Effect of Fiber Content and Water Absorption Behaviour on the Mechanical Properties of Screw Pine Fiber-reinforced Vinyl Ester Composites

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This study examines how water exposure affects the mechanical properties of randomly oriented screw pine fiber reinforced vinyl ester composites. Composite specimens have been produced using the compression moulding process with three different fibre content values (12.75, 26.19, and 40.39 vol.%). For 5 days, the produced composite specimens were immersed in three different water environments: seawater, groundwater, and distilled water, to investigate the effect of water absorption on the mechanical properties of composites. The mechanical properties of composites under wet conditions were compared to those in dry conditions, and they were found to decrease when water particles were absorbed. In both dry and wet conditions, the composite with a volume of 26.19 % has the highest tensile, flexural, and impact strength values. Mechanical properties were drastically lowered in wet conditions compared to dry conditions. It was revealed that exposure to seawater has a significant effect on composite properties when compared to ground and distilled water exposure. The fractured surface of composite specimens was examined under a scanning electron microscope following exposure to three different water conditions. Scanning electron microscopy revealed resin losses, void formation, and microcracks on the surface of the wet composite specimens.
Keywords: screw pine fibers, vinyl ester, water absorption, mechanical properties, scanning electron microscopy.

1. INTRODUCTION

In recent years, as per the environmental aspect synthetic fibers and particles are being replaced by the plant based natural cellulose fibers and particles in the polymer composite system [1 – 3]. An extensive range of investigations conducted by researchers on natural fibres and particles showed their potential as excellent reinforcing agents in polymer matrices [4 – 7]. It has a large attention among material engineers for its desired qualities of easy availability, cost-effective, lower density, eco-friendly, and renewability as well as appropriate mechanical properties making them a potential replacement for synthetic fibers [8 – 10]. Although natural fibres provide numerous advantages, they also have certain disadvantages, such as a higher moisture absorption tendency, low thermal stability, and quality variations, among others. Among these, moisture absorption by natural fibres causes dimensional changes in polymer composites, resulting in composite failure.

Several researchers recently reported on the effects of moisture or water absorption on the mechanical properties of natural fiber-reinforced polymer composites. The moisture absorption behaviour of the natural fibre influences the long-term performance of the composite [11, 12]. Kim and Seo [13] studied the effect of water absorption fatigue on the mechanical behaviour of sisal fibre reinforced epoxy composites and reported that water absorption has significant effects on composite mechanical properties. Das and Biswas [14] investigated the effect of different fibre lengths and content on the mechanical properties of coir fibre reinforced epoxy composites at wet conditions. The authors found that mechanical properties reach their highest level at 12 mm of fibre length and 15 wt.% fibre content. They also found that as the fibre content and length increased, correspondingly increased the hardness and tensile modulus. Janis et al. [15] examined how wood fibre content and coupling agent concentration affect WPC mechanical properties. The composites' microhardness, flexural properties, and wood fibre reinforcement improved. As fibre content increased, polymer melt impact strength, water resistance, and fluidity decreased. Virgin and recycled HDPE composites reinforced with coniferous wood fibres had good mechanical properties. The optimal wood fibre content was 50 – 60 wt.%. Emrah and Mürşit [16] investigated HDPE and $CaCO_3$ coated/pigmented wood-free paper fibre

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composites' physical, mechanical, and thermal properties. Cellulose-HDPE composites with higher $CaCO_3$ ratios had better moisture resistance, flexural strength, and tensile strength. The density of cellulose-HDPE composites increased by $CaCO_3$. The plastic matrix had appropriate $CaCO_3$ and cellulose fibre dispersion, according to scanning electron microscopy. As $CaCO_3$ increased, DSC analysis showed sample crystallinity decreased. Sekar et al. [17] evaluated the effects of three different chemical treatments on the mechanical properties of Phenol Formaldehyde (PF) composites reinforced with Calotropis Gigantea Fibres, based on the fibre content. The results showed that the composites exhibit superior mechanical properties at 40 wt.% in the untreated condition. Composites made with alkali-treated fibres exhibit superior mechanical properties when compared to other treated fibre composites. Athijayamani et al. [18] studied the mechanical properties of vinyl ester hybrid composites reinforced with alkali-treated Smilax zeylanica and sisal fibres under wet conditions. The results revealed that the percentage of water particle absorption in alkali-treated hybrid fibre composites is lower than in untreated hybrid fibre composites. The mechanical properties of alkali-treated hybrid fibre composites were marginally reduced when wet composites compared to dry composites. The alkali-treated smilax zeylanica and sisal fiber-reinforced vinyl ester hybrid composites exhibit greater resistance to water particle penetration. Dinesh et al. [19] investigated the mechanical properties of date seed and neem powder reinforced epoxy composites. The results demonstrate that adding neem gum powder in increasing quantities reduces mechanical properties such as tensile and flexural. It does not, however, affect the composite material's impact strength. The water absorption behaviour of a hybrid interwoven cellulosic-cellulosic fibre reinforced epoxy composite (kenaf/hemp and kenaf/jute) and its effect on its mechanical properties were examined by Maslinda et al. [20]. The infusion process was used to produce the composites. The composite specimen was immersed in tap water at room-temperature until it reached the saturation point in order to perform the water absorption test. The authors concluded that fibre hybridisation enhanced mechanical and water-resistant characteristics. Although numerous studies have been conducted on the effect of water or moisture absorption on the mechanical properties of natural fibre polymer composites, the investigation into fibre geometries and water absorption behaviour has been limited to the Screw Pine Fibre (SPF) reinforced Vinyl Ester (VE) composite. As a result, the current study investigated the effect of water absorption behaviour and fibre content on the mechanical properties of the randomly oriented SPF/VE composite. The composites were produced using a hot press compression moulding process with varied fibre content. The fibre length was kept constant at 13 mm. The fractured surfaces of the composite specimens were investigated with scanning electron microscopy (SEM).

2. EXPERIMENTAL DETAILS

2.1. Materials

The plant known as the screw pine (Fig. 1a and b) is in the genus *Pandanus*. It comes from the word "*Pandan*" which is the Indonesian name for a tree that is also known as "screw pine" and is in the *Pandanaceae* family. The SPFs (Fig. 1 c) were derived from the stem of the screw pine plant and utilised as reinforcement in their natural state. Table 1 shows the chemical composition of the screw pine fibers. The polymer resin matrix utilised in this work was a VE resin procured from GVR Enterprise in Madurai, Tamil Nadu, India. The resin was combined with a catalyst (6 % cobalt naphthenate), promoter (N–N dimethyl aniline), and accelerator (Methyl Ethyl Ketone Peroxide).

a b

c

Fig. 1. Digital images: a, b – screw pine plant (pandanaceae family); c – extracted screw pine fiber bundle

Table 1. The chemical composition of the screw pine fibers

Composition	Percentage, %
Cellulose	38 – 53
Hemicellulose	7.5 – 12.6
Lignin	Around 5.3
Wax	Around 0.3
Moisture	Around 5.6.

2.2. Preparation of composites

The compression moulding technique (Fig. 2) was used to produce the composite plates using a mould box of 150 × 150 × 3 mm.

Fig. 2. Digital image of hot press compression moulding machine [21]

The pressure was set at 2 bars, while the temperature was set at 120°C. Before the process, the releasing agent was applied by spraying it into the mould box to facilitate the effortless removal of the cured composite plate.

The SPF was carefully chopped for the required geometry and then combined with VE resin utilising a mechanical stirrer for 30 minutes. The catalyst, promoter, and accelerator were added into the mixture and subsequently agitated for 15 minutes using a mechanical stirrer.

After that, the mixture was put into the mould and placed within the moulding machine under closed conditions. After removing the mould box from the machine, it was thereafter left to cure at room temperature for 12 hours. Afterward, the cured composite plates were cautiously extracted and precisely cut in accordance with the mechanical testing standards specified by ASTM.

2.3. Mechanical testing

The composite specimens were obtained by cutting them from the manufactured composite plates. Following that, these specimens were subjected to mechanical tests, which included tensile, flexural, and impact tests.

The maximum tensile stress a material can withstand before failing is known as its tensile strength. A uniaxial load is applied via the specimen's two ends during the test. The specimen is 150 × 20 × 3 mm. A computerised universal testing machine was used to perform a tensile test, following the ASTM D 638-14 [22] standard. The test was conducted at a crosshead speed of 2 mm/minute, using a gauge length of 50 mm.

The flexure test method evaluates the behaviour of materials subjected to simple beam loading. Typically, the specimen is placed on a support span and the load is given to the centre by the loading nose, resulting in three points bending at a specified speed. The size of the specimen is 150 × 20 × 3 mm. The test was performed using the same machine as specified by ASTM D 790-17 [23]. The experiment was conducted at a crosshead speed of 2 mm/minute, using a gauge length of 50 mm.

The impact test measures the toughness and notch sensitivity of engineered materials. Izod Impact test specimens with size of 65 × 20 × 3 mm are machined into square or round sections. The specimen is clamped vertically on the anvil towards the Hammer. An Izod impact test was performed using an Izod impact testing apparatus in accordance with ISO 180:19 [24]. Each combination was investigated using a total of five samples, and the resulting mean values were recorded.

2.4. SEM study

The Hitachi S-3000N scanning electron microscope was utilised to examine the fractured surfaces of the composite specimen subsequent to the mechanical test.

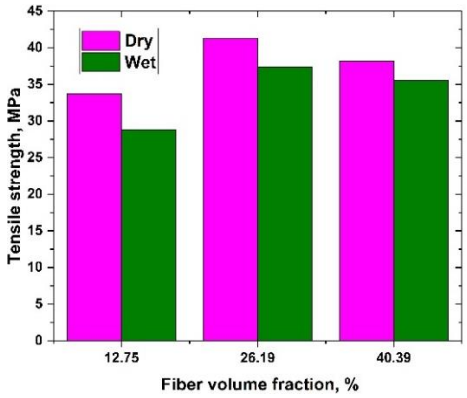
3. RESULTS AND DISCUSSION

3.1. The effect of fiber content and seawater exposure on the properties of composite

Mechanical tests such as tensile, flexural, and impact were conducted on both dry and wet composite specimens to investigate the effect of seawater absorption on the mechanical properties of the composites.

3.1.1. Tensile strength

Fig. 3 a shows the tensile strength of the SPF/VE composite in both dry and wet conditions. Significantly, the tensile strength of the SPF/VE composite increased up to a volume fraction of 26.19 % at both the dry and wet conditions and subsequently decreased. Compared to a dry condition composite with 12.75 vol.% fibre, the composites with 26.19 vol.% fibre showed an increase of 22.6 % in tensile strength. Nevertheless, the tensile strength value of composites with a volume fraction of 40.39 % decreased by 7.5 %, when compared to composites with a volume fraction of 26.19 % under dry conditions.



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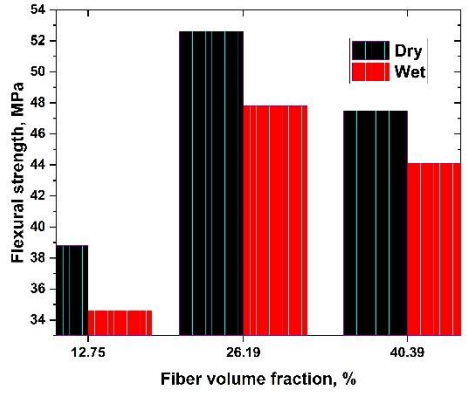

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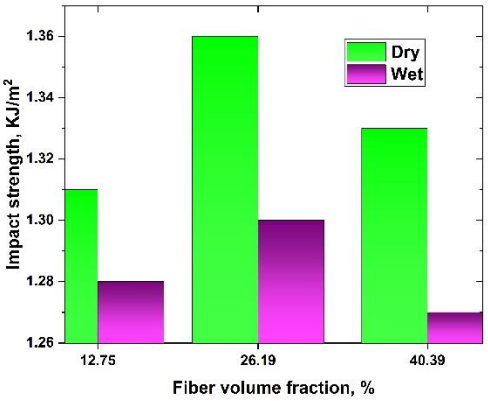


c

Fig. 3. Mechanical properties: a – tensile; b – flexural; c – impact of composite exposed to the seawater environment

In addition, the tensile properties of the SPF/VE composite significantly decreased when exposed to wet conditions, in contrast to the attributes observed in the dry composite specimen. Even in wet conditions, tensile properties exhibited a decrease following the inclusion of 26.19 vol.% of fibres. The tensile strength value of composite with a volume fraction of 40.39 % dropped by 5.1 % compared to composites with a volume fraction of 26.19 % at wet conditions. The wet composite specimens with 26.19 vol.% of fibre content, exhibited a reduction of 9.4 % in tensile strength compared to the dry composite specimen. At a composite volume fraction of 12.75 %, reduction of 14.5 % was found in tensile strength compared to the dry composite with a volume fraction of 12.75 %.

3.1.2. Flexural strength

The flexural properties of SPF/VE composite in both dry and wet conditions are presented in Fig. 3 b. The same trend observed during tensile tests was also identified during flexural testing of SPF/VE composite specimens. Significantly, the flexural properties of the SPF/VE composite increased up to a volume fraction of 26.19 % at both the dry and wet conditions and subsequently decreased.

Compared to a dry condition composite with 12.75 vol.% fibre, the composites with 26.19 vol.% fibre showed an increase of 22.6 % in flexural strength. Nevertheless, the flexural strength values of composites with a volume fraction of 40.39 % decreased by 9.7 % when compared to composites with a volume fraction of 26.19 % under dry conditions.

In addition, the flexural properties of the SPF/VE composite significantly decreased when exposed to wet condition, in contrast to the attributes observed in the dry composite specimens. Even in wet condition, the flexural properties exhibited a decrease following the inclusion of 26.19 vol.% of fibres. The flexural strength value of composite with a volume fraction of 40.39 % dropped by 7.7 % compared to composites with a volume fraction of 26.19 % at wet conditions.

The wet composite specimen, with 26.19 vol.% of fibre content, exhibited a reduction of 9.1 % in flexural strength compared to the dry composite specimen. At a composite volume fraction of 12.75 %, a reduction of 10.8 % was found in flexural strength compared to the dry composite with a volume fraction of 12.75 %.

3.1.3. Impact strength

Fig. 3 c presents the impact strength of the SPF/VE composite in both dry and wet conditions. Significantly, the impact strength of the SPF/VE composite increased up to a volume fraction of 26.19 % at both the dry and wet conditions and subsequently decreased. Compared to a dry condition composite with 12.75 vol.% fibre, the composites with 26.19 vol.% fibre showed an increase of 3.8 % in impact strength. Nevertheless, the impact strength values of composite with a volume fraction of 40.39 % decreased by 2.2 %, when compared to composite with a volume fraction of 26.19 % under dry conditions.

In addition, the impact strength of the SPF/VE composite significantly decreased when exposed to wet conditions, in contrast to the attributes observed in the dry composite specimens. Even in wet conditions, the impact strength exhibited a decrease following the inclusion of 26.19 vol.% of fibres. The impact strength value of composite with a volume fraction of 40.39 % dropped by 3.1 %, compared to composite with a volume fraction of 26.19 % at wet conditions. At a composite volume fraction of 12.75 %, a reduction of 3.1 % was found in impact strength, compared to the dry composite with a volume fraction of 12.75 %. Composites containing 26.19 and 40.39 vol.% of fibre showed almost identical impact strength values under wet condition, as shown in Fig. 3 c.

The reduction of mechanical properties of the composite specimens resulted from the immersion of composite specimens in seawater environments, leading to the formation of hydrogen bonds between water molecules and cellulose. The penetration of water particles into the fiber-matrix area resulted in dimensional changes of the composite specimens and weakened interfacial bonding, thereby decreasing the mechanical properties. Dimensional changes occurred owing to fibre expansion, resulting in fibre separation from the matrix resulting in weak fiber-matrix adhesion. The poor interface decreased the composite's elongation property, failing crack propagation [25].

Fig. 4 a depicts the loss of resin content on the surface of the composite specimen, as well as void forms caused by seawater exposure. The exposure to sea water altered the characteristics and structure of the fibres, matrix, and their interface. Fig. 4 b illustrates crack formation caused by damage to the fiber-matrix contact. Capillarity and transport via micro fractures become active following composite deterioration. Cracking causes composite specimens to deteriorate quickly. Water particles actively attack the composite interface, causing the fibre and matrix de-bonding, as illustrated in Fig. 4 c.

3.2. The effect of fiber content and groundwater exposure on the properties of composite

The influence of fibre content and groundwater absorption on the mechanical properties of composites is demonstrated in Fig. 5 a – c.

3.2.1. Tensile strength

The effect of fibre content and groundwater absorption on the tensile strength of composites is illustrated in Fig. 5 a. As found in the seawater environment, the tensile strength of wet composites decreased as compared to dry composites in the groundwater environment. A linearly increasing tendency was also found up to 26.19 vol.% in wet conditions before dropping. Composites have a maximum tensile strength of 38.5 MPa, when the fibre content is 26.19 vol.% in wet conditions. A composite with a fibre content of 12.75 % has a tensile strength of 33.7 MPa at dry and 29.6 MPa under wet condition, respectively, resulting in a 12.2 % of reduction.

3.2.2. Flexural strength

Fig. 5 b presented the influence of fibre content and ground water absorption on the flexural strength of composites. As found in the seawater environment, the flexural strength of wet composites decreased as compared to dry composites. A linearly increasing tendency was also

found in flexural strength up to 26.19 vol.% in wet conditions before dropping.

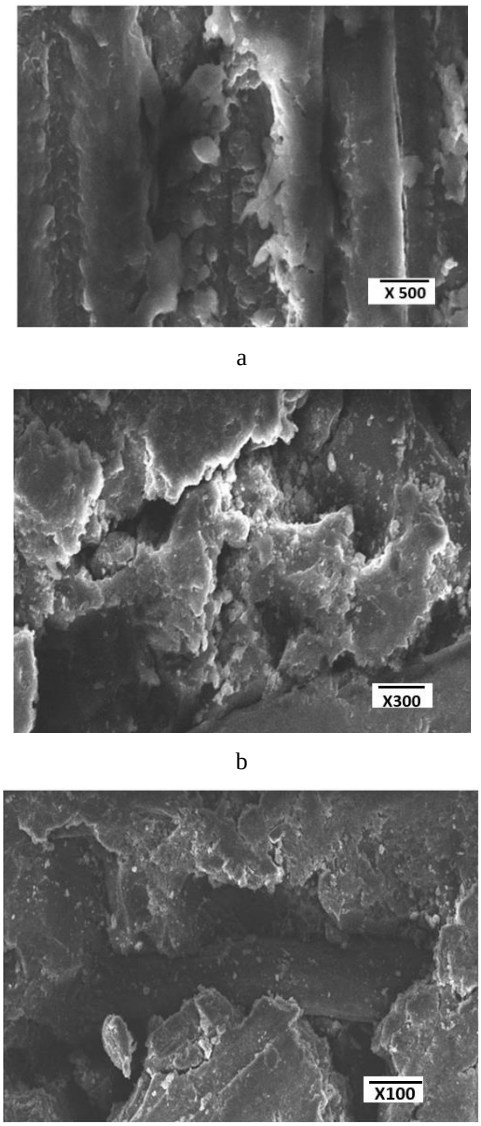


Fig. 4. SEM images of the tensile fracture surface of composite exposed to the seawater environment

Composites have a maximum flexural strength of 50.6 MPa, when the fibre content is 26.19 vol.% in wet conditions. In comparison to dry composite specimens, there was a 3.8 % loss in flexural strength value at 26.19 % volume.

3.2.3. Impact strength

The influence of fibre content and groundwater absorption on the impact strength of composites is demonstrated in Fig. 5 c. The same trends attained in the tensile and flexural strength values were also observed in the impact strength values at both the dry and wet conditions.

As found in the seawater environment, the impact strength of wet composites decreased as compared to dry composites. As shown in tensile and flexural strength, a linearly increasing tendency was also found in the impact strength up to 26.19 vol.% in wet conditions before dropping. Composites have a maximum impact strength of 1.30 kJ/m^2 when the fibre content is 26.19 vol.% in wet conditions. At wet conditions, the 40.39 vol.% composite had an impact strength of 1.27 kJ/m^2, which was 2.3 % lower than the 26.19 vol.% composite.

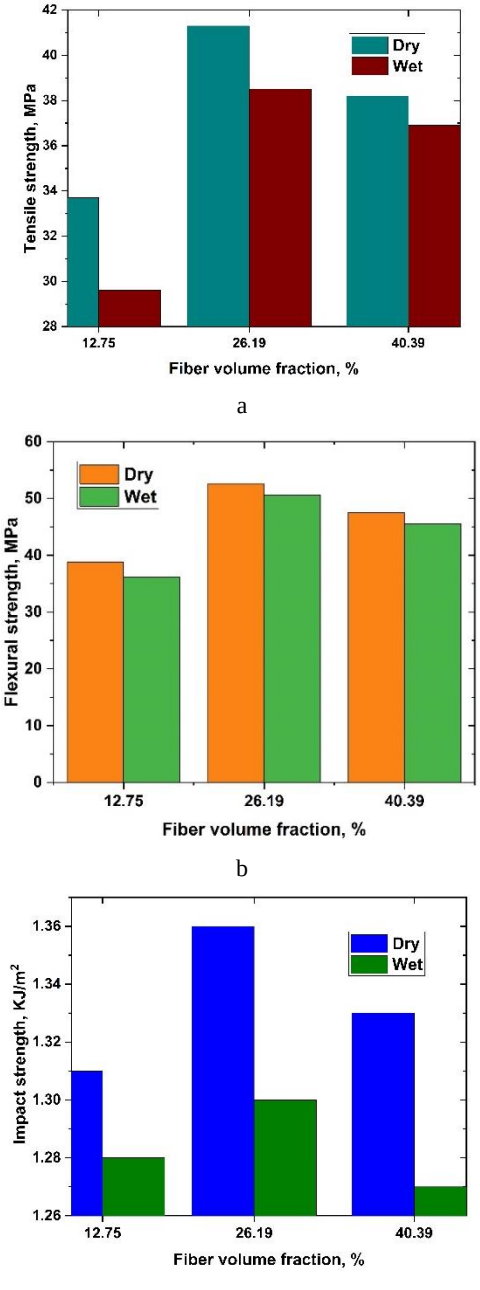
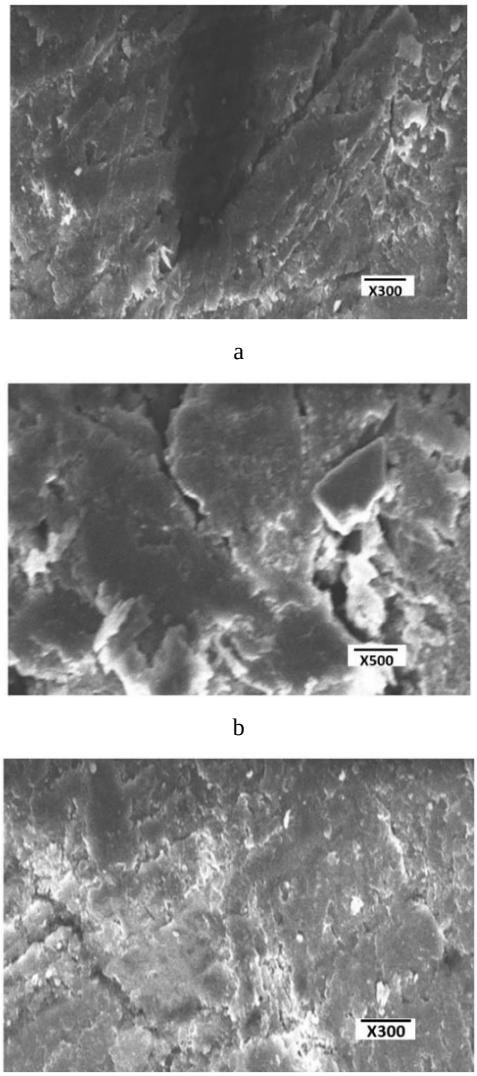


Fig. 5. Mechanical properties: a – tensile; b – flexural; c – impact of composite exposed to the ground water environment

Furthermore, the ranges of increase in tensile, flexural, and impact strength values in all fiber volume fractions were lower in ground water immersed composite specimens than in dry composite specimens. This could be because the immersion of composite specimens in groundwater impacted the bonding between the fiber-matrix interface and caused debonding, resulting in a reduction in the mechanical properties of the composites (Fig. 6 a).

Typically, when the composites are exposed to water environments, the drop in mechanical properties is more

significant for composites with a higher volume fraction of fibers compared to dry composite specimens.



a

b

c

Fig. 6. SEM images of the tensile fracture surface of composite exposed to the groundwater environment

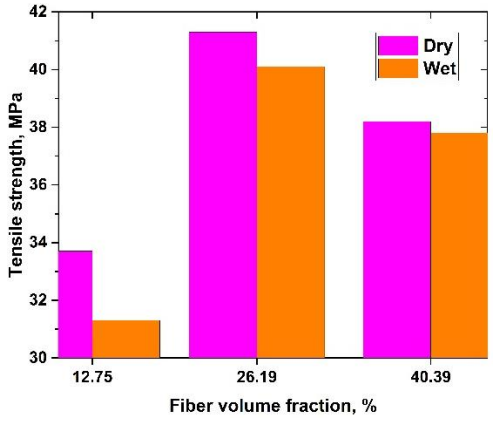
Cellulose fibers undergo swelling when they encounter moisture from the environment, allowing the fiber-matrix interface to be accessible. As a consequence, shear stress occurred at the interface between the fiber and matrix, causing the fibers to separate, the layers to detach, and the overall structural integrity to be damaged [26]. Consequently, the strength of the composite specimens suffered a decrease when exposed to immersion in a ground water environment (Fig. 6 b and c).

3.3. The effect of fiber content and distilled water exposure on the properties of composite

Fig. 7 presents the mechanical properties, such as tensile, flexural, and impact, of composites immersed in distilled water. The same relevant trends have been found regarding the effect of seawater and groundwater absorption on the mechanical properties of composites during distilled water absorption.

3.3.1. Tensile strength

Fig. 7 a presents the tensile strength values of composite specimens immersed in the distilled water environment. The tensile strength of a composite with a 12.75 vol.% fibre content was found to be 31.3 MPa at wet conditions. When compared to the dry composite specimen, their strength decreased by 7.1 %.



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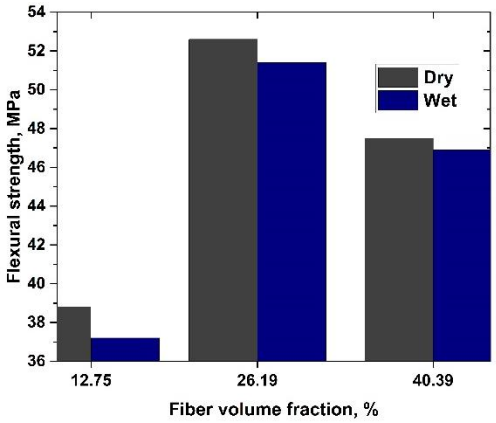

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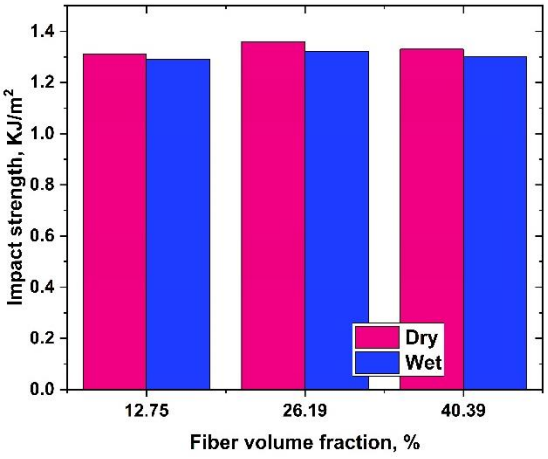

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Fig. 7. Mechanical properties: a – tensile; b – flexural; c – impact of composite exposed to the distilled water environment

This reduction in the tensile strength may be due to the changes in the strength of the fiber- matrix interaction. The variation in the fibre matrix interface could be the basic

cause of a decrease in the mechanical properties of water absorbed composite specimens. Furthermore, as water particles penetrated the macro-voids and free space of the matrix, additional cavities and cracks formed, serving as a water transport pathway within the composites and weakening interfacial bonding [27]. In the wet condition (distilled water environment), 26.19 vol.% of the composite provided the highest level of tensile strength. Beyond this level of volume fraction, the tensile strength values decrease. A composite with a fibre content of 40.39 % has tensile strength of 38.2 MPa at dry and 37.8 MPa under wet condition, respectively, resulting in 1.1 % of reduction.

3.3.2. Flexural strength

Fig. 7 b presents the flexural strength of composites immersed in the distilled water environment. The flexural strength of a composite with a 12.75 vol.% fibre content was found to be 37.2 MPa at wet conditions. When compared to the dry composite specimen, the strength was decreased by 4.1 %. The flexural strength reached a maximum value of 51.4 MPa at a volume fraction of 26.19 % at wet conditions. A decrease of 2.3 % in flexural strength was observed as compared to the composite at dry condition.

3.3.3. Impact strength

The effect of fibre content and distilled water absorption on the impact strength of composites is illustrated in Fig. 7 c. The impact strength of a composite with a 12.75 vol.% fibre content was found to be 1.29 kJ/m^2 at wet conditions. As identified in both the tensile and flexural strength values, the impact strength value increased up to 26.19, and then dropped. When compared to the 40.39 vol.%, an improvement of 1.6 % was achieved in 26.19 vol.% at wet conditions. Composite with 26.19 vol.% of fibers at wet condition shows an impact strength of 1.32 kJ/m^2, while that composite at dry condition shows an impact strength of 1.36 kJ/m^2. A reduction of 2.9 % was observed at the wet composite.

The void and crack forms on the surface of composite specimens were like those found in sea and ground waters (Fig. 8). However, unlike in the sea and ground waters, the surface and interface of the composite specimens exposed to distilled water were unaffected.

3.4. Comparison of effects of water immersion on mechanical properties of composites

The tensile, flexural, and impact strength values have consistently increased at a composite with 26.19 vol.% of fibre content in both dry and wet conditions, before decreasing. However, the strength values of composites significantly decreased under wet conditions compared to dry conditions, as shown in Fig. 9. Based on the results shown in Fig. 7, it was noted that the effects of exposing the composite to distilled water had little effect on its mechanical properties compared to other water environments. The composite specimens exposed to distilled water environments exhibited the least level of strength reduction compared to those exposed to sea and groundwater environments. Seawater exhibits a more significant influence than distilled or groundwater on the mechanical properties of composites due to the presence of chloride ions (NaCl, $MgCl_2$, and $CaCl_2$) in seawater. Seawater possesses a greater density than freshwater due to the presence of dissolved salts. The tensile, flexural, and impact properties of SPF/VE composites were typically affected by three different water exposures (sea, ground, and distilled).

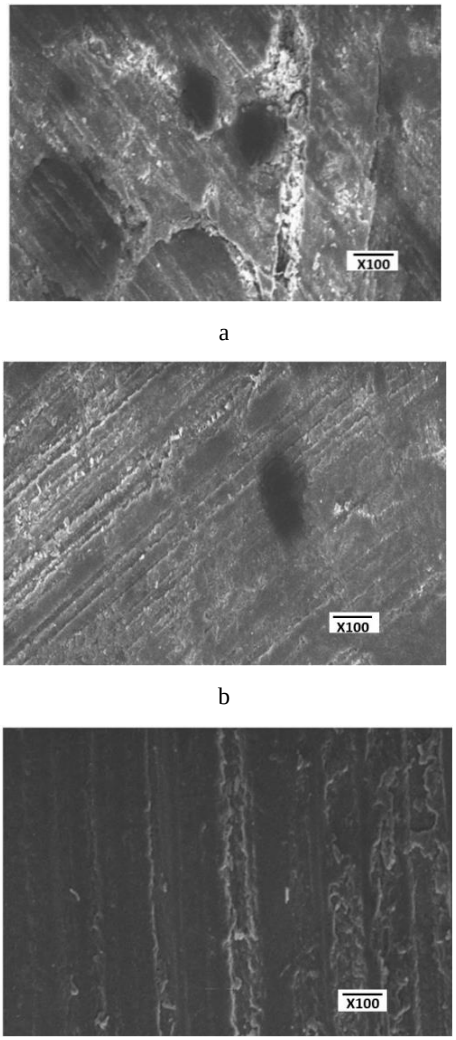


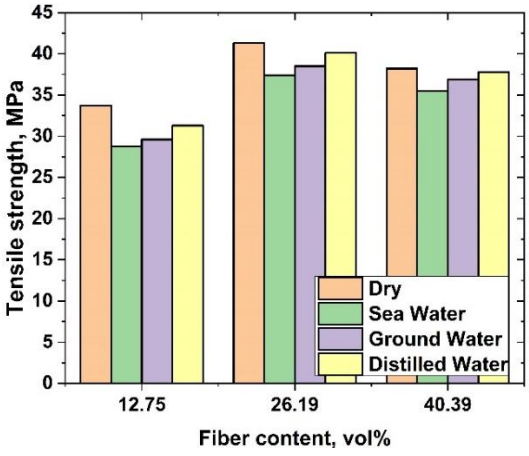
Fig. 8. SEM images of the tensile fracture surface of composite exposed to the distilled water

The wet composite specimens had much lower mechanical properties than the dry composite specimens. This could be attributed to water particles absorbed by the composite samples, which reduces intermolecular hydrogen bonding between cellulose molecules in the reinforcing agent while increasing intermolecular hydrogen bonding between water particles and cellulose molecules in the fibre. Furthermore, this action reduces the interfacial adhesion between the fibre and matrix, resulting in lower mechanical strength values.

4. CONCLUSIONS

The effects of fiber content and three different water exposures (seawater, groundwater, and distilled water) on the mechanical properties of SPF/VE composites were investigated. Mechanical properties such as tensile, flexural,

and impact strength were found to increase with increasing fiber content in both dry and wet conditions up to 26.1 vol.% before decreasing.



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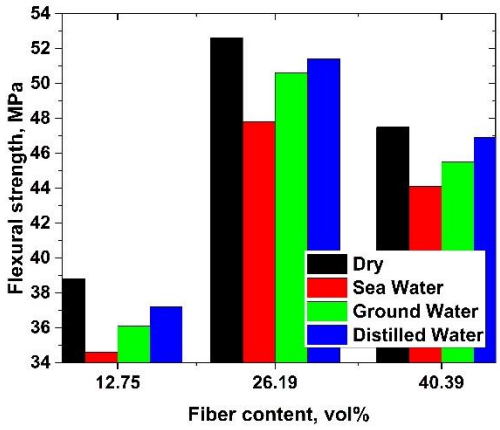

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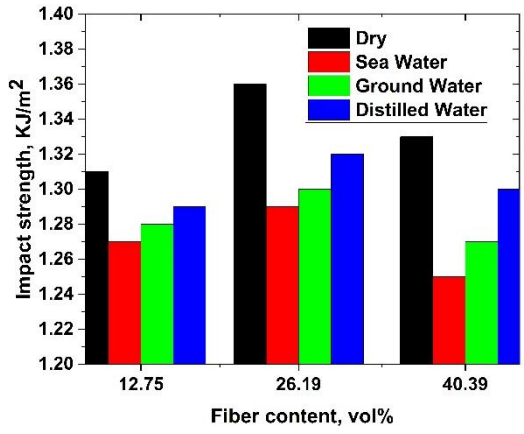

c

Fig. 9. Comparison of mechanical properties: a – tensile; b – flexural; c – impact of composite exposed to the sea, ground, and distilled water environments

Composite specimens with 40.39 vol.% showed lower level of mechanical properties compared to composite specimens with 26.19 vol.% at all water exposures.

The wet composite specimens had much lower mechanical properties than the dry composite specimens. Seawater exposure has a greater impact on the mechanical properties of SPF/VE composites than distilled or groundwater exposure because of the presence of chloride ions (NaCl, $MgCl_2$, and $CaCl_2$) in seawater. The SEM examination was carried out on the fractured surface of composite specimens at wet conditions and detected resin loss, void development, and microfractures on the surface of the wet composite specimens. Moreover, the properties (physical, thermal, and mechanical, etc.) and water absorption resistance of the natural fiber reinforced polymer composites can be improved by surface treatments (mercerization, acetylation, benzoylation, silane treatment, and peroxide treatment, etc.) of natural fiber using several chemical agents.

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