensile strength decreased with plasticizer content from 15 – 45 %. The reduction of hydrogen bonds between starch intermolecular chains due to the formation of starch (amylose) – plasticizer complexes may be responsible for the plasticizing effect of different plasticizer concentrations. An antiplasticization effect was obtained for the percent elongation (E %) of G- and GS-plasticized films at 45 % plasticizer concentration, while plasticization behaviour was found at lower concentrations. The current study verified that plasticizer type and concentration affects the mechanical and thermal properties of plasticized SPS films.

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References

[1] J. Sahari, S. M. Sapuan, E. S. Zainudin, and M. a. Maleque, “A New Approach to Use Arenga Pinnata as Sustainable Biopolymer: Effects of Plasticizers on Physical Properties,” *Procedia Chem.*, vol. 4, pp. 254–259, Jan. 2012.

[2] M. R. Ishak, S. M. Sapuan, Z. Leman, M. Z. a Rahman, U. M. K. Anwar, and J. P. Siregar, “Sugar palm (Arenga pinnata): Its fibres, polymers and composites.,” *Carbohydr. Polym.*, vol. 91, no. 2, pp. 699–710, Jan. 2013.

[3] W. Elberson and L. Oyen, “Sugar palm (Arenga pinnata),” 2010.
[4] D. R. Adawiyah, T. Sasaki, and K. Kohyama, “Characterization of arenga starch in comparison with sago starch,” *Carbohydr. Polym.*, vol. 92, no. 2, pp. 2306–2313, 2013.
[5] J. Sahari, S. M. Sapuan, Z. N. Ismarrubie, and M. Z. A. Rahman, “Comparative study of physical properties based on different parts of sugar palm fibre reinforced unsaturated polyester composites,” *Key Eng. Mater.*, vol. 471, pp. 455–460, 2011.
[6] M. Li, D. Li, L.-J. Wang, and B. Adhikari, “Creep behavior of starch-based nanocomposite films with cellulose nanofibrils.,” *Carbohydr. Polym.*, vol. 117, pp. 957–63, Mar. 2015.
[7] N. R. Savadekar and S. T. Mhaske, “Synthesis of nano cellulose fibers and effect on thermoplastics starch based films,” *Carbohydr. Polym.*, vol. 89, no. 1, pp. 146–151, 2012.
[8] K. M. Dang and R. Yoksan, “Development of thermoplastic starch blown film by incorporating plasticized chitosan.,” *Carbohydr. Polym.*, vol. 115, pp. 575–81, Jan. 2015.
[9] L. Dai, C. Qiu, L. Xiong, and Q. Sun, “Characterisation of corn starch-based films reinforced with taro starch nanoparticles.,” *Food Chem.*, vol. 174, pp. 82–8, May 2015.
[10] D. Muscat, B. Adhikari, R. Adhikari, and D. S. Chaudhary, “Comparative study of film forming behaviour of low and high amylose starches using glycerol and xylitol as plasticizers,” *J. Food Eng.*, vol. 109, no. 2, pp. 189–201, Mar. 2012.
[11] D. R. Tapia-Blácido, P. J. do Amaral Sobral, and F. C. Menegalli, “Optimization of amaranth flour films plasticized with glycerol and sorbitol by multi-response analysis,” *LWT - Food Sci. Technol.*, vol. 44, no. 8, pp. 1731–1738, Oct. 2011.
[12] T. J. Gutiérrez, N. J. Morales, E. Pérez, M. S. Tapia, and L. Famá, “Physico-chemical properties of edible films derived from native and phosphated cush-cush yam and cassava starches,” *Food Packag. Shelf Life*, vol. 3, pp. 1–8, Mar. 2015.
[13] P. V. A. Bergo, R. A. Carvalho, P. J. A. Sobral, R. M. C. Dos Santos, F. B. R. Da Silva, J. M. Prison, J. Solorza-Feria, and A. Habitante, “Physical properties of edible films based on cassava starch as affected by the plasticizer concentration,” *Packag. Technol. Sci.*, vol. 21, no. 2, pp. 85–89, 2008.
[14] S. Mali, L. S. Sakanaka, F. Yamashita, and M. V. E. Grossmann, “Water sorption and mechanical properties of cassava starch films and their relation to plasticizing effect,” *Carbohydr. Polym.*, vol. 60, no. 3, pp. 283–289, May 2005.
[15] M. Rodríguez, J. Oses, K. Ziani, and J. I. Mate, “Combined effect of plasticizers and surfactants on the physical properties of starch based edible films,” *Food Res. Int.*, vol. 39, no. 8, pp. 840–846, 2006.
[16] R. A. Talja, H. Helén, Y. H. Roos, and K. Jouppila, “Effect of type and content of binary polyol mixtures on physical and mechanical properties of starch-based edible films,” *Carbohydr. Polym.*, vol. 71, no. 2, pp. 269–276, 2008.

[17] A. B. Dias, C. M. O. Müller, F. D. S. Larotonda, and J. B. Laurindo, “Biodegradable films based on rice starch and rice flour,” *J. Cereal Sci.*, vol. 51, no. 2, pp. 213–219, Mar. 2010.

[18] M. Ghasemlou, F. Khodaiyan, and A. Oromiehie, “Physical, mechanical, barrier, and thermal properties of polyol-plasticized biodegradable edible film made from kefiran,” *Carbohydr. Polym.*, vol. 84, no. 1, pp. 477–483, Feb. 2011.

[19] S. M. A. Razavi, A. Mohammad Amini, and Y. Zahedi, “Characterisation of a new biodegradable edible film based on sage seed gum: Influence of plasticiser type and concentration,” *Food Hydrocoll.*, vol. 43, pp. 290–298, Jan. 2015.

[20] D. R. Tapia-Blácido, P. J. D. A. Sobral, and F. C. Menegalli, “Effect of drying conditions and plasticizer type on some physical and mechanical properties of amaranth flour films,” *LWT - Food Sci. Technol.*, vol. 50, no. 2, pp. 392–400, Mar. 2013.

[21] J. O. de Moraes, “Propriedades de filmes de amido incorporados de nanoargilas e fibras de celulose,” 2009.

[22] A. Kurt and T. Kahyaoglu, “Characterization of a new biodegradable edible film made from salep glucomannan.,” *Carbohydr. Polym.*, vol. 104, pp. 50–8, Apr. 2014.

[23] P. Suppakul, B. Chalernsook, B. Ratisuthawat, S. Prapasitthi, and N. Munchukangwan, “Empirical modeling of moisture sorption characteristics and mechanical and barrier properties of cassava flour film and their relation to plasticizing–antiplasticizing effects,” *LWT - Food Sci. Technol.*, vol. 50, no. 1, pp. 290–297, Jan. 2013.

[24] P. Suppakul, B. Chalernsook, B. Ratisuthawat, S. Prapasitthi, and N. Munchukangwan, “Empirical modeling of moisture sorption characteristics and mechanical and barrier properties of cassava flour film and their relation to plasticizing–antiplasticizing effects,” *LWT-Food Sci. Technol.*, vol. 50, no. 1, pp. 290–297, 2013.

[25] R. A. Talja, H. Helén, Y. H. Roos, and K. Jouppila, “Effect of various polyols and polyol contents on physical and mechanical properties of potato starch-based films.,” *Carbohydr. Polym.*, vol. 67, no. 3, pp. 288–295, 2007.

[26] E. da Rosa Zavareze, V. Z. Pinto, B. Klein, S. L. M. El Halal, M. C. Elias, C. Prentice-Hernández, and A. R. G. Dias, “Development of oxidised and heat–moisture treated potato starch film,” *Food Chem.*, vol. 132, no. 1, pp. 344–350, 2012.

[27] J. Sahari, S. M. Sapuan, E. S. Zainudin, and M. A. Maleque, “Thermo-mechanical behaviors of thermoplastic starch derived from sugar palm tree (Arenga pinnata),” *Carbohydr. Polym.*, vol. 92, no. 2, pp. 1711–1716, 2013.

The Effects of Fiber Thermal Properties on Millet Husk Filled High Density Polyethylene Composites

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Abstract. This paper present a study on the ability of agro waste (millet husk fibers) from cereal millet (pennisetum glaucum) as filler for thermoplastic composites as an alternative for inorganic filler and other fibers. The fiber thermal properties were investigated by means of thermogravimetric analyzer (TGA) and thermal decomposition of the fiber was found to be stable at 245 °C. The moisture content was determined using oven-dry value analysis, thus indicated stability for the millet husk (MH) fiber- high density polyethylene (HDPE) matrix interaction in MH-HDPE composites fabrication. Hence, these properties suggest that the fiber could be one of the future raw materials for plastic composites filler.

Keywords: millet; husk; cereal; composite

Introduction

Millet husk is an agro waste product obtains from the milling process of grain cereal millet. Agro waste and natural fiber composites are not altogether a new phenomenon. In last few decades, there are concerns towards "environmental friendly composites materials". Several studies are going on in an effort to adapt more applications for materials from unwanted resources [1]. In an attempt to gain robust active thermoplastic composites that would become useful in varieties of areas with functional products such as millet husk, researches are currently intensified. Thus, these lignocellulosic cereal waste are environmental attuned, renewable, biodegradable, weight reduction and have a tendency to provide the sources for agro waste composites [2, 3]. Agro waste are annually renewable fibers and are obtainable in large quantities throughout the globe [4]. The local use of millet husk includes; packaging for livestock feed, bedding as pillow, and rest mattress. The major producers of millet are from Asia and Africa with estimated 180 million metric tons in 2008 and greater percentage from India and Nigeria. [4] A variety of cellulose containing reinforcements for biodegradable composites has been proposed in literature, but little or no attention has been paid to millet husk fibers. The scientific study on millet husk here was investigated, the chemical compositions, fiber size chemistry and thermal degradation characteristics were determined with the aim to establish the viability in processing and production of composites. Additionally to

proffer it as possible reinforcement/filler for thermoplastic composites as alternative to inorganic fibers that has direct influence on environment and health [5].

Materials and Methods

Materials

The millet husks fibers were collected from farm site, dumped after milling at Bulumkutu Kasuwa Area of Maiduguri Borno, Nigeria. Two different varieties of fibers were collected. For purpose of this study, 4 kg of materials were collected.

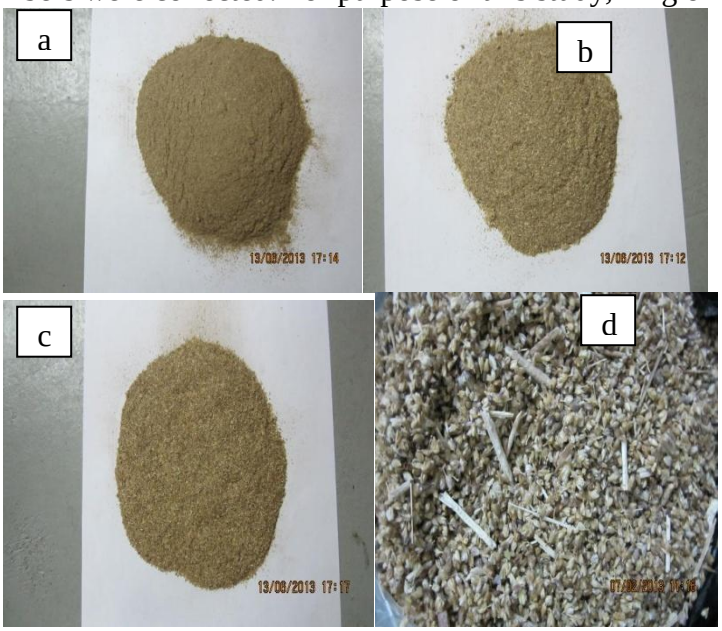

Figure 1.Millet husks with different pulverized size. a) 250 µm, b) 500 µm, c) 750 µm and d)unpulverized

Fiber Thermogravimetric Analysis:

The thermogravimetric analysis (TGA) method employed are; dynamic heating from temperature range of 30 °C-900 °C with heating rate of 10 °C/min under 10 mL/min nitrogen flow with sample subjected to the instrument range of 5 to 10 mg. the instrument model is TGA/SDTA 851e Mettler Toledo (Switzerland).

Results and Discussion

Fiber Thermogravimetric Analysis

The thermogravimetric analysis (TGA) indicates that the specimen weight is slowly lost up over time to a temperature of about 245 °C. The gradual weight loss at a point is presumably due to the loss of moisture soak up from the environment and crumble of low molecular weight make ups. The TGA thermogram also shows that between 245–370 °C, an unexpected weight loss occurred. It is believed that this likely corresponds to the decomposition of cellulose and hemicelluloses with emerges of carbon residues. Thus, similar trend was observed by [6]. From 370 °C, sample loss gradually occurs again showing certain trend to stabilize. Above temperature of 370 °C, breakdown of sample's lignin and carbon residues occurs. Hence, these outcomes indicate that the maximum allowed temperature for processing the millet husk fibers

in order to be compatible in thermoplastic composites should exceed 245 ˚C. Consequently, similar methods of investigation were used with variety results observed by several authors;

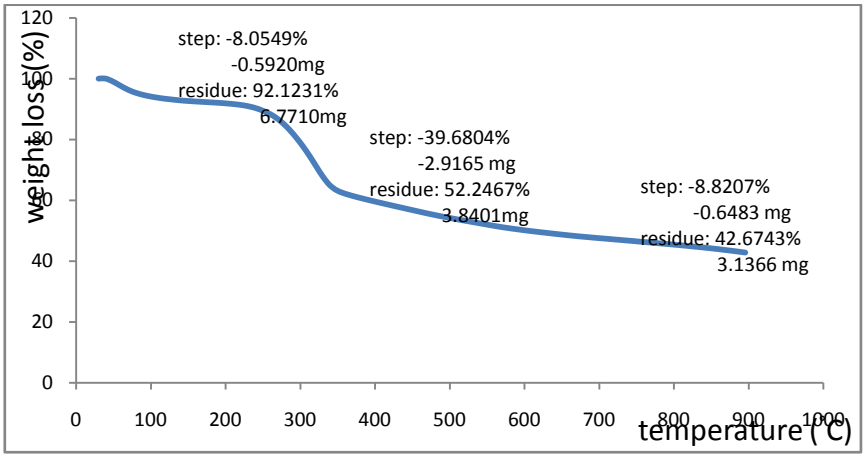

Figure 2 TGA Thermogram of millet husk fiber

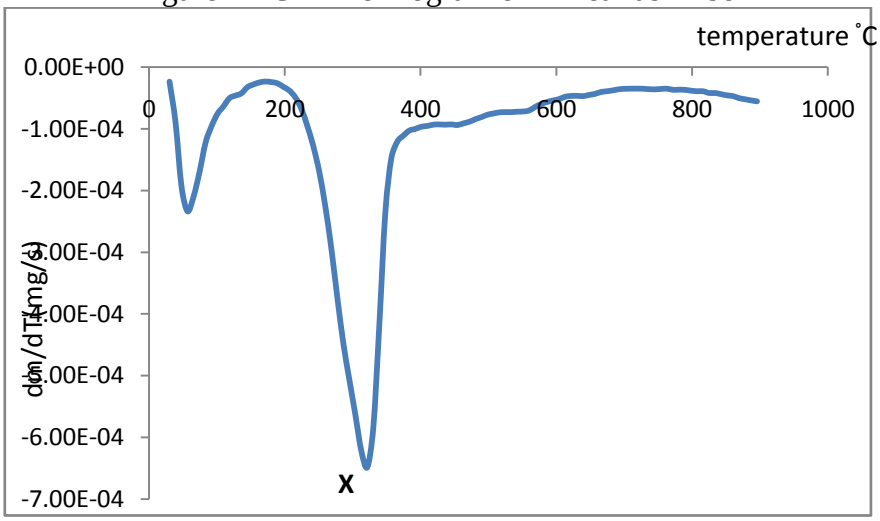

Figure 3. DTG Derivative thermogram of millet husk fiber

The millet husk cellulose is fairly higher than that of lignin, a similar weight loss rate was observed. This can be seen from thermogram in Figure 2, the difference between hemicelluloses and cellulose decompositions was partially not realized but it was understood that hemicelluloses decomposed at lower temperature when compared to cellulose [6] . Hence, it can be pointed out in the derivative thermogravimetric (DTG) curve the position X shown in Figure 3 it could have been initial cellulose broken down point, but the temperature was higher compared to the value of other agro waste fibers [7]. More so, the features of thermal analysis of millet husk fiber shown in Figure 2 presumed that the entire moisture content of the fibers were eliminated prior 100 ˚C. The weight at 100 ˚C temperature is considered as initial weight and the starting broken down temperature of millet husk fibers were defined at 1 % weight

loss. The effect of the thermal characteristics of the millet husk fiber is that when the temperature of the process becomes higher than 245 °C, there is tendency of volatile compound production, which is captured into accumulation of aggregate mixture. Thus, causing voids within the end product and could invariably affect mechanical properties of thermoplastic composites.

Conclusion

This study examines the viability of using millet husk fibers from cereal by product as alternative fillers for natural and inorganic fibers for thermoplastic composites formations. From this study, it shows that millet husk is thermally stable at temperature of about 245 °C, which represent the useful temperature for composites production compare other fibers such as wood, barley, rice husk.

References:

[1] Sapuan, S.M., et al., *Mechanical properties of soil buried kenaf fibre reinforced thermoplastic polyurethane composites.* Materials & Design, 2013. 50(0): p. 467-470.

[2] Suddel, B.C.and W. Evans, *natural fiber, biopolymers and biocomposites. In: Mohanty AK, Misra M, Drzal LT,editors. New York: Taylor and Francis; .* polymer and environment, 2005: p. 231-259.

[3] Mohanty, A.K., M. Misra, and D. LT., *sustainable biocmposites from renewable resources: opportunity and challenges in the green materials world.* journal of polymer and environment, 2002. 10: p. 19-26.

[4] Andrzej, K., et al., *Barley husk and coconut shell reinforced polypropylene composites: The effect of fibre physical, chemical and surface properties.* Composites Science and Technology 2010. 70: p. 840–846.

[5] Sdrobiş, A.D., et al., *Low density polyethylene composites containing cellulose pulp fibers.* Composites Part B: Engineering, 2012. 43(4): p. 1873-1880.

[6] Heuze, V.and G. Tran. *Pearl Millet (Pennisetum Glaucum) grain*. PrograImme by INRA, CIRAD, ATZ and FAO 2012 [cited August 27, 2012.

Oil Palm Mesocarp Fibre: Non –linear Mechanical Behaviour

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Abstract. This work investigates the non-linear mechanical behaviour of oil palm mesocarp fibres using tensile tests, microstructure observation and finite element models. The scanning electron microscopy images showed the surface of the fibres with partly embedded silica bodies, while the cross section contained cell walls structure. Viscoelastic behaviour was observed when the fibres were relaxed over time after being stretched.

Keywords: Finite element method; Oil palm mesocarp fibres; Viscoelastic

Introduction

Malaysia is considered as one of the world's largest oil palm producer with production of ~19.2 million tonnes in 2013 (~47% of world palm oil supply) [1]. This high volume of production suggested that Oil Palm Mesocarp Fibres (OPMF) can be used for biocomposite [2] or biocompost [3,4] application.Therefore a detail mechanical and microstructural study of OPMF is needed. However, only a few mechanical studies on OPMF (and other oil palm fibres) are available. For example, previous work by Yusoff et al. [5] and Gunawan et al. [6] assumed that oil palm fibres are a linear elastic and homogenous material.It is deemed that the behaviour of oil palm fibres, in particular OPMF is more complex than originally assumed by these researchers.

Materials and methods

Sample preparation

OPMF were obtained from Besout Palm Oil Mill (Sungkai, Perak, Malaysia). The samples were then kept in a controlled environmental condition of $-20\ ^{0}C$ to avoid the growth of fungus. The samples being washed and dried at $105\ ^{0}C$ for 24 hours (with the moisture content kept below 5%).

Microstructure analysis

The morphology of OPMF was analysed using a scanning electron microscope (SEM) (model E-1010, Hitachi, Japan). The samples were mounted on an aluminium stub and were sputter coated with platinum. An acceleration voltage range of 15-25 kV was used to obtain the SEM micrographs.

Mechanical tests

Mechanical tests were conducted using a Texture Analyzer (model TA-XT, Stable Micro Systems Ltd) at a controlled ambient temperature. The apparent diameters were kept between the ranges of 0.1 mm to 0.25 mm samples height were retained constant at 20 mm. The range of diameters of OPMF in this case (0.1 to 0.25 mm) are comparable to oil palm empty fruit bunch fibres with 0.4 to 0.72 mm by Gunawan et al. (2009)).Tensile tests were performed following the method by Yusoff et al. [5] at a constant crosshead speeds of 10 mm/s, 1 mm/s and 0.1 mm/s.

Viscoelastic material model theory

The viscoelastic model used in this work assumes a separable time and strain dependent material behaviour, as well as a homogeneous and isotropic material [8]. For this, the relaxation stress under a step strain loading history is defined as a function of time, $g(t)$, and strain, $\sigma_0(\varepsilon)$ through $\sigma(\varepsilon,t)=\sigma_0(\varepsilon)g(t)$. The time function is represented by the Prony series:

$$g(t)=g_\infty+\sum_{i=1}^{N}g_i\exp\left(-\frac{t}{\xi_i}\right) \tag{1}$$

where t and ξ_i are time and relaxation time constants respectively, and g_i and g_∞ are dimensionless constants. The Prony series consist of a series of Maxwell elements (springs and dampers) connected in parallel with a spring.The total stress can be obtained using the Leaderman form of the convolution integral [11] which is given by the algebraic sum of the entire past loading history at time t:

$$\sigma(\varepsilon,t)=\int_0^t g(t-s)\frac{d\sigma_0(\varepsilon)}{ds}ds \tag{2}$$

Combining the previous two equations using the numerical algorithm of finite time increments [10] which the detail derivation is provided elsewhere [8,9] yields:

$$\sigma(t_{n+1})=g_\infty\sigma_0(t_{n+1}) +\lambda(t_{n+1})\sum_{i=1}^{N}\left(\exp\left(-\frac{\Delta t}{\xi_i}\right)h_i(t_n)+g_i\frac{1-\exp\left(-\frac{\Delta t}{\xi_i}\right)}{\frac{\Delta t}{\xi_i}}\left[P_0(t_{n+1})-P_0(t_n)\right]\right). \tag{3}$$

where P_0 represents the nominal stress term, which is related to the true stress, σ_0 through: $\sigma_0(t_n)=P_0(t_n)\cdot\lambda(t_n)$. The term, $h_i(t)=\int_0^t g_i\exp\left(-\frac{t-s}{\xi_i}\right)\frac{d\sigma_0(s)}{ds}ds$. The stress, σ_0 in Eq. (3) is obtained using a linear elastic equation as: $\sigma_0(t_{n+1})=E_f\varepsilon(t_{n+1})$, where E_f is the fibre elastic modulus. Note that an analytical evaluation of Eq. (3) presented by Mohammed [9] yielded similar results to those of numerical results in Abaqus (single-element model).

Results and discussion

Microstructure of oil palm mesocarp fibres

Figure 1(a) shows the overall structure of oil palm fruitlets, while figure (b) shows single fibre with occurrence of silica bodies embedded on the outer surface of the fibre. Figures (c) and (d) show zoom in images of a single fibre with silica bodies.On the other hand, cell walls structure of oil palm fibres (Figure (e)) has been reported by Shamsudin et al. [7], which also showed that no trace of silica bodies within the cross-section.

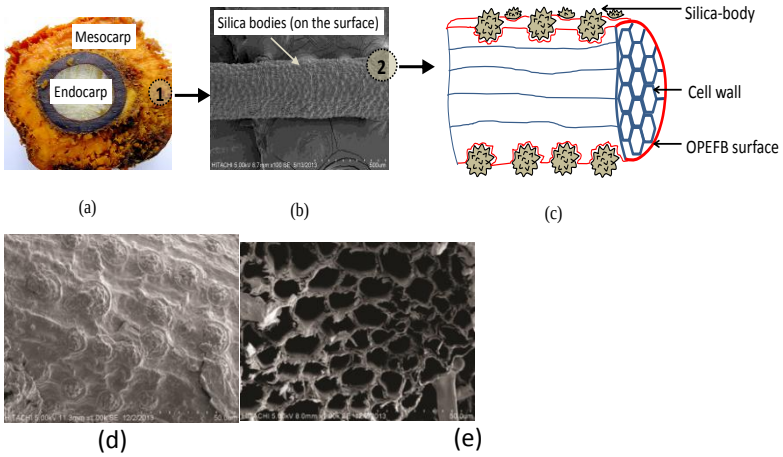
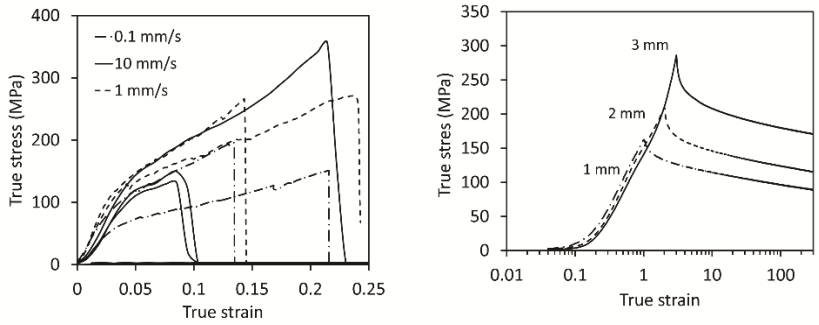


Fig. 1. (a) oil palm fuitlets, (b) single OPMF strands, (c, d)silica bodies embedded om the surface of OPMF and (e) OPMF cell wall structure.

Mechanical tests

Tensile tests results for a few fibres are shown in Fig. 2a, where the time dependent behaviour is not obvious. This can be due to difficulties in obtaining fibres from a controlled batch (i.e. similar ripeness, sizes, and tree variations). However, the stress-strain curves in Fig. 2a show similar pattern, which can be separated into elastic, plastic and fracture regions. In the first region (strain less than 0.04), damage within the microstructure of OPMF is thought to be minimal (linear elastic behaviour). For strains beyond 0.04, plastic region is observed, which is related to microstructure damages in OPMF, namely silica-body and fibre interface, cell walls and cellulose-hemicellulose-lignin interface damages, as suggested by Omar et al. [12]. The latter two damages are well established for lignocellulosic materials like wood [13].Finally in the fracture region, fracture or total failure of OPMF is observed from the sudden drop of stress in Fig. 2a.



(a) (b)

Fig. 2. (a) Tensile tests at different speeds, and (b) stress relaxation

Viscoelastic behaviour is observed from relaxation tests results at all deformation values (1, 2 and 3 mm), as shown by the steady reduction of stress at the constant strain over time in Fig. 2b. However, the rate dependent behaviour (stiffer stress-strain curves at higher deformation/strain rates) is not pronounced for tests shown in Fig. 2a, as would normally be observed for viscoelastic materials tested at different speed/rate, which can be due to the different damages mechanism discussed before.

Viscoelastic material model fitting to relaxation test result

The viscoelastic model (Eq. 3) was fitted to the stress relaxation test data, as shown in Fig. 3, where the model parameters used are shown in Table 1. The fibre elastic modulus shown in Table 1 was within the values of elastic modulus from the tests result obtained from mechanical test, whereas the Prony series was fitted to the relaxation test data (1 mm deformation) using a least squares method (Goh et al. 2004), where at this small deformation, OPMF is assumed to have minimal microstructural damages.

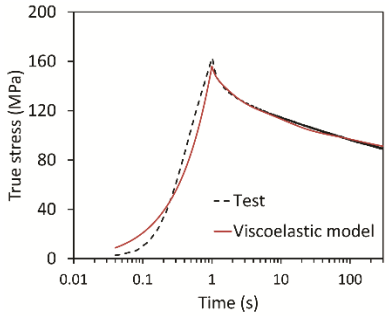


Fig. 3. Fitting of the viscoelastic model to stress relaxation test result at 1 mm deformation using parameters in Table 1

Note that the current model assumes an incompressible material, which means that no cell wall mechanism (cellular structure) is taken into account here. In addition, no plasticity model was introduced in this work in combination with the viscoelastic model, since this would require an additional user-material function (i.e. subroutine in Abaqus). Therefore, both mechanisms (cell wall and plasticity) are proposed in the future to improve the constitutive modelling of OPMF.

Table 1. The viscoelastic model parameters to model OPMF fibres

Silica bodies elastic modulus	Fibre elastic modulus	Prony series constants						
E_{sb} (GPa)	E_f (GPa)	i	1	2	3	4	5	∞
		ξ_i (s)	0.1	1	10	100	1000	
70	4.8	g_i	0.3	0.15	0.1	0.05	0.005	0.35

Conclusions

Investigation on the non-linear mechanical behaviour of oil palm mesocarp fibres wasperformed in this work through tensile tests, microstructure observation and finite elementmodel. Tensile tests conducted using single fibre showed possible viscoelastic behaviourfrom relaxation tests, even though this is not obvious from uniaxial tests at different speed.To improve, in situ tensile testing is proposed in the future to investigate possible microstructural changes that occur during fibre deformation, namely silica bodies and fibre de-bonding and micro fibril angle changes.

References:

[1] Sabil KM, Aziz MA, Lal B and Uemura Y (2013) Effect of torrefaction on the physiochemical properties of oil palm empty fruit bunches, mesocarp fiber and kernel shell, Biomass Bioenergy. 56: 351-360.

[2] Shinoj S, Visvanathan R, Panigrahi S and Kochubabu M (2011) Oil palm fiber (OPF) and its composites: A review, Ind. Crop Prod 33: 7 – 22.

[3] Yunos NSH, Baharuddin AS, MdYunos KF, Naim MN and Nishida H (2012) Physicochemical property changes of oil palm mesocarp fibers treated with high pressure steam, Bioresources 7(4): 5983-5994.

[4] Nordin NIA, Ariffin H, Andou Y, Hassan MA, Shirai Y, Nishida H, Yunus WMZW, Karuppuchamy S and Ibrahim NA (2013) Modification of oil palm mesocarp fibre characteristics using superheated steam treatment, Molecules 18: 9132-9146.

[5] Yusoff MZM, Salit MS and Ismail N (2009) Tensile properties of single oil palm empty fruit bunch (OPEFB) fibre, Sains Malaysia 38(4): 525-529.

[6] Gunawan FE, Homma H, Brodjonegoro SS, Hudin AB and Zainuddin A (2009)Mechanical properties of oil palm empty fruit bunch fibre, Solid Mech Mate Eng 3: 943-951

[7] Shamsudin S, Shah UKM ,Zainudin H, Aziz SA, Kamal SMK, Shirai Y and Hassan MA (2012) Effect of steam pretreatment on oil palm empty fruit bunch for the production of sugars, Biomass Bioenerg. 36: 280-288.

[8] Goh SM, Charalambides MN and Williams JG (2004) Determination of the constitutive constant of non-linear viscoelastic materials, Mech Time-Depend Mater 8: 255-268.

[9] Mohammed, M.A.P., 2012. Mechanical Characterisation, Processing and Microstructure of Wheat Flour Dough, Phd Thesis, Imperial College London.
[10] Kaliske, M., Rothert, M., 1997. Formulation and implementation of three dimensional viscoelasticity at small and finite strains. Comput. Mech. 19, 228-239.
[11] Williams, J.G., 1980. Stress Analysis of Polymer, John Wiley, London, UK
[12] Omar, F.N., Mohammed, M.A.P., Bahruddin, A.S., 2014. Microstructure modelling of silica bodies from oil palm empty fruit bunch (OPEFB) fibres. Bioresources 9(1), 938-951.
[13] Burgert, I., 2006. Exploring the micromechanical design of plant cell walls, Am. J. Bot. 93(10), 1391-1401.