Effects of moisture absorption and thickness swelling behaviors on mechanical performances of *Carica papaya* fiber reinforced polymeric composites

A. Saravanakumaar[a,*], A. Senthilkumar[a], B. Muthu Chozha Rajan[a], Rajini Nagarajan[b,*], Sikiru O. Ismail[c], Faruq Mohammad[d], Hamad A. Al-Lohedan[d]

[a]Department of Mechanical Engineering, Sethu Institute of Technology, Virudhunagar-626115, Tamil Nadu, India

[b]Department of Mechanical Engineering, Kalasalingam Academy of Research and Education, Anand Nagar, Krishnankovil, Srivilliputhur

[c]Department of Engineering, School of Physics, Engineering and Computer Science, University of Hertfordshire, AL10 9AB, England, United Kingdom

[d]Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh, Kingdom of Saudi Arabia 11451

***Corresponding authors:**

A. Saravanakumaar, E-mail ID: asaravanakumaar1972@gmail.com, Tel. No: +91 962 953 9865

Rajini Nagarajan, Email ID: rajiniklu@gmail.com, Tel. No: +91 9942139392

Abstract

In this study, composite materials were made from *Carica papaya* fibers (CPFs), as a reinforcing element in polypropylene (PP), polyester (P) and epoxy (E) matrices, using compression molding technique. Experiments were conducted to evaluate the input parameters with their output responses, specifically density and thickness. Various CPF reinforced PP, P and E composite specimens with varied fiber orientations of 0^{o}, 45^{o} and 90^{o} as well as percentages of fiber contents of 10, 20 and 30 wt.% were prepared, according to the ASTM D 570 standard. From the results obtained, it was observed that CPF/E composites with fewer fraction of CPF and orientation of 90^{o} exhibited less water absorption throughout the whole duration of immersion. Water saturated CPF/E composite specimen, designated as E8, with orientation of 0^{o} and fiber content of 20 wt.% showed the highest tensile, flexural strengths and Shore D Hardness of 119, 115 MPa and 85, respectively. Also, CPF/E composite specimen (E7) with 90^{o} and 10 wt.% recorded the lowest tensile strength of 32 MPa, and CPF/E composite (E3) with 90º and 30 wt.% showed the lowest flexural strength of 41 MPa. Hence, it was evident that optimum CPF reinforced polymeric composite can be used for some outdoor engineering applications.

Keywords: Natural fiber composites; *Carica papaya* fibers (CPFs); Water uptake; Thickness swelling; Mechanical properties; Scanning electron microscopy.

Introduction

In the recent years, natural fiber composites have attracted several car manufacturers and suppliers of automobile accessories, such as door panels, seat backs, headliners, package trays, dashboards and interior parts (Espert, Vilaplana, and Karlsson 2004; Senthilkumar et al. 2018). Natural fibers offer benefits, including reductions in weight, cost, less dependence on fossil fuel sources and recyclability. Natural fibers are used as substitutes for artificial polymer composites, such as glass and carbon fibers based types. The availability of natural fibers and the simplicity of their processes make them products of an attractive feature (Atiqah 2020; Krishnasamy et al. 2019; Chandrasekar et al. 2020; Senthilkumar et al. 2020).

However, poor resistance to water absorption is the major disadvantage of natural fibers, especially during outdoor applications. Water absorption has undesirable effects on dimensional stability and mechanical properties of natural fiber reinforced polymer (FRP)composites (Ashori and Sheshmani 2010; Thiagamani et al. 2019; Senthilkumar, et al. 2018). Therefore, it is important to understand the water absorption behaviors of the natural FRP composites and also finding some ways to minimize the water absorption of natural fiber composites. When compared with recycled newspaper and poplar wood flour FRP composites, it was observed that recycled newspaper FRP composites recorded the lowest percentage water absorption and thickness swelling. This behavior was attributed to the low holocellulose and high lignin content (Akil et al. 2009).

Moving forward, a comparative study on jute FRP composites immersed in sea water, distilled water and acidic solutions has been conducted. It was reported that the exposure of composite materials to aqueous environment resulted to a significant drop in their strengths (Nacher et al. 2007). By comparing jute fibers reinforced epoxy and bio-epoxy, as matrices, the epoxy based composites recorded good results against water absorption. By analyzing the mechanical properties of polyester resin, it was evident that its internal structure with a high cross-linking rate has a smaller capacity to absorb water than a structure with a low cross-linking rate (Aridi et al. 2016). Rice husk reinforced polypropylene composites, after various liquid uptakes, showed weak filler-matrix interfacial adhesion, causing porosity and micro cracking (Narendar and Dasan 2013). Water absorption and thickness swelling of the treated hybrid composites were reduced, due to a better jute fiber-matrix interfacial bonding (Kommula et al. 2014). It is expected that fibers for reinforcement of composites must be subjected to water absorption with various liquids or chemicals, depending on their applications (Melkamu, Kahsay, and Tesfay 2019; Chow, Leu, and Mohd Ishak 2014; T Alomayri et al. 2014).

Furthermore, various experimental results confirmed that mechanical properties were severely affected by the moisture absorption of composite specimens. Bi-directional woven mats of jute, hemp and flax fibers reinforced epoxy composite specimens showed a maximum of 47% reduction in tensile strength. But, their hardness values were almost unaffected, after water absorption (Chaudhary, Bajpai, and Maheshwari 2018a). Scanning electron microscopy (SEM) images of Teak wood reinforced epoxy composite specimen depicted a poor fiber-matrix interfacial bonding. The weak adhesion increased with an increase in the teak wood fiber content, usually above 16% (Venkateshwaran, ElayaPerumal, and Jagatheeshwaran 2011).

The Shore D Hardness value of pineapple/high density poly ethylene (HDPE) composite decreased when the fiber content reached 25% (Singh Bahra, Gupta, and Aggarwal 2017). Mengkuang fiber content of 10% reinforced HDPE/natural rubber (NR) composite specimens recorded lowest water uptake and the highest tensile strain values (Abdullah and Che Aslan 2019). Cotton fabric reinforced geopolymer composites considerably exhibited improved fracture toughness, impact and flexural strengths. Reduction in mechanical properties was observed when the composite specimens were immersed in water for an extended period (Alomayri et al. 2014).

Therefore, it was evident that there is a need to study the effects of water absorption and thickness swelling behaviors on mechanical properties of natural FRP composites, specifically *Carica papaya* fiber (CPF) reinforced in various matrices, fiber orientations and contents, because they are scarcely or not available in the previous works. Hence, this investigation focuses on water absorption, thickness swelling behaviors and their effects on mechanical properties of CPF reinforced polypropylene (PP), polyester (P) and epoxy (E) composites with fiber orientations of 0^o, 45^o and 90^o as well as percentages of fiber contents of 20, 30 and 40 wt.%.

Materials and method

Materials

Carica papaya (CP) trees are abundantly available all over the world. The CPFs were extracted from the bark of CP plants/trees of average height of 10 m, each (Saravanakumaar et al. 2019). The CP plants are located in Madurai region, Tamilnadu, South India.

Extraction of fibers

Raw CP plants were extracted and the stem of the CP plant was cut into small pieces. The sliced stems were submerged in water for a maximum period of 4 weeks for microbial degradation (Saravanakumaar et al. 2019). The fibers were separated and thoroughly washed in fresh running water. Then, they were allowed to dry in the shadow of sun to allow maximum moisture removal.

Alkali treatment of fibers

The alkali treatment of CPFs, with 5% of sodium hydroxide (NaOH) and soaking time of 60 minutes showed a better cellulose degradation temperature and surface roughness when compared with raw CPF(Saravanakumaar et al. 2018). Therefore, the CPFs were soaked in beakers, containing 5% (w/v) concentrations of NaOH for 60 minutes at room temperature. The treated fibers were washed with deionized water to remove excess NaOH from their surfaces. The washed fibers were dried at room temperature for 60 hours. The treated CPFs were preserved properly and used as a reinforcement of the composite specimens, during fabrication stage.

Preparation of composites

The chemical compositions of optimally treated cellulosic CPFs are presented in Table 1. Typical mechanical properties of optimally treated CPF are similarly presented in Table 2. Three different thermoset resins (PP, P, and E) were used in this study. Various composite specimens were prepared with different combinations of resins, fiber weight percentages and fiber orientations. Table 3 presents formulations of the prepared combinations and their respective specimen designations. A total of 27 different types of combinations were prepared. The cutting edges were sealed, using matrix resin to prevent exposure of the fibers (Masoodi and Pillai 2012). Figs 1(a), (b) and (c) respectively show the prepared CPF reinforced composite specimens with 0^{o}, 45^{o} and 90^{o} fiber orientations for swelling behavior tests, according to the ASTM standard D570 – 98.

Figure 1. Prepared composite specimens with CPFs of (a) 0^o, (b) 45^o and (c) 90^o orientations for swelling behavior test in accordance with the ASTM standard D570 – 98.

Table 1. Chemical properties of alkali treated CPF (Saravanakumaar et al. 2018).

Table 2. Mechanical properties of alkali treated CPF (Saravanakumaar et al. 2020; Saravanakumaar et al. 2022).

Table 3. Comparison of the studied formulations.

Characterization

Swelling measurement

According to the ASTM D 570 standard (Standard 2010), the thickness swelling and water absorption tests were conducted. The weight and thickness of each specimen were measured before testing. For each type, four specimens were prepared and submerged in distilled water at room temperature. After a constant period of time, the specimens were withdrawn from the water and wiped/dried to remove the surface moisture. The thickness swelling was calculated, using Equation (1).The test continued for several days, until a constant weight of specimens at saturation point was obtained (Jawaid et al. 2013).

$$T_c = \frac{T_t - T_I}{T_I} \, x \, 100 \qquad (1)$$

Where T_c =thickness swelling percentage, T_t= thickness at time t and T_I = initial thickness.

Using Equation (2), the volume swelling was estimated.

$$V_c = \frac{V_t - V_I}{V_I} \, x \, 100 \qquad (2)$$

Where V_c = percentage of volume change, V_t = volume at time t and V_I = initial volume.

Water absorption measurement

Before starting the test, the exact weight and density of the dry specimen were measured. Similar to swelling measurements, the specimens were measured and recorded after they were wiped off by a dry wipe. The percentage of water absorption was estimated using Equation (3).

$$W_c = \frac{W_t - W_I}{W_I} \, x \, 100 \qquad (3)$$

Where W_c = percentage of weight change, W_t = weight at time t and W_I = initial weight. Using the percentages of weight and volume changes W_c and V_c respectively, the density of the composite was calculated. The density used for the comparison of the results obtained was estimated from Equation (4).

$$D_c = \frac{D_t - D_I}{D_I} \; x \; 100 \qquad (4)$$

Where D_c = percentage of density change, D_t = density at time t and D_I = initial density.

SEM analysis

SEM observations was conducted by using Tescan VEGA3 scanning electron microscope, with accelerated voltage of 20 kV and vacuum level of 1.5×10^{-3} Pafor better visualization of the affected surfaces of the CPFs. The specimens were coated with a 10 nm of gold layer for better conductivity of the specimen, using a beam sputter coater of 15 kV.

Mechanical properties

Tensile test

Tensile test were conducted in accordance with the ASTM D 3039 standard, using INSTRON – 5500R universal testing machine (UTM). Six specimens in each orientation with relative humidity of 65% were tested, using a cross-head speed of 2 mm/min for gauge length of 50 mm. A strain gauge was used to find the elongation. The elongation of the specimen during loading were recorded and analyzed.

Flexural test

Composite specimens for flexural test were prepared and tested according to the ASTM D790 standard. Three point flexural tests were similarly conducted on INSTRON – 5500R UTM, with a cross-head speed of 2.5 mm/min. Six specimens in each orientation were tested and the average values were obtained and analyzed.

Hardness test

Shore D Hardness tester was used to obtain the hardness values of the various specimens. The hardness values of the composite specimens were obtained after 100 days of water immersion. A force of 44.64N was applied by the indenter of the Shore D Hardness tester. The indentation was made by the steel rod indenter, with diameter of 1.1 to 1.4 mm and a conical point angle of 30º.

Results and discussion

Swelling analysis

Presence of polar groups in the lignocellulosic materials is the main reason for their poor absorption resistance (Salim, Asik, and Sarjadi 2021). Due to the water absorption, the fiber-matrix interface and the cell wall were wet and moisture built up, implying fiber swelling (Akinyemi and Omoniyi 2020). Fiber swelling in the composite specimen was responsible for the changes in the external appearance and dimensions, particularly in their thicknesses. Ramasubbu and Madasamy (2020) indicated that the water absorption initiated at the outer layer of the composite proceeded slowly into the bulk of the matrix. Similar to the water absorption, the swelling behavior of the CPFs increased with the fiber contents (Saw et al. 2014). The hydrophilic properties of lignocellulosic materials supported the capillary action to cuase the intake of water when the samples were soaked in water and thus increased the dimensions of composite. Figure 2 depicts the comparison of thickness swelling values as the *Carica papaya* fiber reinforced polypropylene (CPF/PP) composites, varied from 0 to100 days of immersion.

Figure 2. Comparison of thickness swelling values of different CPF/PP composite specimens.

Also, the thickness swelling of the CPF reinforced composites followed a trend similar to the water absorption. The swelling behavior increased with the immersion time until an equilibrium condition was attained. Saw et al. (2014) also confirmed that the water absorption of hybrid composites followed a trend similar to their thickness swelling behaviors, increasing with immersion time until an equilibrium condition was attained. Even after 80 days of immersion, the swelling behavior of high fiber content composite specimens PP3, PP9 and PP6 experience an increasing trend. Water penetrated easily into the composites, because of the poor interaction between the matrix and fiber (Sanjeevi et al. 2021). All the composites showed an increase in the thickness swelling with the incorporation of CPFs. The composite specimens PP1 and PP4 fabricated with fiber content of 10 wt.% recorded lower thickness swelling, and this result true for all the composite specimens. However, the composite specimen PP7 had a high thickness swelling, because of its fiber orientation (Dress, Woldemariam, and Redda 2021).

. The CPF orientation significantly influenced the water uptake and thickness swelling behaviors. Figs 3 and 4 show the thickness swelling behaviors of *Carica papaya* fiber reinforced polyester (CPF/P) and CPF/E composite specimens, respectively. As immersion time increased, water penetrated the inter phase of the fibers and loosed the cellular cellulose network structure of the CPFs, resulting to high thickness swelling (Thybring, Kymäläinen, and Rautkari 2018).

Figure 3. Comparison of thickness swelling values of different CPF/P composite specimens.

Figure 4. Comparison of thickness swelling values of different CPF/E composite specimens.

All the three categories of CPF reinforced polymeric composite specimens recorded same increasing trend of thickness swelling. After 100 days, the percentages of increase in thickness swelling were in the following order: P3(13.8),PP3(10.6),E3(10.2)(highest) > P5(13.4),PP5(10.4),E5(9.6) > P7(12.8),PP7(10.0),E7(9.4) > P9(12.7),PP9(9.1),E9(9.2) > P6(12.6),PP6(9.0),E6(9.1) > P2(12.5),PP2(8.9),E2(8.5) > P4(12.2),PP4(8.3),E4(8.4) > P8(11.8),PP8(8.2),E8(8.3) > P1(11.7),PP1(8.1),E1(8.2%)(lowest). Thus, it can be concluded that the water exposure time increased with the thickness swelling values of the composites. Composites fails disastrously, because of the stress on the surrounding matrix caused by the swelling of the fiber, leading to micro cracking (Alomayri et al. 2014). For outdoor applications, the dimensional stability of composites is more important and it would be affected when the fiber–matrix adhesion is weak.

Water absorption analysis

Water absorption rates of the nine CPF/PP composite specimens with different orientations and fiber fractions are shown in Figure 5. Firstly, Figure 5 shows evidently that an increase in immersion time increased the water absorption, until the equilibrium state was reached.

Figure 5. Comparison of water absorption values of CPF/PP composite specimens.

At initial days, the rapid uptake of water in all specimens indicated that the nature of cellulosic materials and the matrix content aided the composites to take up high quantity of water. Anbukarasi and Kalaiselvam (2015) confirmed that the epoxy matrix absorbed little water when more fibres were added to the matrix, causing an increased water absorption behavior of the composites, due to the presence of hydroxyl group in the fibres. The inner fibrillar space of the cellulosic structure retained water. Cracks and micro voids were developed on the surface of the composite specimen (Azeem et al. 2017). The capillary action of the fiber conducted the water molecules into the material. It later filled the cracks and micro voids on the surface of the composite specimen. Furthermore, the analysis of results in Figure 5 shows that the fiber content in the composite specimen increased with the water absorption rate.

In case of CPF composite specimens PP3, PP6 and PP9 with fiber content of 20 wt.% and PP2, PP5 and PP7 with fiber content of 30 wt.%, there was an increase in water absorption from 10 to 13%. These results supported the aforementioned points. This is predictable, because CPFs are hydrophilic in nature, therefore the high fiber content resulted to an increase in water absorption. When the fiber content was 10 wt.% in composite specimens PP1, PP4 and PP7, the water absorption rate was significantly reduced. materials. Venkatasudhahar, Ravichandran, and Dilipraja (2021) confirmed that the treated fiber performed well and improved the bonding between resin and fiber. However, it was observed that the water absorption rate significantly reduced with alkali treated fibers (Rajesh, Prasad, and Gupta 2018).

Figure 6. Comparison of water absorption values of CPF/P composite specimens.

Figure 7. Comparison of water absorption values of CPF/E composite specimens.

Moreover, Figs 6 and 7 show that the assessment of repeatability of the test results (Saravanakumaar et al. 2018). Three specimens were used for each test and the average test result was plotted. CPF/P and CPF/E composite samples with different fiber orientations and weights were tested for water absorption and thickness swelling. Comparing with both CPF/P and CPF/PP composite specimens, CPF/E specimen mostly restricted the water uptake and thickness swelling. The results showed that for the same fiber weight and orientation, different results were obtained for various resins in the following order P1 > PP1 > E1. It has been reported for E resin that there was water intake of about 0% (Khalil et al. 2007). It can be inferred that different Es that come in contact with water may not behave the same way and hence it is not prudent to attribute same water intake for different E resins. This can be supported by the results from the study conducted by Chaudhary, Bajpai, and Maheshwari (2018), it was reported that from all the developed composites, flax/epoxy composite recorded the lowest weight gain, due to moisture absorption at saturation level and relatively less fiber-matrix interface damage with epoxy.

Figure 8 compares 0^{o}, 45^{o} and 90^{o}orientations of the CPFs, it was observed that the 90^{o}orientation recorded the greatest water absorption than the other orientations. The rates of water absorption were in the following order: P3,PP3,E3 (highest) > P5,PP5,E5 > P7,PP7,E7 > P9,PP9,E9 > P6,PP6,E6 > P2,PP2,E2 > P4, PP4, E4 > P8,PP8,E8 > P1,PP1,E1 (lowest). By analyzing all the composites, it was observed that the weight gained when they were exposed to water increased with their fiber weights. This was due to the large number of tubular structures in the fiber, which speed up the penetration of water (Amran et al. 2020).

Water saturation analysis, using graphs obtained from all the three classes of composite specimens, is presented for further discussion (Figure 9). From the analysis, saturation levels for water absorption were identified for the CPF/P, CPF/PP and CPF/E specimens as 10, 7 and 6%, respectively. Very few specimens recorded variations in their water uptakes; but from minimum to average level of water absorption, CPF/E specimens exhibited the most minimum level of water saturation. Mass balance calculations for epoxy resins indicated that only 6–8% of the void volume was occupied by water at saturation (Abdelmola and Carlsson 2019). Plain epoxy resins exhibited water saturation of 6–8% (Abdelmola and Carlsson 2019). Similarly, this present experimental work established that CPF/E specimens saturated with an average water uptake of 6%. It was also evident that CPF specimens possessed reduced voids and hence there was a moderate water intake of the specimens.

Figure 8. Comparison of water absorption of different composite specimens with various CPF orientations.

Figure 9. Water saturation of the various CPF reinforced polymeric composite specimens.

SEM analysis

Figure10 shows the SEM analysis of the moisture absorbed composite specimens. From Figure 10(a), the water-absorbed matrix layer was evidently visible and it confirmed that the maximum amount of water was absorbed within the first 10 days of immersion. Molecules present inside the water absorbed at surfaces of the composite specimen were visible in Figs. 10(b) and (c). Figure 10(d) depicts the surface decomposition of the composite specimen, because of the immersion of specimen in water for a long period of 100 days

Figure 10. SEM micrographs of the moisture absorbed specimens, depicting (a) water absorbed matrix, (b) water absorption and surface swelling, (c) voids and (d) decomposition after 100 days.

Mechanical properties

Mechanical properties of all the three classes of composites specimens were obtained after tests and their average values were presented for further discussion (Figure 11). The comparative analysis on mechanical properties, including tensile, flexural strengths and Shore D Hardness of different combinations of the composite specimens is subsequently and respectively elucidated.

Figure 11. Mechanical properties of the various CPF reinforced polymeric composite specimens.

Tensile strengths

The tensile strengths of the composite specimens are shown in Figure 12. It was observed that the water immersed CPF/E composite specimens E3 and E8 recorded minimum and maximum tensile strengths of 30 and 119.6MPa, respectively (Figure 12). Binu Kumar et al. (2020) confirmed that the moisture uptake behavior of composite reduced its tensile strength, due to the breakage of H-bond. From the comparison, it was further observed that the fiber orientation and the fiber weight percentage highly influenced the tensile strengths of the composite specimens (Dress, Woldemariam, and Redda 2021). The same trend of effect was observed with CPF/P and CPF/PP composite specimens. The CPF/P composite specimens P4 and P6 recorded minimum and maximum values of 42.00 and 103.13MPa, respectively. Also, CPF/PP composite specimens PP1 and PP6 recorded minimum and maximum values of 50 and 60 MPa, respectively. From the previous details, the CPF/E composite specimens exhibited the highest tensile strength when compared with the other two composite counterparts because of the strong adhesion in between matrix and the fiber. Ramasubbu and Madasamy (2020) also confirmed that the maximum values of tensile strength of kenaf fiber composite, due to strong adhesion between fibers and epoxy matrix, can be attributed to the uniform transfer of stress from matrix to the fibers. The maximum tensile strength achieved by the composites can be expressed in the following order: 119.60 (E8) > 103.13 (P6) > 60.00MPa (PP6). All the composite specimens with fiber orientation of 0^o recorded the highest tensile strengths. But, when different matrices were used, the results were different. The lowest tensile strengths achieved by the composites follow this order: 50 (PP1) > 42 (P4) > 30MPa (E3). From this trend, it can be established that the fiber orientation influenced their tensile strength more than the fiber weight.

Figure 12. Comparison of tensile strengths of the various CPF reinforced polymeric composite specimens.

Flexural strengths

Figure 13 comparatively shows the flexural strengths of all the CPF reinforced polymer composite specimens. The maximum flexural strength of 115.8 MPa was recorded by the CPF/E composite E8 with fiber orientation of 0^o and content of 20 wt.%. The minimum flexural strength of 41.6 MPa was obtained from the CPF/E composite specimen E3 with fiber orientation of 90^o and content of 30 wt.%. Velusamy et al. (2019) reported that the flexural modulus strongly depends on both fiber length and volume fraction. CPF/P composites recorded the minimum and maximum flexural strengths of 43 and 100 MPa, respectively. The CPF/PP composite specimens with fiber attributes of 90^o/10 wt.% and 0^o/30 wt.% achieved lower and higher flexural strengths of 63 and 73MPa, respectively. Higher values of flexural strengths with their matrices were compared with each other, it showed that the CPF/E composites with fiber orientation of 0^o recorded the highest value and their results are in the following order: 115.8 (E8) > 100.0 (P1) > 73.0 MPa (PP6). Similar to the tensile strength values, the lower value of flexural strength followed the reverse of the higher values of flexural strength. The lower values of flexural strength results are in this order: 63.0 (PP7) > 43.0 (P3) > 41.6 MPa (E3). Therefore, it was evident that both fiber orientation and weight fraction had greater influence on the flexural strengths of the CPF composites (Cordin, Bechtold, and Pham 2018). This was in a close agreement with the similar report on sugar palm fiber (Binu Kumar et al. 2020).

Figure 13. Comparison of flexural strengths of the various CPF reinforced /polymeric composite specimens.

Shore D Hardness analysis

After analyzing the recorded results of Shore D Hardness (Figure 14), it was observed that their variation was very little among the same polymer matrix specimens. From all the composites, water saturated CPF/E composite specimens produced the highest Shore D Hardness value of 85.6, followed by the CPF/P composite specimens of 85.1. The CPF/PP composite specimen recorded the highest value of 84.7. The highest Shore D values of the three CPF reinforced polymer matrices ranged from 84.7 to 85.6.

This variation range was very small when compared among themselves. The reason for the low variation can be traced to the hydrophobic nature of the polymers; they repel water. Even though, natural fibers, including CPFs have tendency of absorbing water, but the CPF laminated with PP, P and E types of polymers produced very small difference in their hardness values. The lowest values of their hardness values were 74.4, 80.4 and 80.5 for CPF/P, CPF/PP and CPF/E composite specimens, respectively. Chaudhary, Bajpai, and Maheshwari (2018) concluded that the interfacial adhesion between fiber and matrix, percentage of fiber loading, treatment of fiber-matrix and hybridization determine the value of hardness (shore D) of composite material. The reason behind the marginal difference was the softening nature of the polymer layers outside the CPF composites (Yan, Kasal, and Huang 2016).

Figure 14. Comparison of hardness values of the various CPF reinforced polymeric composite specimens.

Analysis of mechanical properties

CPF/E composites

Composite specimens, after a long time of immersion in water exhibited some molecular change in structure of hydrogen bonds (H-bonds). The H-bonds were formed between CPFs and E matrix at higher or macromolecular level. This formation of bonds weakened the bonding between the CPF-E interface and therefore, the tensile strength of the CPF/E composite specimen reduced (Ghani et al. 2012). The tensile, flexural and the Shore D Hardness values of the CPF/E composite samples are presented in Table 4. From the results obtained, it was observed that CPF/E composite specimens with fiber orientation of 0^orecorded the highest values of mechanical properties (tensile, flexural and hardness). When considering the fiber weight percentage, the 20wt.% fiber composite exhibited the highest values of mechanical properties, among all the weight percentages. Perinbakannan, Karuppusamy, and Ramar (2021) reported that the layer sequence has a significant contribution to the performance of composites. The lowest value was achieved with the CPF/E composite with higher fiber weight fraction and orientation of 90^o. It was significantly evident that the fiber extracted along the water uptake direction has a very poor fiber-matrix bonding in the CPF/E composite system and lower values in all the types of mechanical tests considered (Alhuthali and Low 2015).

Table 4. Mechanical properties of the CPF/E composite specimens.

CPF/P composites

The mechanical properties, such as tensile, flexural and the Shore D Hardness of the CPF/P composite specimens are presented in Table 5. By comparing the tensile strength values obtained from the CPF/P composite, the highest value of 103 MPa was achieved with specimen P6 with fiber orientation and weight of 0^o and 30 wt.%. The lowest value was 42 MPa, as recorded by the CPF/P composite specimen P4with fiber orientation and weight of 90^o and 30 wt.%, respectively. CPF/P composite specimen P1 with fiber orientation and weight of 0^o and 10 wt.% recorded flexural strength of 100 MPa. The lowest flexural value of 43 MPa was recorded by the CPF/P composite specimen P3 with fiber orientation and weight of 90^o and 30 wt.%, respectively. Ravikumar et al. (2021) stated that reinforcing jute fiber in polyester matrix has less significant enhancement on its flexural property.

Comparing the hardness values of the composite specimens, there was insignificant difference. The lowest and highest Shore D Hardness values were 74 and 85, respectively. The hardness values were not only depended on the fiber weight fraction, but also on the orientation (Karsli and Aytac 2013).

Table 5. Mechanical properties of the CPF/P composite specimens.

CPF/PP composites

The tensile, flexural and Shore D Hardness values of CPF/PP composites are presented in Table 6. After analyzing the results obtained (Table 6), it was observed that the CPF/PP composites specimens exhibited the lowest tensile strengths when compared with the CPF/E and CPF/P composite counterparts. This can be traced to the quality of the CPF/PP composite. The order of the highest values in their tensile strengths was E>P>PP. The CPF/PP composites recorded lowest and highest flexural strength of 63 and 73 MPa, respectively.

Table 6. Mechanical properties of the CPF/PP composite specimens.

All the CPF/PP composite specimens recorded moderate difference in their flexural strengths, because of the quality and degree of ability of CPFs to withstand the applied flexural load or force. The hardness value mostly depended on the surface of the polymer. Hence, moderate difference in the Shore D Hardness values of all the specimens was observed, because their surfaces were same for all the specimens. Also, the small difference in their hardness values can be attributed to the softening of the polymer layer (Saharudin, Atif, and Inam 2017).

Conclusions

Twenty seven different sets of CPF reinforced PP, P and E composite specimens with fiber orientation of 0^o, $45^o$and 90^o as well as percentages of fiber weights of 10, 20 and 30 wt.% have been fabricated, using compression molding technique. The effects of water absorption on the swelling behaviors and more importantly mechanical properties of the various specimens were analyzed. Therefore, the following conclusions can be drawn from this study:

- Initially, all the composite specimens showed high rates of water uptake, but their rates of swelling decreased gradually with time. Also, CPF/E composite specimens exhibited lower water absorption rate and thickness swelling than the CPF/PP and CPF/P counterparts.It was observed that their rates of water absorption and thickness swelling were in this order: CPF/E<CPF/PP<CPF/P.

- Also, adding more CPFs to the composite specimen resulted to higher water absorption and higher rate of swelling. Composite specimens with fiber contents of 20 and 30 wt.% recorded water absorption rates of 10 and 13%, respectively. These depended on the capillary action of the fiber, which conducted water molecules into the composite system. Also, the nature of cellulosic CPFs and matrix content in the composites were responsible for their high quantity of water absorption.
- Comparing different CPF orientations of 0^{o}, $45^{o}$and 90^{o}, it was observed that composite specimen with orientation of 90^{o} recorded highest water absorption when compared with other orientations.This can be attributed to the natural water absorbing system of the *Carica papaya* plant, which is from bottom to top and considered to be orientation of 90^{o}.
- From the analysis of water saturation level, CPF/E composite specimens recorded lower percentage when compared with other polymeric counterparts.
- Analysis of their tensile and flexural strengths depicted that CPF orientation influenced their water absorption rates more than the fiber fractions or contents.
- The Shore D Hardness values of all the CPF reinforced polymer composite specimens varied within a very small range.
- From the composite morphology analysis, it was significantly evident that the fiber fracture and internal cracks on their surfaces were the major responsible damage responses and factors for the swelling behaviors of the CPF reinforced composites.
- This investigation provided a great support to the environmental impact and sturdiness of CPF/E composites. It can be customized by fiber fraction and orientation of 10 wt% and 0^{o} respectively, as it exhibited less water absorption and swelling throughout the whole duration of immersion.

Summarily, this study has evidently presented optimal 0^{o} orientation and 20 wt.% CPF/E composite specimen E8 with highest Shore D Hardness, tensile and flexural strengths as a competitive bio-composite material for various open air uses.

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References

Abdelmola, F. A., and Carlsson, L. A. 2019. Water uptake in epoxy matrix with voids: Experiments and modeling. *Journal of Composite materials* 53:1049-65.

Abdullah, M.Z., and N.H. Che Aslan. 2019. Performance evaluation of composite from recycled polypropylene reinforced with mengkuang leaf fiber. *Resources* 8:1-8.

Akil, H.M., L.W. Cheng, Z.A.M. Ishak, A.A. Bakar, and M.A.A. Rahman. 2009. Water absorption study on pultruded jute fibre reinforced unsaturated polyester composites. *Composites Science and Technology* 69:1942–8.

Akinyemi, B. A., and Omoniyi, T. E. 2020. Effect of experimental wet and dry cycles on bamboo fibre reinforced acrylic polymer modified cement composites. *Journal of the Mechanical Behavior of Materials* 291:86-93.

Alhuthali, A. M., and Low, I. M. 2015. Effect of prolonged water absorption on mechanical properties in cellulose fiber reinforced vinyl-ester composites. *Polymer Engineering and Science* 5512:2685-97.

Alomayri, T, H. Assaedi, F. U. A. Shaikh, and I. M. Low. 2014. Effect of water absorption on the mechanical properties of cotton fabric-reinforced geopolymer composites. *Journal of Asian Ceramic Societies* 2:223–30.

Alomayri, T., L. Vickers, F. U. A. Shaikh, and I.-M. Low. 2014. Mechanical properties of cotton fabric reinforced geopolymer composites at 200–1000 C. *Journal of Advanced Ceramics* 3:184–93.

Amran, Y. M., Alyousef, R., Alabduljabbar, H., Alaskar, A., and Alrshoudi, F. 2020. Properties and water penetration of structural concrete wrapped with CFRP. *Results in Engineering* 5:100094.

Anbukarasi, K., and Kalaiselvam, S. 2015. Study of effect of fibre volume and dimension on mechanical, thermal, and water absorption behaviour of luffa reinforced epoxy composites. *Materials and Design* 66:321-30.

Aridi, N. A. M., S. M. Sapuan, E. S. Zainudin, and F. M. AL-Oqla. 2016. Investigating morphological and performance deterioration of injection-molded rice husk–polypropylene composites due to various liquid uptakes. *International Journal of Polymer Analysis and Characterization* 21:675–85.

Ashori, A., and S. Sheshmani. 2010. Hybrid composites made from recycled materials: moisture absorption and thickness swelling behavior. *Bioresource Technology* 101:4717-20.

Atiqah, A. 2020. Characterization and Interface of Natural and Synthetic Hybrid Composites. Encyclopedia of Renewable and Sustainable Materials. Vol. 4. Elsevier Ltd. https://doi.org/10.1016/B978-0-12-803581-8.10805-7.

Azeem, M., Boughattas, A., Wiener, J., and Havelka, A. 2017. Mechanism of liquid water transport in fabrics: A review. *Fibres and Textiles*, 244:58-65.

Binu Kumar, V. J., Bensam Raj, J., Karuppasamy, R., and Thanigaivelan, R. 2020. Influence of chemical treatment and moisture absorption on tensile behavior of neem/banana fibers reinforced hybrid composites: an experimental investigation. *Journal of Natural Fibers* 1-12.

Chandrasekar, M., I. Siva, T .S. M. Kumar, K. Senthilkumar, S. Siengchin, and N. Rajini. 2020. Influence of fibre inter-ply orientation on the mechanical and free vibration properties of banana fibre reinforced polyester composite laminates. *Journal of Polymers and the Environment* 1–12.

Chaudhary, V., P. K. Bajpai, and , S. Maheshwari. 2018a. Effect of moisture absorption on the mechanical performance of natural fiber reinforced woven hybrid bio-composites. *Journal of Natural Fibers* 17:84-100.

Chaudhary, V., P. K. Bajpai, and S. Maheshwari. 2018b. An investigation on wear and dynamic mechanical behavior of jute/hemp/flax reinforced composites and its hybrids for tribological applications. *Fibers and Polymers* 19:403–15.

Chow, W. S., Y. Y. Leu, and Z. A. Mohd Ishak. 2014. Water absorption of poly lactic acid nanocomposites: Effects of nanofillers and maleated rubbers. *Polymer-Plastics Technology and Engineering* 53:858–63.

Cordin, M., Bechtold, T., and Pham, T. 2018. Effect of fibre orientation on the mechanical properties of polypropylene–lyocell composites. *Cellulose 25*12:7197-210.

Dress, G. A., Woldemariam, M. H., and Redda, D. T. 2021. Influence of fiber orientation on impact resistance behavior of woven sisal fiber reinforced polyester composite. *Advances in Materials Science and Engineering* 2021:1-11.

Espert, A., F. Vilaplana, and S. Karlsson. 2004. Comparison of water absorption in natural cellulosic fibres from wood and one-year crops in polypropylene composites and its influence on their mechanical properties. *Composites Part A: Applied Science and Manufacturing* 35:1267–76.

Ghani, M. A. A., Z. Salleh, K. M. Hyie, M. N. Berhan, Y. M. D. Taib, and M. A. I. Bakri. 2012. Mechanical properties of kenaf/fiberglass polyester hybrid composite. *Procedia Engineering* 41:1654–9.

Jawaid, M., O. Y. Alothman, M. T. Paridah, and H. P. S. Abdul Khalil. 2013. Effect of fiber treatment on dimensional stability and chemical resistance properties of hybrid composites. *International Journal of Polymer Analysis and Characterization* 18: 608–16.

Karsli, N. G., and Aytac, A. 2013. Tensile and thermomechanical properties of short carbon fiber reinforced polyamide 6 composites. *Composites Part B: Engineering* 51:270-5.

Khalil, H. P. S. A., A. M. Issam, M. T. A. Shakri, R. Suriani, and A. Y. Awang. 2007. Conventional agro-composites from chemically modified fibres. *Industrial Crops and Products* 26:315–23.

Kommula, V. P., K. O. Reddy, M. Shukla, T. Marwala, and A. V. Rajulu. 2014. Mechanical properties, water absorption, and chemical resistance of napier grass fiber strand–reinforced epoxy resin composites. *International Journal of Polymer Analysis and Characterization* 19:693–708.

Krishnasamy, S., S. M. K. Thiagamani, C. Muthukumar, J. Tengsuthiwat, R. Nagarajan, S. Siengchin, and S. O. Ismail. 2019. Effects of stacking sequences on static, dynamic mechanical and thermal properties of completely biodegradable green epoxy hybrid composites. *Materials Research Express* 6:105351.

Kumaar, A. S., Senthilkumar, A., Saravanakumar, S. S., Senthamaraikannan, P., Loganathan, L., and Muthu Chozha Rajan, B. 2020. Mechanical properties of alkali-treated carica papaya fiber-reinforced epoxy composites. *Journal of Natural Fibers* 1-11.

Masoodi, R., and K. M. Pillai. 2012. A study on moisture absorption and swelling in bio-based jute-epoxy composites. *Journal of Reinforced Plastics and Composites* 31:285–94.

Melkamu, A., M. B. Kahsay, and A. G. Tesfay. 2019. Mechanical and water-absorption properties of sisal fiber agave sisalana-reinforced polyester composite. *Journal of Natural Fibers* 16:877–85.

Nacher, L. S., J. E. C. Amoros, M. D. S. Moya, and J. L. Martinez. 2007. Mechanical properties of polyester resins in saline water environments. *International Journal of Polymer Analysis and Characterization* 12:373–90.

Narendar, R., and K. P. Dasan. 2013. Effect of chemical treatment on the mechanical and water absorption properties of coir pith/nylon/epoxy sandwich composites. *International Journal of Polymer Analysis and Characterization* 18:369–76.

Perinbakannan, A. S., Karuppusamy, M., and Ramar, K. 2021. Mechanical and water transport characterization of indian almond – banana fibers reinforced hybrid composites for structural applications. *Journal of Natural Fibers* 1-11.

Rajesh, G., Prasad, A. R., and Gupta, A. V. S. S. K. S. 2018. Water absorption characteristics of successive alkali treated jute/polylactic acid composites. *Materials Today: Proceedings*, 511:24414-21.

Ramasubbu, R., and Madasamy, S. 2020. Fabrication of automobile component using hybrid natural fiber reinforced polymer composite. *Journal of Natural Fibers* 1-11.

RaviKumar, P., Rajeshkumar, G., Prakash Maran, J., Al-Dhabi, N. A., and Karuppiah, P. 2021. Evaluation of mechanical and water absorption behaviors of jute/carbon fiber reinforced polyester hybrid composites. *Journal of Natural Fibers* 1-13.

Saharudin, M. S., Atif, R., and Inam, F. 2017. Effect of short-term water exposure on the mechanical properties of halloysite nanotube-multi layer graphene reinforced polyester nanocomposites. *Polymers* 9:1-18.

Salim, R. M., Asik, J., and Sarjadi, M. S. 2021. Chemical functional groups of extractives, cellulose and lignin extracted from native Leucaena leucocephala bark. *Wood Science and Technology* 552:295-313.

Sanjeevi, S., Shanmugam, V., Kumar, S., Ganesan, V., Sas, G., Johnson, D. J., Shanmugam, M., Ayyanar, A., Naresh, K., Neisiany, R. N., and Das, O. 2021. Effects of water absorption on the mechanical properties of hybrid natural fibre/phenol formaldehyde composites. *Scientific Reports* 111:1-11.

Saravanakumaar. A., A. Senthilkumar, T. Sornakumar, S. S. Saravanakumar, and V.P. Arthanariesewaran. 2019. Physicochemical properties of new cellulosic fiber extracted from carica papaya bark. *Journal of Natural Fibers* 16:175–84.

Saravanakumaar, A., A. Senthilkumar, S.S. Saravanakumar, M. R. Sanjay, and A. Khan. 2018. Impact of alkali treatment on physico-chemical, thermal, structural and tensile properties of *Carica papaya* bark fibers. *International Journal of Polymer Analysis and Characterization* 23:529–36.

Saw, S. K., Akhtar, K., Yadav, N., and Singh, A. K. 2014. Hybrid composites made from jute/coir fibers: Water absorption, thickness swelling, density, morphology, and mechanical properties. *Journal of Natural Fibers* 111:39-53.

Senthilkumar, K., I. Siva, N. Rajini, J. T. W. Jappes, and S. Siengchin. 2018. Mechanical characteristics of tri-layer eco-friendly polymer composites for interior parts of aerospace application. *Sustainable Composites for Aerospace Applications*, 35–53. Elsevier.

Senthilkumar, K., N. Saba, N. Rajini, M. Chandrasekar, M. Jawaid, S. Siengchin, and O.Y. Alotman. 2018. Mechanical properties evaluation of sisal fibre reinforced polymer composites: A review. *Construction and Building Materials* 174:713-29.

Senthilkumar, K., T. Ungtrakul, M. Chandrasekar, T. S. M. Kumar, N. Rajini, S. Siengchin, H. Pulikkalparambil, J. Parameswaranpillai, and N. Ayrilmis. 2020. Performance of sisal/hemp bio-based epoxy composites under accelerated weathering. *Journal of Polymers and the Environment* 1–13.

Singh Bahra, M., V. K. Gupta, and L. Aggarwal. 2017. Effect of fibre content on mechanical properties and water absorption behaviour of pineapple/HDPE composite. *Materials Today: Proceedings* 4:3207–14.

Standard, A. D570, 1998. 2010. "Test Method for Water Absorption of Plastics," ASTM International, West Conshohocken, PA, 2010, DOI: 10.1520/D0570-98R10E01.

Thiagamani, S. M. K., S. Krishnasamy, C. Muthukumar, J. Tengsuthiwat, R. Nagarajan, S. Siengchin, and S. O. Ismail. 2019. Investigation into mechanical, absorption and swelling behaviour of hemp/sisal fibre reinforced bioepoxy hybrid composites: Effects of stacking sequences. *International Journal of Biological Macromolecules* 140:637–46.

Thybring, E. E., Kymäläinen, M., and Rautkari, L. 2018. Experimental techniques for characterising water in wood covering the range from dry to fully water-saturated. *Wood Science and Technology* 522:297-329.

Velusamy, K., Navaneethakrishnan, P., Rajeshkumar, G., and Sathishkumar, T. P. 2019. The influence of fiber content and length on mechanical and water absorption properties of Calotropis Gigantea fiber reinforced epoxy composites. *Journal of Industrial Textiles* 488:1274-90.

Venkatasudhahar, M., Ravichandran, A. T., and Dilipraja, N. 2021. Effect of stacking sequence on mechanical and moisture absorption properties of abaca-kenaf-carbon fiber reinforced hybrid composites. *Journal of Natural Fibers* 1-12.

Venkateshwaran, N., A. ElayaPerumal, and M.S. Jagatheeshwaran. 2011. Effect of fiber length and fiber content on mechanical properties of banana fiber/epoxy composite. *Journal of Reinforced Plastics and Composites* 30:1621–7.

Yan, L., Kasal, B., and Huang, L. 2016. A review of recent research on the use of cellulosic fibres, their fibre fabric reinforced cementitious, geo-polymer and polymer composites in civil engineering. *Composites Part B: Engineering* 92:94-132.

Table 1. Chemical properties of alkali treated CPF (Saravanakumaar et al. 2018).

Name	Cellulose (wt.%)	Hemi cellulose (wt.%)	Lignin (wt.%)	Wax (wt.%)	Moisture content (%)	Density (Kg/m^3)	Ash (%)
Alkali treated CPF	69.47	3.46	9.74	0.36	5.87	967	2.65

Table 2. Mechanical properties of alkali treated CPF ((Saravanakumaar et al. 2020; Saravanakumaar et al. 2022.

Parameter	Alkali treated CPF
Tensile strength (MPa)	548±14.600
Young's modulus (GPa)	12.64±1.532
Elongation at break (%)	1.83 ± 0.040

Table 3. Comparison of the studied formulations.

Resin with specimen code	Fiber weight (wt.%)	Fiber orientation (°)
PP1, P1, E1	10	0
PP2, P2, E2	20	45
PP3, P3, E3	30	90
PP4, P4, E4	10	45
PP5, P5, E5	20	90
PP6, P6, E6	30	0
PP7, P7, E7	10	90
PP8, P8, E8	20	0
PP9, P9, E9	30	45

Note: PP, P and E represent polypropylene, polyester and epoxy, respectively.

Table 4. Mechanical properties of the CPF/E composite specimens.

Resin with specimen code	Fiber weight (wt.%)	Fiber orientation (°)	Tensile strength (MPa)	Flexural strength (MPa)	Hardness (Shore D)
E1	10	0	100.3	105.7	81
E2	20	45	95.2	91.3	80
E3	30	90	30.0	41.6	79
E4	10	45	81.0	88.2	79
E5	20	90	46.3	57.6	80
E6	30	0	84.2	102.3	79
E7	10	90	32.4	51.7	82
E8	20	0	119.6	115.8	85
E9	30	45	71.6	84.3	78

Table 5. Mechanical properties of the CPF/P composite specimens.

Resin with specimen code	Fiber weight (wt.%)	Fiber orientation (°)	Tensile strength (MPa)	Flexural strength (MPa)	Hardness (Shore D)
P1	10	0	72	100	79
P2	20	45	63	94.6	81
P3	30	90	60	43	78
P4	10	45	42	91	76
P5	20	90	58	61	78
P6	30	0	103.13	80	83
P7	10	90	74	54	74

P8	20	0	86	90	85
P9	30	45	84	86	81

Table 6. Mechanical properties of the CPF/PP composite specimens.

Resin with specimen code	Fiber weight (wt.%)	Fiber orientation (^{o})	Tensile strength (MPa)	Flexural strength (MPa)	Hardness (Shore D)
PP1	10	0	50	67	83
PP2	20	45	56	69	84
PP3	30	90	54	68	82
PP4	10	45	54	67	81
PP5	20	90	50	65	83
PP6	30	0	60	73	85

PP7	10	90	48	63	80
PP8	20	0	55	70	84
PP9	30	45	50	68	82

Figure 1. Prepared composite specimens with CPFs of (a) 0^o, (b) 45^o and (c) 90^o orientations for swelling behavior test in accordance with the ASTM standard D570 – 98.

Figure 2. Comparison of thickness swelling values of different CPF/PP composite specimens.

Figure 3. Comparison of thickness swelling values of different CPF/P composite specimens.

Figure 4. Comparison of thickness swelling values of different CPF/E composite specimens.

Figure 5. Comparison of water absorption values of CPF/PP composite specimens.

Figure 6. Comparison of water absorption values of CPF/P composite specimens.

Figure 7. Comparison of water absorption values of CPF/E composite specimens.

Figure 8. Comparison of water absorption of different composite specimens with various CPF orientations.

Figure 9. Water saturation of the various CPF reinforced polymeric composite specimens.

Figure 10. SEM micrographs of the moisture absorbed specimens, depicting (a) water absorbed matrix, (b) water absorption and surface swelling, (c) voids and (d) decomposition after 100 days.

Figure 11. Mechanical properties of the various CPF reinforced polymeric composite specimens.

Figure 12. Comparison of tensile strengths of the various CPF reinforced polymeric composite specimens.

Figure 13. Comparison of flexural strengths of the various CPF reinforced polymeric composite specimens.

Figure 14. Comparison of hardness values of the various CPF reinforced polymeric composite specimens.