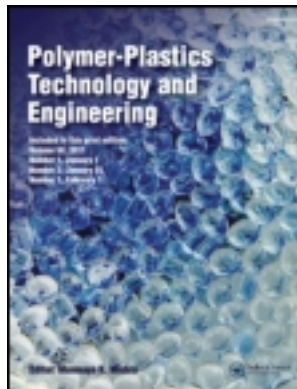


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Characterization and Properties of Recycled Polypropylene/Coconut Shell Powder Composites: Effect of Sodium Dodecyl Sulfate Modification

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This research work focuses on the utilization of coconut shell powder (CSP) as filler in recycled polypropylene (rPP). Sodium Dodecyl Sulfate (SDS) was used as coupling agent in these composites. The effect of filler content and SDS on tensile properties, thermal properties, water absorption and morphology of rPP/CSP composites were investigated. In this study, modified rPP/CSP composites with SDS show significant increased tensile properties, thermal stability, crystallinity and low water absorption compared unmodified rPP/CSP composites. Those improvements were contributed by the coupling effect of SDS.

Keywords Coconut shell powder; Composites; Recycled polypropylene; Sodium dodecyl sulfate

INTRODUCTION

Coconut (*Cocosnucrifera*) is the fourth important crop in Malaysia in term of acreage, after oil palm, rubber and paddy^[1–3]. The coconut shell is agricultural co-product abundance and available in low cost. Generally, coconut shell is used to producing active carbon, mosquito coil and charcoal. Coconut shell is easily process into powder form and called as coconut shell powder (CSP). The CSP have potential as filler for polymeric material and it showed better properties compared to mineral filler (e.g, calcium carbonate, kaolin, mica, and talc), such as low cost, renewable, minimal health hazard, low density, less abrasion to machine, biodegradable, and eco-friendly^[4–7].

Polypropylene (PP) is common thermoplastic, which are widely used in automobile, household appliance and construction industry. The disposals of PP become a waste materials and causing environmental issue due to its chemically stabilized state for long service life. The rate of recycling PP has increased significantly for economic and environmental reasons in current's society. The properties of recycle polypropylene (rPP) is lower compared to virgin PP, but rPP is used to mixed with other virgin thermoplastic

materials to achieve low cost end product. Nevertheless, the rPP can compounded with natural filler to produce composites for intended technical application without losing the ecological and economic advantage of the recycled materials.

Nowadays, composites materials made from biomass and thermoplastic material are gain interest among researchers and industry due to the environmental issue and economic factors. Currently, there are many eco-friendly product made from natural filler based composites materials has been successfully marketed. In Malaysia, Melsom Biodegradable Enterprise already successfully produced a series of eco-friendly tableware from rice husks filled thermoplastic composites. In general, incorporation of unmodified natural filler would show poor properties of its composites and incompatibility due to the polarity different between hydrophilic natural filler and hydrophobic polymer matrix^[1,7,8]. Alternatively, the properties of composites can improved via filler modification such as alkaline treatment^[9–11], esterification^[1,3,12,13], silane treatment^[2,16], use of compatibilizers^[16–21] and other chemical^[22].

Filler modification is important in natural filler based composites as well as to improve the wettability, dispersion and filler-matrix interaction. There are many studies on various kind of natural filler added in rPP to produce composites, such as oil palm co-product^[8], wood saw dust^[23], and rice husk^[24]. In this research, the sodium dodecyl sulfate (SDS) was used coupling agent to modify the CSP as to improve the properties rPP/CSP composites. The effect of sodium dodecyl sulfate (SDS) on mechanical properties, thermal properties, water absorption and morphology of rPP/CSP composites has been investigated.

METHODOLOGY

Materials

The recycled polypropylene (rPP) used was supplied by Toray Plastics (Malaysia) Sdn. Bhd. The coconut shell was obtained from market, Perlis. Firstly, the coconut shell was cleaned and crushed into small pieces. The small pieces of coconut shell were grinded into coconut shell powder

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(CSP) and dried in oven at 80°C for 24 h. The average particle size of the CSP was 215 µm, by using Malvern Particle Size Analyzer Instrument. The sodium dodecyl sulfate (SDS) and ethanol were obtained from Sigma Aldrich (Penang, Malaysia).

Chemical Modification

The SDS powder (3% by weight of CSP) was dissolved in ethanol. The CSP was added into SDS solution and continually stirred with mechanical stirrer for 2 hand left for overnight. The modified CSP was filtered and dried in oven at 80°C for 24 h to remove the ethanol.

Compound and Moulding procedure

The PP/CSP composites were compounded by using Z-Blade mixer (MCN ELEC Co. Taiwan) at temperature 180°C with rotor speed 50 rpm. First, rPP was transferred into mixing chamber for 8 min until it homogeneously melts. Afterward, CSP was added to melted rPP and continually for 7 min. Total compounding time used was 15 min. The rPP/CSP composites were moulded into tensile bar with 1 mm thickness by using compression moulding model GT 7014A at 180°C. The moulding cycle was involved 4 min pre-heat, compression under pressure at 100 kgf/cm² for 1 min. Next, the samples were cooling under same pressure for 5 min. Table 1 shows formulation of rPP/CSP composites.

Tensile Testing

Tensile testing was carried out by using Instron machine model 5566 according to ASTM D638. The test was performed at 23 ± 2°C with cross-head speed of 30 mm/min.

Water Absorption

The water absorption test on rPP/CSP composites was carried according to ASTM D570. The samples of rPP/CSP composites were immersed in distilled water at room temperature. The water absorption was determined by weighting the sample at regular intervals. A Mettler Balance Model AJ150 with precision of ±1 mg was used. The percentage of water absorption, M_t was calculated by formulation below:

$$M_t = \frac{W_N - W_d}{W_d} \times \%$$

Where W_d and W_n are original dried weight and weight after exposure, respectively.

Differential Scanning Calorimetry (DSC) Analysis

The DSC analysis was carried out by using DSC Q10, Research Instrument. The sample was cut into small piece and placed in close aluminum pan with sample weight in range 7 ± 2 mg. The specimen was heated from 30°C to 200°C with a heating rate of 10°C/min under nitrogen

atmosphere. The nitrogen gas flow rate was 50 ml/min. The degree of crystallinity of composites (X_c) can be evaluated from DSC data by using the following equation:

$$X_c = (\Delta H_f / \Delta H_f^0) \times 100$$

where ΔH_f was the heat fusion of the rPP composites, and ΔH_f^0 was the heat fusion for 100% crystalline PP ($\Delta H_{100} = 206 \text{ J/g}$).

Thermogravimetric Analysis (TGA)

The TGA analysis was carried out by using TGA Pyris Diamond Perkin-Elmer apparatus. Sample (7 ± 2 mg) was undergoes thermal scan from 30°C to 700°C in open platinum pan at heating rate of 10°C/min under nitrogen atmosphere. The nitrogen flow rate was 50 ml/min.

Morphology Analysis

Tensile fracture surfaces of rPP/CSP composites were examined with scanning electron microscope (SEM), model JEOL JSM-6460 LA. The fracture surfaces of the samples were sputter coated with thin layer of gold before analyzed.

Fourier Transmission Infrared (FTIR) Spectroscopy

A Perkin-Elmer Spectrum FTIR, Model Paragon 1000 was used to characterize chemical group in treated and untreated CSP. The Attenuated Total Reflectance (ATR) method was used. The sample was recorded with 4 scans in the frequency range 4000–650 cm⁻¹ with a resolution of 4 cm⁻¹.

RESULTS AND DISCUSSION

Tensile Properties

The effect of CSP content and SDS on tensile strength of rPP/CSP composites is showed in Figure 1. The addition

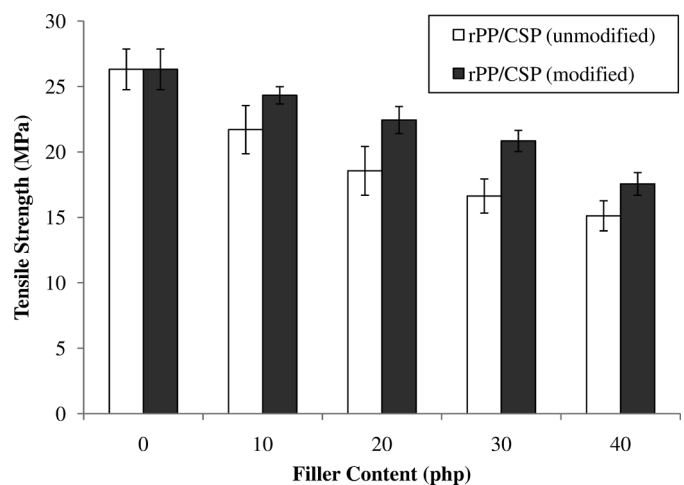


FIG. 1. Effect of filler content and SDS on tensile strength of unmodified and modified rPP/CSP composites.

of CSP reduced the tensile strength of both composites. The hydrophilic CSP and hydrophobic rPP are incompatible which cause the weak interfacial interaction. The weak interfacial interaction due to poor stress transfer between filler-matrix and it might contributed to poor tensile properties. As the increasing of CSP content, the area of weak interfacial also increased. Thus, the increasing of CSP content decreased the tensile strength of rPP/CSP composites. This result trend is similar to previous study on polylactic acid/coconut shell powder biocomposites^[1-3]. However, the modified rPP/CSP composites with SDS showed higher tensile strength compared to unmodified rPP/CSP composites. The increment was due to the coupling agent effect from SDS as present of SDS improved the compatibility between filler and matrix. The improvement is further enhanced the filler-matrix interaction which increased the tensile strength of rPP/CSP composites.

Figure 2 shows the elongation at break of unmodified and modified rPP/CSP composites with SDS. It can be seen, the elongation at break dramatically decreased as incorporated with CSP. The presence of CSP inherent the polymer chain mobility causes the composites failure after little of elongation. This was a common trend that also found by previous study and other researchers^[1-3,8,13,14]. Nevertheless, SDS modification was reduced the elongation at break of rPP/CSP composites. The presence of SDS providing CSP a hydrophobic surface as the long alkyl chains were attached on the CSP surface via covalent bonding, leading to increase the compatibility with and enhanced the interfacial bonding between CSP and rPP matrix. As results, the ductility of rPP/CSP composites was decreased by the enhanced interfacial bonding. Amri et al.^[22] also reported, the similar effect of SDS modification on elongation at break of chitosan filled polypropylene composites.

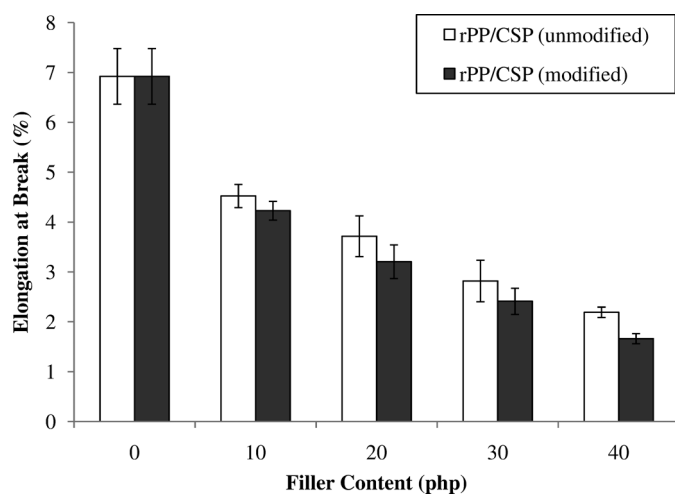


FIG. 2. Effect of filler content and SDS on elongation at break of unmodified and modified rPP/CSP composites.

On the other hands, the increase of CSP content increased the tensile modulus of unmodified and modified rPP/CSP composites as showed in Figure 3. The friction between CSP particles and rPP matrix form a rigid interface which hinder the movement of the rPP chains. This was assigned to more stiff and rigid composites. The CSP is giving stronger stiffening effect on rPP/CSP composites as the CSP content increased. Some researchers also reported that the CSP increased the tensile modulus of polyvinyl alcohol/CSP composites^[25,26]. Moreover, the higher stiffness of composites can be observed on modified rPP/CSP composites. This might be due to the better filler-matrix interaction provided by the SDS modification.

Water Absorption

Figure 4 illustrates the water absorption of unmodified and modified rPP/CSP composites. It can be found that the water absorption of both rPP/CSP composites increased with CSP content and immersion time. At similar CSP content, unmodified rPP/CSP composites exhibits higher water absorption compared modified rPP/CSP composites. The structure of CSP consists of hydroxyl groups that give the hydrophilic properties and tendency to absorb water. Therefore, the water uptake ability of composites is increased with increasing of filler content. Salmah et al.^[17] and Rozman et al.^[27] also found that the unmodified natural filler (rubber wood and rice husk) have more hydroxyl group to bond with water results the higher water absorption to its composites. The SDS modification reduced the hydrophilic behavior of CSP by reacting with the hydroxyl groups of CSP. The hydrophilic properties of CSP were significantly reduced by SDS modification and it was proven by FTIR analysis.

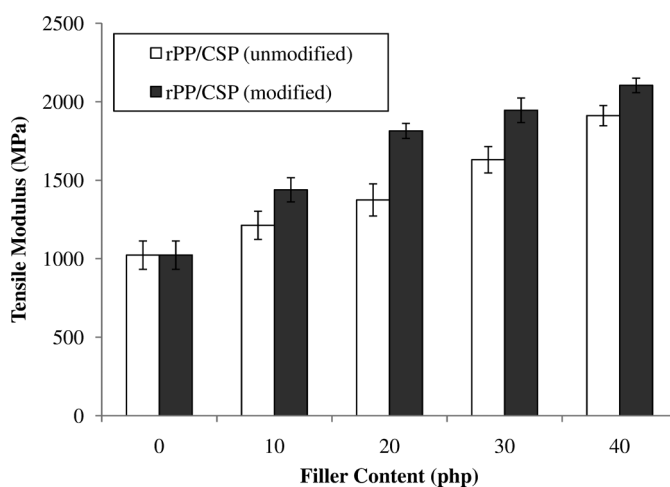


FIG. 3. Effect of filler content and SDS on tensile modulus of unmodified and modified rPP/CSP composites.

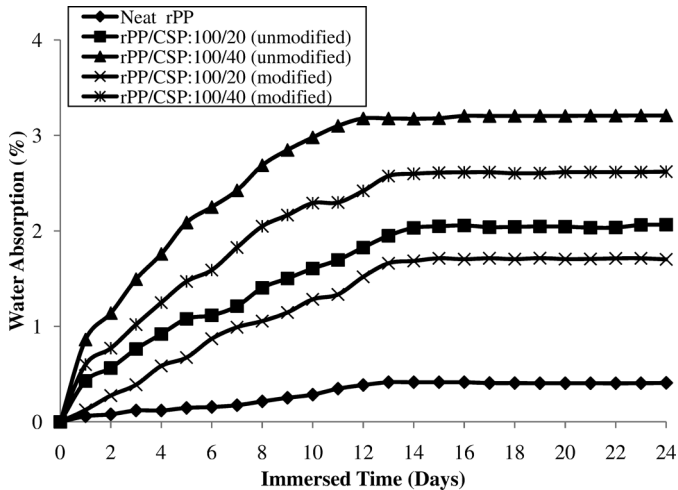


FIG. 4. Water absorption of unmodified and modified rPP/CSP composites.

Morphology Study

Figures 5 and 6 show SEM micrographs of tensile fracture surface of unmodified rPP/CSP composites at 20 php and 40 php of CSP content. From the SEM micrographs show there are discontinue phase exits between CSP and rPP matrix. This is mainly cause by the poor wetting of hydrophilic filler by hydrophobic polymeric matrix. Besides, there are numerous of holes cause by filler pull out. All of this indicated the incompatibility and poor filler-matrix interfacial adhesion. The SEM micrographs of tensile fracture surface of modified rPP/CSP are show in Figures 7 and 8. The modified CSP with SDS show better compatibility with rPP matrix as the modified CSP particles are found embed inside the rPP matrix. There absent of discontinue phase better filler-matrix and holes

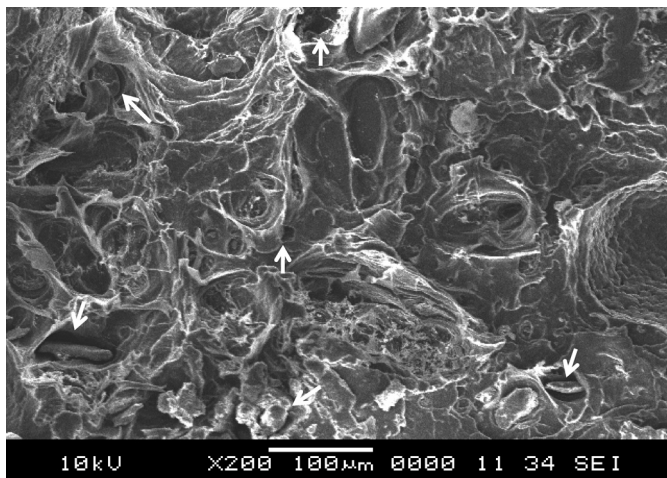


FIG. 5. Scanning electron micrograph of tensile fracture surface of unmodified rPP/CSP composites (20 php filler content).

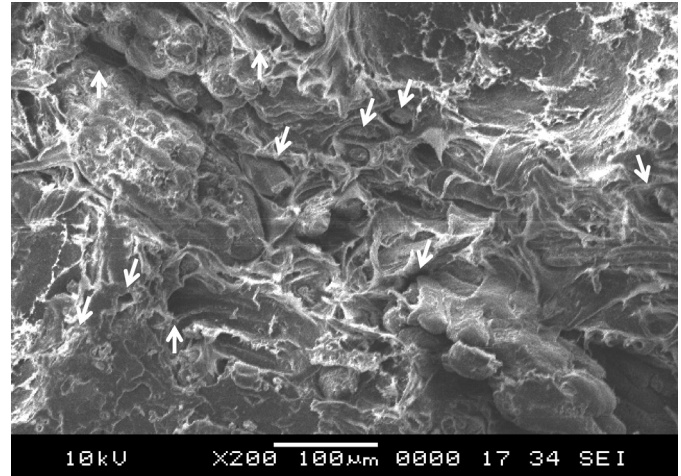


FIG. 6. Scanning electron micrograph of tensile fracture surface of unmodified rPP/CSP composites (40 php filler content).

cause by filler pull out evidence the better filler-matrix adhesion are achieved by SDS modification.

FTIR Analysis

The FTIR spectra of unmodified and modified CSP were illustrated in Figure 9. The main characteristic peaks of CSP are summarized in Table 2. The broad peak at $3000\text{--}3800\text{ cm}^{-1}$ was assigned to hydroxyl (-OH) groups of CSP. The hydrophilicity of CSP was reflected in this peak as the presence of -OH groups on the surface of CSP. The intensity of -OH groups absorption band was significantly reduced in modified CSP. The indicated that the hydrophilic properties of CSP was reduced as the -OH groups are reacted with SDS. Furthermore, the peak at 1727 cm^{-1} was assigned as carboxyl (C=O) groups from

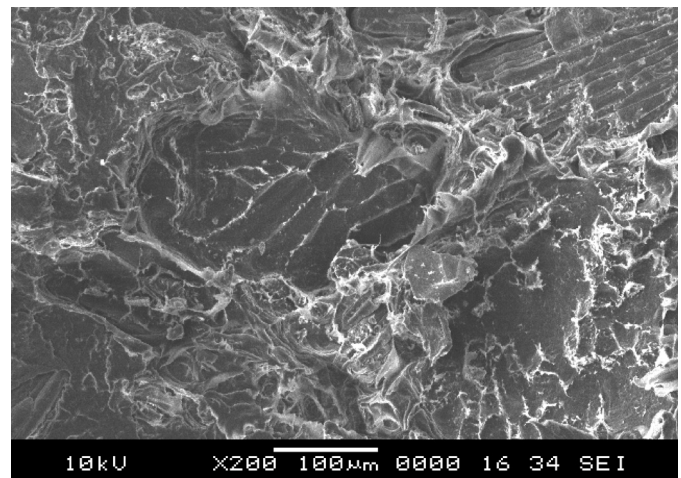


FIG. 7. Scanning electron micrograph of tensile fracture surface of modified rPP/CSP composites (20 php filler content).

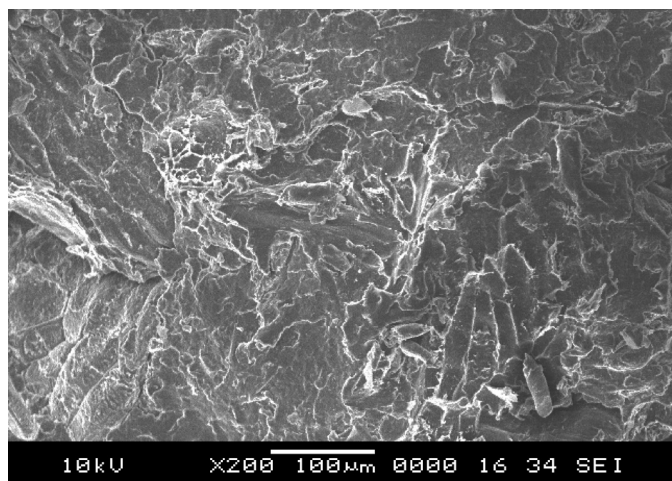


FIG. 8. Scanning electron micrograph of tensile fracture surface of modified rPP/CSP composites (40 php filler content).

hemicelluloses of CSP. The intensity of the peak is increased after modified with SDS. This is due to the additional peak attributed by the covalent bond between SDS and CSP. Figure 10 illustrates the chemical reaction between CSP and SDS.

Thermogravimetric Analysis (TGA) Study

The TGA and DTG curves of neat rPP, unmodified and modified rPP/CSP composites were showed in Figures 11 and 12. The TGA data was summarized in Table 3. From DTG curves show, neat rPP was decomposed in single step at temperature 250–500°C. The unmodified and modified rPP/CSP composites decomposed in 3 steps, included: i) decomposition of hemicellulose at temperature 200–350°C^[1–3], ii) decomposition of lignin and cellulose at

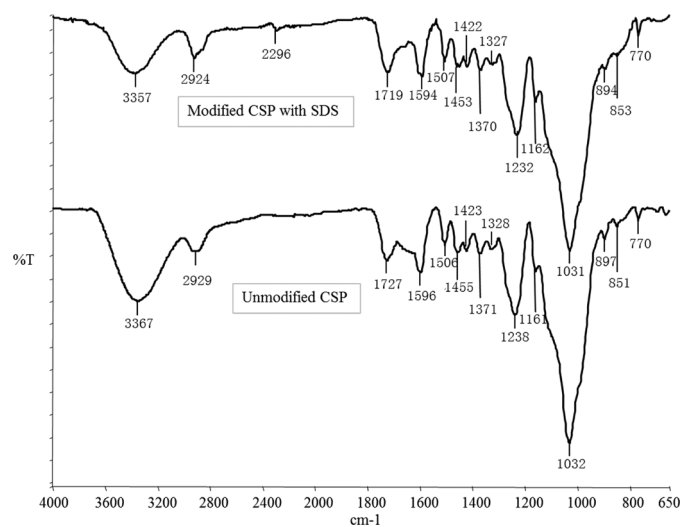


FIG. 9. FTIR spectra of unmodified and modified CSP with SDS.

TABLE 1
Formulation of rPP/CSP composites

Materials	Unmodified rPP/CSP	Modified rPP/CSP
PLA (php)	100	100
rPP (php)	0, 15, 30, 45, 60	15, 30, 45, 60
SDS (%)*	–	3

*3% based on weight of CSP.

temperature 350–400°C^[1–3], and iii) decomposition of rPP matrix at temperature 250–500°C.

From the temperature at 5% weight loss ($T_{d5\%}$) found that the rPP/CSP composites show a lower $T_{d5\%}$ compared to neat rPP and it increased with the increasing of the CSP content. The early thermal decomposition of rPP/CSP composites is attributed by the decomposition of low thermal stability hemicellulose from CSP. The decomposition temperature at maximum rate (T_{dmax}) of composites also reflected to the thermal decomposition temperature of rPP matrix. The T_{dmax} indicated the addition of CSP is significantly improved the thermal stability of rPP matrix at high temperature. The T_{dmax} and residue content at 700°C of rPP/CSP composites increased with CSP content, it mean CSP given thermal insulative behavior to rPP matrix.

This is because of the high thermal stability pyrolysis product from the thermal decomposition of CSP providing

TABLE 2
Main functional group of coconut shell powder

Wave number (cm ⁻¹)	Compounds
3000–3800	Hydroxyl (-OH) groups from cellulose, hemicellulose and lignin
2929	C-H stretching
1727	Carboxyl (C=O) groups of the acetyl group from hemicellulose
1596, 1506	Conjugated C-O group from aromatic skeletal in lignin
1455	C-H groups from lignin
1423	CH ₂ groups deformation from cellulose or C-H deformation in lignin
1371	C-H groups deformation in cellulose and hemicellulose
1238	C-O groups from acetyl group in lignin
1161	C-O-C groups of cellulose and hemicellulose
1000–1150	C-O-C and C-O groups from main carbohydrates of cellulose and lignin
700–900	C-H vibration in lignin

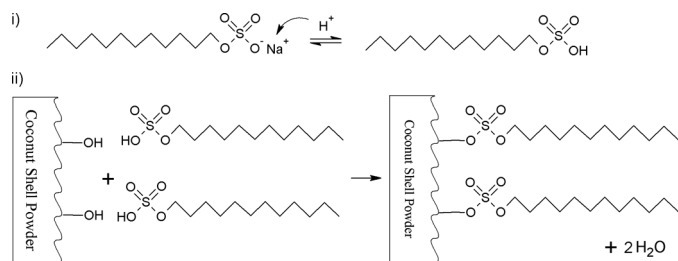


FIG. 10. Schematic reaction between CSP and SDS.

rPP matrix a thermal protecting layer and inhibits the process of thermal decomposition. Moreover, the modified rPP/CSP composites with SDS have better thermal stability than unmodified rPP/CSP composites as it can observe from the increment of $T_{d5\%}$, T_{dmax} and residue content at 700°C. It can be explained as the improvement of the filler-matrix interaction. According to Araujo et al.^[28] and Arbelaz et al.^[29], the curaua fiber and flax fiber modified with coupling agent exhibit an improved thermal stability of composites.

Differential Scanning Calorimetry (DSC) Study

Figure 13 shows the DSC curves for unmodified and modified rPP/CSP composites. The DSC data of both composites was summarized in Table 4. The crystallinity of rPP/CSP composites was reduced as the CSP content increased. This might be due to the presence of CSP hindered the crystallization of PP chains in composites. However, the modified rPP/CSP composites have higher crystallinity than unmodified rPP/CSP composites. It may be indicated that the modified CSP promote the migration and diffusion of PP chain to the CSP surface

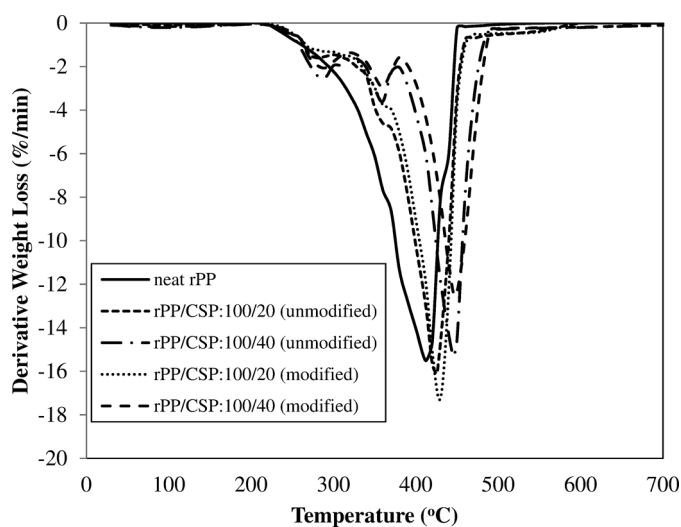


FIG. 11. DTG curves of neat rPP, unmodified and modified rPP/CSP composites.

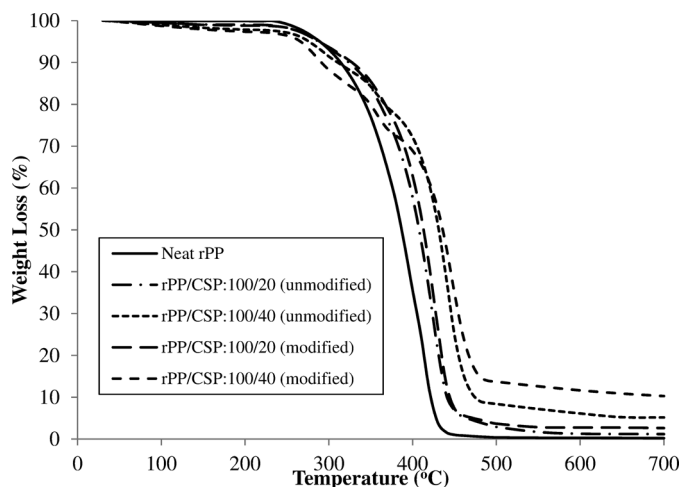


FIG. 12. TGA curves of neat rPP, unmodified and modified rPP/CSP composites.

TABLE 3
TGA data of unmodified and modified rPP/CSP composites

Sample	$T_{d5\%}$ (°C)	T_{dmax} (°C)	Residue at 700°C (%)
Neat rPP	290	412	0.22
rPP/CSP: 100/20 (unmodified)	287	426	1.19
rPP/CSP: 100/40 (unmodified)	266	445	5.16
rPP/CSP: 100/20 (modified)	298	431	2.63
rPP/CSP: 100/40 (modified)	276	448	10.27

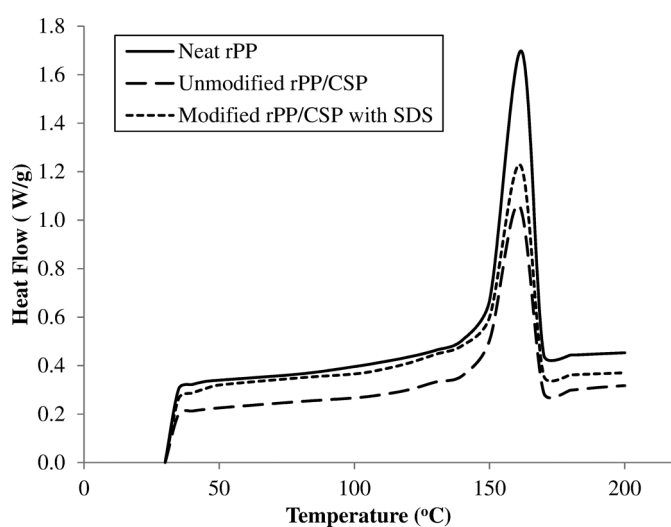


FIG. 13. DSC curves of neat rPP, unmodified and modified rPP/CSP composites.

TABLE 4
DSC data of unmodified and modified
rPP/CSP composites

Sample	T _m (°C)	X _c (%)
Neat rPP	163	43
rPP/CSP: 100/20 (unmodified)	163	41
rPP/CSP: 100/40 (unmodified)	162	29
rPP/CSP: 100/20 (modified)	161	34
rPP/CSP: 100/40 (modified)	162	32

for crystallization. Therefore, SDS modification of CSP shows nucleating effect on the rPP/CSP composites. The melting temperature (T_m) of rPP/CSP composites were not significantly change with the CSP content and SDS modification.

CONCLUSION

The increasing of CSP content was reduced the tensile strength and elongation at break of rPP/CSP composites. However, the tensile modulus of the composites increased after added CSP. The water absorption of rPP/CSP composites increased with CSP content and immersion time as presence of hydrophilic group from CSP. The poor tensile properties due to the incompatible between hydrophilic CSP and hydrophobic rPP matrix and weak interfacial interaction can be observed through SEM. The SDS modification was improved the tensile strength and tensile modulus of rPP/CSP composites. The water absorption of modified rPP/CSP composites was lower compared to unmodified rPP/CSP composites.

The FTIR result show modified CSPs have lower hydrophilic characteristics and might contribute to lower water absorption. The addition of CSP and SDS modification enhanced the thermal stability of rPP/CSP composites. The crystallinity of rPP/CSP composites was decreased at more CSP content, but the crystallinity of composites increased after modified with SDS. SEM micrograph was proven the SDS enhanced the filler-matrix interaction of modified rPP/CSP composites.

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