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RESEARCH ARTICLE

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Olive fiber reinforced epoxy composites: Dimensional Stability, and mechanical properties

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Abstract

The processing of olive (*Olea europaea L.*) oil results in large quantities of solid waste consisting primarily of tree pruning remainders from olive trees growing. This has led to the idea of utilizing the leftovers into a value-added product; natural composite materials are utilized as alternatives to environmentally damaging synthetic materials. This study deals with the evaluation of the filling properties of the residue; olive tree small branch (OTS), olive tree big brunch (OTB) and olive tree leaves powder, for epoxy matrix biocomposite. Olive powder reinforced epoxy composite was processed at 40% filler loading. In this paper, the various forms of impacts of filler within epoxy on the physical and mechanical of epoxy composites were scrutinized by tensile, flexural, impact, thickness swelling, and water absorption tests. From the observation, it was discovered that the tensile and flexural strength of epoxy composite reinforced with OTS and OTB increased by about 27% and 47%, respectively. The impact strength values of OTS and OTB epoxy composites were 8.61, 7.88, and 5.97 J/m, respectively, while the virgin epoxy increased up to 16 J/m. The microstructure of the composites is also analyzed using scanning electron microscopy to analyze interfacial bonding.

KEYWORDS

epoxy composites, mechanical properties, natural composites, olive tree

1 | INTRODUCTION

Advanced technology in composite materials has made a great contribution to humankind in modern engineering. Over the past decade, biocomposite production has become a very significant subset in the field of materials for household and industrial applications, primarily due to their high specific rigidity, low thermal expansion, fatigue behavior, and corrosion resistance.[1,2] The utilization of natural fibers in polymer matrix composites are not new to the industry revolution 4.0 due to the increasingly environmental awareness. The increased use of natural fibers, apart from their environmental benefits and renewable resources, as the most developed agricultural products bring wealth to rural areas, is very important for developing countries.[3,4] Additionally, natural fiber waste can be treated to create biodegradable, inexpensive, readily available, long-lasting, and robust

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polymer composites with high-performance fiber qualities.[5] Surface modification treatments were applied to the natural fiber reinforcing composite in order to boost the hydrophilic properties of the structure.[6] Lignocellulosic raw materials obtained from residues of olive are utilized as additives for polymer-based composites for the reason that biocomposites have been achieving more consent since the crisis of oil.[6,7]

In the context of the resource's efficiency, renewable composites and inexpensive materials play a significant role in the use of plant waste and agricultural fibers.[8,9] Natural fibers, which are used to substitute synthetic fibers are the best solution to that utilized in composites, where due to their degradability, availability, lightweight, and relatively cheap.[5,10] For the use of agricultural waste and fiber, such as palm oil, jute, sugar cane, bamboo, rice husk, sisal, etc., every country is trying to make use of plants growing on its land. The attempt to discover new fibers in the research community is still ongoing, with potentially important implications for the composites industry, particularly in engineering, medical, geosciences and geophysical disciplines, as well as on civilization.[8,11,12]

The olive tree (*Olea europaea L.*) is thought to be the very first trees to be grown in human history,[8] it is one of the most economically valuable fruit trees commonly planted in Mediterranean countries. Olive oil is high in unsaturated fatty acids and gives the human body many benefits.[13] As people consume olive, this illustrates the great economic and social value of this crop and the potential benefits to be obtained from the use of some of its by-products.[14] Alshammari et al.,[13] have reported on characteristics of Physico-chemical, thermal, and morphological properties on different parts of *Olea europaea L.* While Kaya et al.,[3] have investigated the properties of polypropylene (PP) composites reinforced with olive pomace (OP). The effect of treated olive husk flour (OHF) on the mechanical properties and thermal behavior of polyvinyl chloride composite also has been studied by Boukerrou et al.,[14] In 2020, Bartoli et al.,[15] evaluated the influence of the biochar morphologies relating to biochar epoxy composites and determined the tunability of the properties of composite in accordance with the biochar's thermal history. Researchers are trying to make efficient use of olive trees in the appropriate application areas to attain a value-added output.

In this experiment, in order to acquire new sustainable composites, olive tree small branch (OTS), olive tree big branch (OTB) and olive tree leaves (OTL) powder were obtained from pruning residues farms in Al-Jouf (Latitude: 32.29°; Longitude:42.37°) Kingdom of Saudi Arabia is mixed with epoxy resin. This research aims to show that whether OTS, OTB, and OTL, olive residues' byproducts can be utilized in a new polymer composite.

2 | EXPERIMENTAL WORK

2.1 | Materials details and composite manufacturing

This project entails a liquid epoxy resin (D.E.R.™ 324™) chemically a reaction product of epichlorohydrin and bisphenol A, supplied by Dow Chemical Pacific, Singapore, and corresponding hardener (Jointmine 905-3S) obtained from Epochemie International Pte Ltd, Taiwan is mixed in a ratio of 2:1 by weight. As specified in Table 1, epoxy resin and hardener were used as received from the manufacturer.

Three types of olive samples were classified: leaves (OTL), a bough which was a main branch of olive (OTB) and twigs (OTS). The OTL, OTB, and OTS samples were put away in a research facility oven for 3–4 days at a temperature extend of 70–80°C before being pulverized to a fine powder with an average particle size of 250–500 μm using a grinder. After drying in the oven at 60°C for 24 h, the OTL, OTS, and OTB are reinforced in epoxy resin with total filler loading at 40% by weight (density 1.2 g/cm^3). The mixture is then mechanically stirred (epoxy filled with different olive parts) and gradually poured into a stainless-steel mold with the necessary dimension of 150 × 150 × 4 mm, then coated with a thin silicon spray layer. The entire mixture in the mold is cured at a hot press for 110°C for 10 min under a constant pressure of 250 bar before it is moved to a cold press. The composite was than cooled in a cold press under a constant pressure of 250 bar for 5 min before it is demolded for testing. An epoxy without reinforcement was prepared as a reference .

2.2 | Tensile analysis

In agreement with the standard of ASTM D 638–03, the tensile assessment was carried out using the Bluehill INSTRON 5567 Universal Testing Machine (Shakopee, USA) with a grasp on control of 30 kN. The measurement was carried out at an encompassing temperature of 23 ± 3°C also relative humidity of 50 ± 10% using a sample dimension of 160 × 20 × 5 mm. The testing speed for the three different filler specimen's types (as OTL, OTS, and OTB) was calculated by using Equation 1, while the support span was 16 times the specimen depth. The standard specimen was placed at the ends of the test instrument's holding grips. The machine is outlined to prolong the test sample at a uniform rate and ceaselessly and cumulatively calculate the momentary connected load and the resultant prolongation via the extensometer.

$$R = \frac{0.01L^2}{6d} \tag{1}$$

TABLE 1 Properties of epoxy resin type D.E.R.™ 324 (information P, resin LE. D.E.R.™ 324 n.d.:1–5) and Jointmine 905-3S properties (LTD EIP, cycloaliphatic amines, 757,516)

Epoxy: D.E.R.™ 324	Density 25°C (g/ml)	Binder gel time (100g) (min)	Tensile strength (MPa)	Flexural strength (MPa)	Compressive Strength (MPa)
	1.16	24	13.7	31.4	87.9
Hardener: Jointmine 905-3S	Amine value (mg KOH/g)	Viscosity (25°C, cPs)	Color (Gardener)	Pot life (mins)	Type
	330 ± 20	200~400	<3	60~70	Cycloaliphatic amine adduct

where R is the rate of crosshead movement (mm/min), L is the span of support (mm), and d is the beam depth (mm).

2.3 | Flexural analysis

Conferring to the standard of ASTM D 790–03, the three-point flexural analysis was tested using Bluehill INSTRON 5567; Shakopee, USA at 23 ± 3°C and relative humidity of 50 ± 10%. The piece test rectangular cross-section rests on two underpins and is stacked midway between the anchors by implies of a stacking nose. The nose and anchors for loading have a hollow surface. From the slant of the initial portion of the load-deflection bend, the flexural modulus is measured.

2.4 | Scanning electron microscope analysis

The microstructure images of the fracture surface morphology of the flexural composite sample were also taken using scanning electron microscope (SEM) type; the EM-30AX (SEM; COXEM, Daejon, Korea) to examine the failure. The picture is captured at a magnification rate of 1000 times at a power supply length of 10 μm at 20 kV. All samples surface was coated with gold before SEM tests.

2.5 | Impact test

Notched Izod impact specimens (70 × 15 × 5 mm) were prepared and tested using Gotech GT-7045-MD (Taichung City, Taiwan) in compliance with the standard of ASTM D256 (2010). The angle of the notch was 45°, and it was 2.5 mm in diameter. There were five replications tested in every study.

2.6 | Water absorption and thickness swelling

The water absorption and thickness swelling rate for the test sample with a dimension of (20 × 20 × 5 mm) were prepared and tested by following the standard of ASTM D 570–988 (2010). Prior to immersion in distilled water, the initial weight and thickness of the test specimen were measured and recorded. The test of specimen's weight and thickness were measured and recorded every 24 h for a week. Water absorption and thickness swelling of the composites were calculated using Equation 2 and Equation 3, respectively.

$$Water\ Absorption\,(\%) = \frac{W_n - W_d}{W_d} \times 100 \quad (2)$$

where W_n is the post-immersion weight of the samples and W_d is the pre-immersion weight of the samples.

$$Thickness\ Swelling\,(\%) = \frac{T_1 - T_o}{T_0} \times 100 \quad (3)$$

where the thickness that swells after soaking is T_1 and the thickness before soaking is T_o.

3 | RESULT AND DISCUSSION

3.1 | Tensile properties

The variation tensile properties; tensile strength and Young's modulus were evaluated as a function of load rate presented in Figure 1, while the stress–strain curve was presented in Figure 2. The tensile test is an inherent property that represents the resistance of the material.[16] As tabulated in Figure 1, tensile strength values were 27.87, 27.97, and 19.61 MPa for OTS, OTB, and OTL epoxy composites, respectively, while virgin epoxy was 20.43 MPa. Apart from OTL composites, the tensile

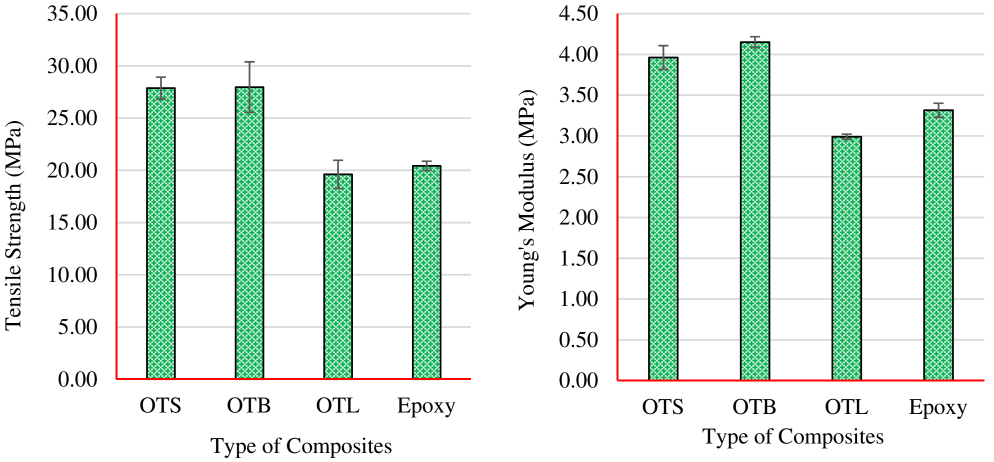

FIGURE 1 Tensile strength and modulus of epoxy filled OTS, OTB, and OTL composites

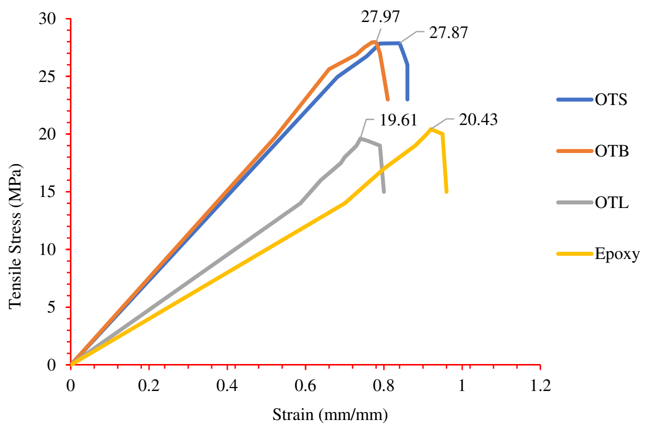

FIGURE 2 Tensile stress–strain curves of epoxy composite filled with OTS, OTB, and OTL fiber

strength of epoxy encompassing olive powder was higher than virgin epoxy, as shown in Figure 1. In general, the epoxy composite reinforced with natural filler exhibited higher tensile strength, which is consistent with previous findings by Huang and Young,[17] Sarikaya et al.,[18] and Ramana, and Ramprasad.[19] Moreover, the tensile strength of the OTB composite exhibited slightly higher compared to the OTS composite. It is noted that OTL composite tensile strength decreased from 20.43 to 19.61 MPa after the incorporation of OTL into epoxy. Based on the finding by Parbin et al.,[20] certain factors have an impact on the tensile properties of the composites manufactured with natural fiber reinforcement, such as moisture absorption, fiber alignment, fiber treatment, fiber distribution, and the use of additives. In addition, based on Magalhães et al.,[21] irregular formed fillers do not include to the tension exchanged to the polymer matrix. In this circumstance, the integrated material coordinates as a filler instead of fortification. This was proven by Alshammari et al.,[13] in their research where compared to olive small branch (OTS) and olive big branch (OTB), olive leaves (OTL) have an asymmetrical size distribution. The OTS particle, meanwhile, was smaller than the OTB particle. It may have been the product of the contrasting configurations of the branch fibers, that is, the more circular form of the OTS, in comparison to the OTB, whichever was like the longitudinal shape.

The modulus of elasticity, or Young's modulus, portrays a material's rigidity: the higher it is, the more rigid it is.[22] Differences between types of composites with the same load rate were observed in modulus values and the highest values were reported with OTB composites (4.15 MPa). The Young's modulus of OTS and OTB composites were increased by 19.3% and 25% while the OTL composite was decreased by 9.6% in comparison to the virgin epoxy. This figure also simply proves the increase of Young's module by incorporating it into the OTS composites. This emergence in the Young modulus indicates that the composites' rigidity has increased. Enhanced Young's modulus of composite within the nearness of filler particles may be due to diminished macromolecular versatility and deformability.[23] It can be admitted that with the fusion of OTS and OTB filler in epoxy, epoxy becomes stiffer.

3.2 | Flexural test

The experimental findings of the composites' flexural strength are shown in Figure 3. The olive fillers; OTS, OTB, and OTL were noted to contribute of improving the composites' flexural strengths as well as their flexural modulus. The composites containing OTB filler was the optimum ones with respect to the aforementioned mechanical strengths. The OTB composites had the best flexural strength among the produced composites, which was 87% higher than the control sample (virgin epoxy). The flexural strength of the epoxy composite containing

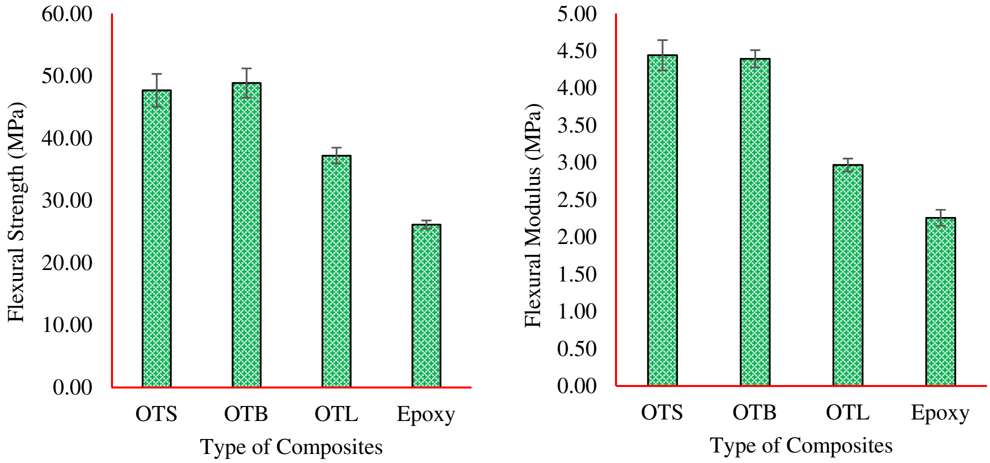

FIGURE 3 Flexural strength properties and flexural modulus of epoxy filled OTS, OTB, and OTL composites

OTS, OTB, and OTL are 47.71, 48.98, and 37.22 MPa, respectively, in comparison to the virgin epoxy (26.15 MPa), which are about 82.45%, 87.3%, and 42.33% higher than those of the virgin epoxy resin. As shown in Figure 3, there were remarkable findings that the flexural modulus increased by 96.46%, 94.69%, and 31.42% when the virgin epoxy was incorporated with OTS, OTB, and OTL fillers, respectively.

It is possible to justify that the addition and types of olive fiber have an impact on the flexural strength and modulus of these epoxy composites. Many researchers[17,19,20] have also indicated that insufficient filler dispersion is the main reason of poor mechanical strength. Flexural modulus can be used to define the stiffness of a material. In this scenario, the inclusion of OTB, OTS, and OTL fibers increases the flexural modulus. This is thought to be due to the high stiffness attribute of olive fibers, which has a positive effect on the overall performance of the epoxy composites.

It is possible to explain the reasons for these patterns by using the same reasons under tensile properties. The OTL composite performed lower in both flexural strength and modulus, 37.22 and 2.97 MPa, respectively. This could occur due to the non-uniform OTL shape contributing to poor filler weighting, which helps to quickly debond the matrix. Furthermore, the composites' flexural strength was reduced might due to the poor dispersion of OTL fiber in epoxy resin. Due to the entanglements of the fibers, OTL agglomerated zones occur. This could operate as stress concentration spots, causing composites to fail. As a result, flexural strength will deteriorate. According to many experts,[18,21,23,24] the reinforcing efficiency of natural fibers in a matrix is affected by a variety of factors such as fiber dispersion in the matrix, volume fraction of fillers, matrix and filler bonding types, and aspect ratio. On the other hand, it should be emphasized that natural fibers have always posed a number of challenges, particularly in the development of high-performance natural fiber-reinforced polymer composites. The propensity of olive fibers to absorb moisture from the atmosphere causes weight and dimension stability to change. Olive fibers' hydrophilic properties have an effect on the weak interfacial interaction between fibers and matrix. Because of this issue, the epoxy composites reinforced OTS, OTB, and OTL will have poor strength and stiffness.

3.3 | Impact properties

The experimental results of the impact strength test for the composite are shown in Figure 4. It appears the comparison of affect strength of OTS, OTB, OTL, and virgin epoxy composites at 40% filler loading. The impact strength values of OTS, OTB, and OTL epoxy composites are 8.61, 7.88, and 5.97 J/m, respectively, while the impact strength of virgin epoxy is 16.14 J/m. The OTS composite displays the maximum impact strength when

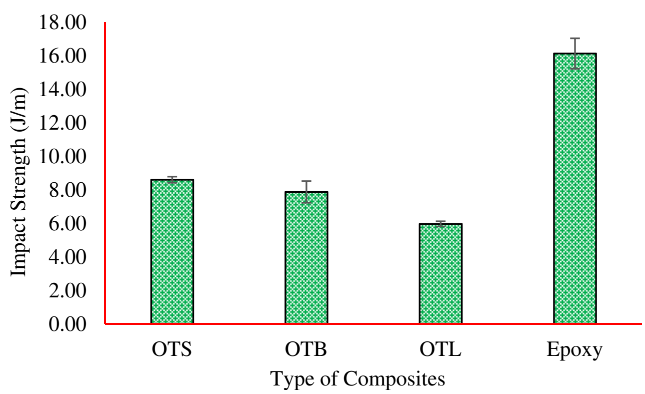

FIGURE 4 Impact strength of epoxy filled OTS, OTB, and OTL composites

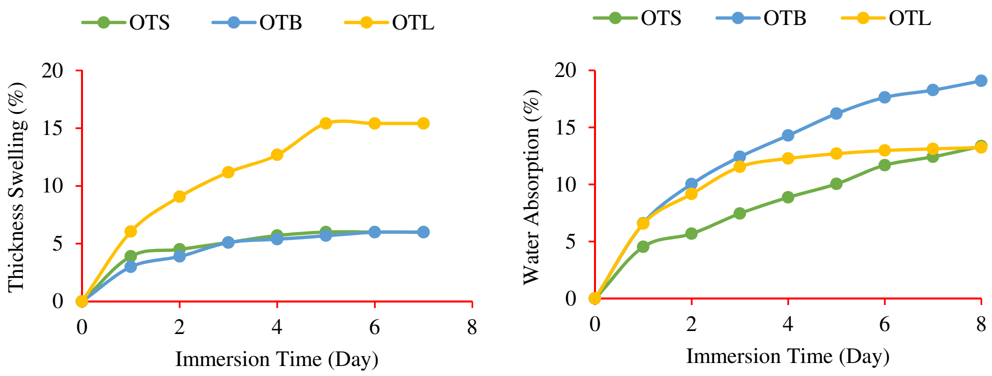

FIGURE 5 Thickness swelling, and water absorption rate of epoxy filled OTS, OTB, and OTL composites

olive tree small branch filler reinforced in the epoxy matrix. This could be attributed to the great interfacial stability of OTS powder with epoxy matrix capability to cope against impact loading. This approach is employed in the construction of airplane bodies to withstand large impact loads imposed by air.[24]

The impact strengths of fiber-reinforced composites are primarily determined by the properties of individual reinforcing fibers, fiber layout and alignment, fiber intertwining, and fiber to resin adhesion.[13] In this case, the reduction in impact strength of the OTS, OTB, and OTL epoxy composites is mainly due to the low strength of natural fiber compared to synthetic fiber. The impact strength graph clearly reveals that the insertion of olive fibers worsens the impact strengths mostly due to the diversion of applied energy, which then allows fracture initiation, crack pinning mechanism, and crack propagation inside the composite materials under stress load. The results are also in line with the findings for jute-carbon/epoxy composite,[25] epoxy treated natural fiber reinforced PLA composite[26] and clams shell in impact properties of jute epoxy composite.[27]

3.4 | Thickness swelling (TS) and water absorption (WA) rate

The outcomes of TS and WA test are shown in Figure 5. It is clear from Figure 5 that with the water absorption, TS of composites increases and thus the correlation between TS and weight gain due to water absorption is similarly indicated. OTL composite showed the highest rate of TS, which is 15.4% after 7 days of immersion. In contrast, TS for OTS and OTB composite showed a moderate rate, with the values 6.0% and 5.98%, respectively. Epoxy resin prevents water absorption of water into the composite because epoxy resin creates a matrix that is water-resistant.[28] However, the weakness of the natural fiber itself, which is badly water-resistant due to the presence of polar groups, will accumulate water molecules by hydrogen bonding.[29] Due to reversible and irreversible swelling of the composite, this is responsible for dimensional changes of epoxy composites, particularly thickness and linear expansion.

From Figure 5, it can be observed that, as time passes, water molecules penetrate deeper into the OTL composite was 2 to 3 times higher compared to OTS and OTB composites. As mentioned by Alshammari et al.,[13] they found fiber shape sizes of olive leaves was non-uniform and tend to agglomerate. Consequently, the higher TS rate of OTL composite can be expected due to the exposure of the OL particle on the surface of the composite.

Water absorption values were found to be increased with immersion time, as shown in Figure 5. The hydrophilic nature of the cellulosic materials allowed the composites to absorb water rapidly within the first several hours, allowing them to retain a large volume of water. It shows that the WA of OTS, OTB, and OTL composites were increased by 13.35%, 19.09%, and 13.24%, respectively after 7 days of immersion. Indifference to TS rate,

TABLE 2 Density of epoxy filled OTS, OTB, and OTL composites

Specimens	Width, W (mm)	Length, L (mm)	Thickness, T (mm)	Mass, m (g)	Fiber volume friction, V (g/cm^3)	Density, ρ (g/cm^3)
OTS	20.22	19.77	0.33	1.51	1.33	1.14
OTB	2036	19.93	0.33	1.47	1.35	1.09
OTL	19.92	19.85	0.33	1.59	1.31	1.22

OTB composite resulted in the highest value of water absorption compared to OTL composites. It indicates clearly that the WA rate of the OTB composite tends to absorb moisture more than the OTL composite. The higher WA of OTB composites might be attributed to higher cellulose content in its structure which have been observed by Alshammari et al.,[13] who claimed that they contain cellulose quantities according to the following order: OTB>OTS>OTL. This is also supported by the fact that the water absorbed by the OTS, OTB, and OTL epoxy composites significantly affected by its density (Table 2). The density of OTB composite is 1.086 g/cm^3, which is lesser than OTL composites, 1.220 g/cm^3 and OTS composite, 1.137 g/cm^3. The lowest density of OTB composite may be due to the existence of voids within the composite, thus leads to greater WA. In addition, this could also be attributed to the establishment of micro-channels, which lead to greater WA levels.[24]

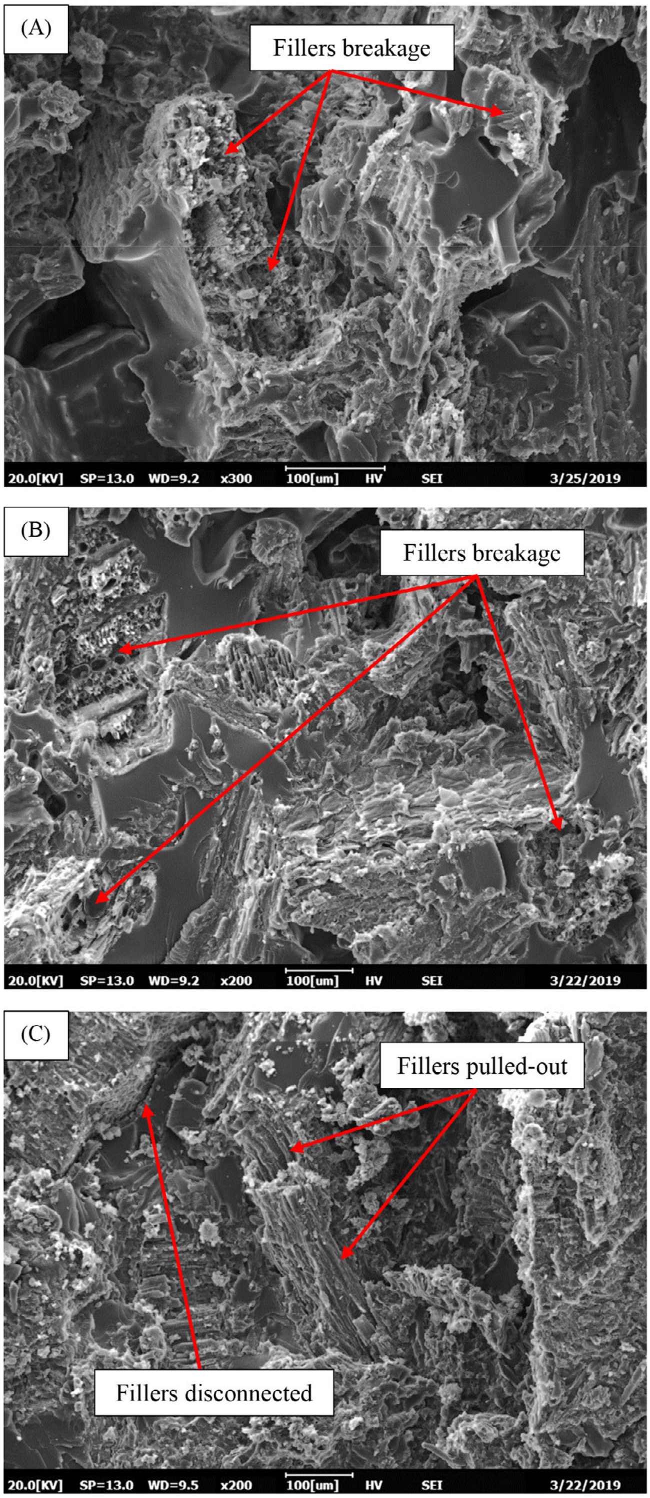

FIGURE 6 SEM micrographs tensile fracture surface of the specimen of epoxy filled (A) OTS (B) OTB, and (C) OTL composites

3.5 | Scanning electron microscopy

Rupture surfaces of the epoxy composite encompassing OTS, OTB, and OTL are shown in Figure 6. Fillers breakage and pull-out were observed throughout the matrix. It can be seen from Figure 6A,B, there was a fillers breakage, which indicates that interfacial bonding between fillers and matrix was slightly stronger than in the OTL composite (Figure 6C). The stress can be pretended to be methodically shifted after the matrix to the fillers. Hence, the composites' tensile strengths and flexural strengths improved with the addition of OTS and OTB fillers. Filler pull-out was traced in OTL composite, Figure 6C which demonstrated a lack of interfacial linking between OTL fillers and the epoxy matrix. Furthermore, the fracture surface showed omnidirectional rupture, which could indicate that the matrix strength was compromised under stress. The findings of mechanical tests were compared to composite SEM pictures.

4 | CONCLUSION

This study investigated the manufacturing and characterization of OTS, OTB, and OTL filled epoxy composites. Tensile and flexural strengths of the composites increased except for tensile strengths filled with OTL composite. A similar trend also experienced by Young's modulus and flexural modulus. With the inclusion of the olive filler, the impact strength of the epoxy composites was lowered by around 50% compared to virgin epoxy. Water absorption and thickness swelling rose with immersion time for all types of composites during the water immersion experiments. OTL composite experienced the highest rate of thickness swelling while OTB composite exhibited the highest rate of water absorption. SEM morphological analysis showed that there was filler pull-out in OTL composite and filler breakage in OTS and OTB composite which indicate the interfacial bonding between the matrix and filler. This study demonstrated that OTS and OTB, the residues of an olive plantation, can be used as a filler in epoxy matrix materials.

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