Dynamic mechanical analysis and effects of moisture on mechanical properties of interwoven hemp/polyethylene terephthalate (PET) hybrid composites

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Abstract

A dynamic mechanical analysis was undertaken to determine the influence of moisture on the mechanical properties of interwoven hemp/polyethylene terephthalate (PET) hybrid composites. Composite laminates were fabricated using vacuum infusion process; form epoxy resin reinforced with interwoven hemp and PET fibres. Woven hemp, woven PET, and interwoven hemp/PET hybrid composites were produced. The hybrid hemp/PET composites yielded the highest final residue % due to the PET fibres which improved the thermal stability. The glass transition temperatures of the woven hemp, woven PET, and interwoven hemp/PET hybrid composites were 68, 67, and 69 °C, respectively. Water absorption tests were conducted, and tensile and flexural tests were conducted on the wet and dry specimens. The water uptake of the hemp/PET hybrid composite was half that of the woven hemp composites. The tensile and flexural strengths of the interwoven hemp/PET hybrid composites were 4% and 22% greater than those of the woven hemp composites, respectively.

Keywords: Hybrid composites; mechanical properties; dynamic mechanical analysis; moisture absorption; natural fibre

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1.0 Introduction

In recent years, growing environmental awareness has driven researchers to investigate the appropriate use of reinforcing materials in the production of polymer composites. Natural fibres, used as reinforcing agents, are in demand because they are relatively inexpensive, renewable, biodegradable, easily handled, non-abrasive, and have excellent mechanical properties and a low density[1–4]. Despite their advantages, natural fibres also have limitations, such as their high level of moisture absorption, low strength, and relatively poor durability; they are also incompatible with polymers[5]. However, through ongoing research, scientists have determined that such reinforcement materials can be hybridised with synthetic fibres, while the performance of natural fibres can be improved by treatment.

Polymer composites have more advantages and can be used in construction and building materials[6,7]. Some researchers have investigated the properties of externally bonded polymer composite systems that can be used to strengthen the structures of polymer-reinforced concrete. These can influence the polymer composite due to the effect of the temperature change and mechanical load[8]. Zhang demonstrated that the addition of macro polypropylene (PP) fibre can significantly improve the mechanical performance of a concrete element[9]. Kenaf-reinforced polymer composites, moulded into lightweight panels, have been used to replace solid and plastic-wood composites due to economic pricing[10].

The mechanical and thermal properties of natural fibres can be improved by hybridising them with either natural or synthetic fibres to form hybrid composites such as okra/glass[11], jute/glass[12], and kenaf/glass[13,14]. Yahaya suggested that kenaf/Kevlar hybrid composites could be used in impact applications because of their satisfactory performance[15]. In addition, Alavudeen stated that the strengths of composites with woven fabrics are typically superior to those of unidirectional and randomly oriented composites[16]. The woven fabric provides an excellent interlocking arrangement between the reinforced fibres, enabling uniform stress distribution, which increases the strength of the composite[17]. A study by Khan demonstrated that the strength of a woven jute-reinforced poly(L-lactic acid) composite was greater when tested along the direction of the warp yarn than along the direction of the weft yarn[18]. In addition, to improve the degree of fibre alignment, weaving, knitting, and braiding can be applied to the production of such polymer composites[19]. These hybrid composites are influenced by several factors, such as the fibre orientation, fibre volume fraction, and fibre/matrix interfacial bonding.

In a previous study, the thermal stability of a matrix polymer was improved with the incorporation of bamboo and glass. A previous researcher determined that the thermal properties of ramie fibre composites improved when they were hybridised with glass fibres[20]. Ridzuan studied the thermal and mechanical properties of *Pennisetum purpureum*/glass-reinforced hybrid composites. When the alkali concentration used for the treatment of the *Pennisetum purpureum* was increased, there was a reduction in the quantity of residue produced[21]. Moreover, the thermal stability of the composites improved when the natural fibres were hybridised with glass fibres; in addition, the improvement was also observed when the natural fibres were chemically modified via treatments with NaOH and silane[22].

The polar groups (hydroxyls) of the cellulose and lignin within the natural fibre allow the absorption of water molecules[5]. Hameem reported that when a composite is exposed to an

environment with high levels of humidity and moisture, water molecules are more likely to penetrate the gaps and cracks resulting from fibre swelling[23]. These factors result in poor fibre/matrix interfacial adhesion within the resultant polymer composite, and impair the mechanical properties of the composite. In addition, Akil found that the water molecules penetrated the composite through three routes, as follows; through the micro gaps between the polymer chain, through the cracks in the polymer matrix caused by fibre swelling, and through the flaws at the composite interphase because of the poor wettability between the fibre and the matrix[24].

In the present study, experimental investigations were conducted to evaluate the thermal, mechanical, and morphological properties of woven and interwoven hybrid composites. Hemp fibre was selected for the investigation because it is locally available and offers excellent water resistance[5]. The mechanical characteristics of the cost-effective natural/synthetic hybrid composites were evaluated. It is expected that the results of this study will provide evidence to support the development of hemp-reinforced composites with improved water resistance for outdoor structural applications.

2.0 Materials and methods

2.1 Materials

The hemp fibre, PET fibre, and epoxy resin were supplied by a local supplier in Malaysia. The hemp and PET yarn were used as reinforcing materials in the form of woven and interwoven fabrics, with dimensions of 300 × 300 mm. EpoxAmite 100 series resin was used as the matrix, by being mixed with a hardener, namely, EpoxAmite 103 slow hardener. An epoxy: hardener ratio of 100:28.4 (g) was used. The properties of the fibres and epoxy resin are listed in **Table 1**. The single-fibre test was conducted for the hemp and PET fibres, while the properties of the epoxy were taken from the literature[25].

2.2 Woven Fabric

A woven fabric sample was produced using a customised wooden frame measuring 400 × 400 mm; nails were placed on both sides of the frame to function as warp-yarn guides. The nails were carefully positioned on the wooden frame to ensure that the fabric remained taut during the weaving process. **Figure 1** shows the weaving process and the finished woven fabric. In the present study, woven hemp, woven PET, and interwoven hemp/PET hybrid fabrics were produced. In the case of the woven fabrics, identical fibre types were used as the warp and weft yarns. Meanwhile, for the interwoven fabric, during the weaving process, hemp and PET fibres were used as the warp and weft yarns, respectively, as shown in **Figure 2**.

2.3 Composite Fabrication

The composites were manufactured using a vacuum-infusion process. Composites were prepared using the different types of fabrics produced as described above, as listed in **Table 2**. The vacuum-infusion machine features a high-vacuum pump (AST 22, AIRSPEC), pressure gauge, suction hose, and a container, which is used to collect any residual resin, as shown in **Figure 3**. Initially, the woven and interwoven hybrid plies were laminated above a glass mould. Next, the resin was mixed, using the weight ratio specified by the manufacturer; the mixture was then allowed to flow through the hose (by evacuating the mould area) and into the residual resin container. Then, the in-mould pressure was controlled such that it was < 300 Pa when the vacuum pump was switched on. Finally, the infused composite was allowed to cure within the mould for 12 h at room temperature (25 °C). The composite plate

was cut using a Dremel 4000 tool to produce specimens with the required dimensions for testing, in accordance with the relevant standards.

2.4 Thermogravimetric Analysis (TGA)

The TGA results define the weight loss of the specimen with increases in the temperature or the duration of the analysis. Tests were performed on the woven and interwoven composites using a Gas Controller GC 200 STAR System Analyser (METTLER TOLEDO). To prevent oxidation, the test was performed under nitrogen at a flow rate of 20 ml/min. A specimen, with a weight of 5–10 mg, was heated on a platinum pan to maintain its temperature during the measurements, which were performed using a thermocouple. Then, the specimen was heated from 30 to 1000 °C at a heating rate of 10 °C/min[21].

2.5 Dynamic Mechanical Analysis (DMA)

The temperature-dependent properties, such as the storage modulus, E' ; loss modulus, E'' ; and damping factor, $\tan \delta$, can be measured using DMA techniques[26]. The loss modulus, E'' , was used to determine the glass transition temperature (T_g) of the woven and interwoven hybrid composites. A Pyris diamond DMA (Perkin Elmer) was employed to perform the tests, for which a double-cantilever clamp was used in bending mode. Specimens, with dimensions of 50 × 10 × 2.3 mm, based on ASTM D5418-07, were evaluated. The samples were heated at a rate of 3 °C/min, and maintained at a temperature of 30–150 °C, under a fixed frequency of 1 Hz[27].

2.6 Water Absorption

The water absorption behaviour of the samples was determined according to ASTM D570-10 [28]. Each specimen, measuring 76.2 × 25.4 × (3.2 ± 0.4) mm, was submerged in a container of distilled water, at room temperature (25 °C), for a period of up to 1400 h. Five specimens of each type of composite were tested. The mass of the water absorbed by each specimen was recorded. The moisture content percentage was calculated using Eq. 2.1, which is expressed as follows:

$$\Delta M(t) = \frac{M_t - M_0}{M_0} \times 100, \quad \text{Eq. 2.1}$$

where M_0 and M_t represent the masses of the dry sample and immersed sample, respectively, at any specific time. Subsequently, the ageing effect, concerning the tensile and flexural properties of the material, was investigated over periods of 0, 6, 24, 100, 400, and 1400 h.

2.7 Tensile Testing

Dog-bone-shaped specimens, with dimensions of 165 × 19 × (3.2 ± 0.4) mm, were prepared, in accordance with ASTM D3039-14[29]. The test was performed using a Shimadzu universal testing machine (UTM) with a load cell of 100 kN. Each specimen was tested for failure under tension; the crosshead speed was 1 mm/min. An extensometer was attached to the gauge length of the specimen. In addition, both directions of the hemp and PET fibres of the interwoven hemp/PET hybrid composite were successively oriented parallel to the loading direction during the test. Five specimens of each type of composite were tested, and the results were recorded. These tests were performed on the specimens in both their dry and wet states.

2.8 Flexural Testing

Flexural testing was conducted using a universal micro tester (INSTRON 5848) with a 2-kN load cell. A crosshead speed of 2.5 mm/min was used[24]. A rectangular specimen was

produced, with dimensions of $125 \times 13 \times (3.2 \pm 0.4)$ mm, in accordance with ASTM D7264-14[30]. A support-span length of 52 mm was used to satisfy the recommended support span-to-depth ratio of 16:1. Five specimens of each type of composite were tested, in both the dry and wet states, and the results were recorded. The specimens that had been immersed in water for various periods were tested, and the data obtained were plotted to obtain flexural stress-strain curves. Here, the flexural strength and failure strain were calculated using Eq. 2.2 and Eq. 2.3, respectively:

$$\sigma_f = \frac{3PL}{2bd^2} \quad \text{Eq. 2.2}$$

$$\varepsilon_f = \frac{6Dd}{L^2} \quad \text{Eq. 2.3}$$

where P , L , D , b , and d represent the bending load, support-span length, bending extension, and the width and depth of the specimen, respectively. The flexural modulus can be obtained from the initial slope of the flexural strength–strain curve.

2.9 Morphological Analysis

Following the tensile and flexural tests, scanning electron microscopy (SEM) analysis was conducted to study the morphological features of the fibre/matrix interface. The fractured surface of the composite was observed using a scanning electron microscope (Hitachi TM-3000), with a magnification of $100\times$. The surface of the fractured portion of the tested composite was uniformly coated with platinum prior to scanning. An accelerating voltage of 3–5 kV was used to obtain the images[23].

3.0 Results and Discussion

3.1 Thermal Analysis

3.1.1 Thermogravimetric Analysis (TGA)

The thermal stability of a composite can be determined using the initial decomposition temperature, final degradation temperature, and the residual content, as shown in

Table 3. The thermal degradation curves obtained for the woven and interwoven hybrid composites are shown in **Figure 4**. The initial degradation temperatures of the woven hemp (HH), woven PET (PP), and interwoven hybrid (HP/PH) composites were 92.5, 100, and 98.8 °C, respectively. At low temperatures, the weight loss of the sample corresponds to the removal of moisture from the polymer matrix[31]. The curve shows that, amongst the samples tested in the present study, the woven hemp (HH) composite underwent weight loss at the lowest temperature, 92.5 °C. This can be explained by the large quantity of moisture that was absorbed by the hemicellulose of the woven hemp fibre. It is possible that the moisture penetrated the multicellular structures at the fibre/matrix interface[25,32,33].

Following the test, the remaining amount of residue can be used to determine the percentage weight loss values of the woven hemp (HH), woven PET (PP), and interwoven hybrid (HP/PH) composites; these composites had final degradation temperatures of 470, 499, and 486 °C, respectively. However, the amount of final residue (char residue) differed for each type of composite. The woven hemp (HH) composite had the lowest final residue percentage of 1.7%, followed by the woven PET (PP), and then interwoven hybrid (HP/PH) composites, with percentages of 2.4 and 9.9%, respectively. As expected, the highest final

residue percentage was determined for the interwoven hybrid (HP/PH) composite; this was influenced by the presence of the PET fibre, which has a relatively high thermal stability. The results also suggest that, following testing, the natural fibre/synthetic fibre hybrid-reinforced composite had a charred structure, which indicates that the synthetic fibre was able to withstand high temperatures because of the compatibility between the fibre and the matrix[31]. Moreover, it can be assumed that the composite exhibited a higher final residue percentage because of the presence of the PET fibre.

3.1.2 Derivative Thermogravimetric Analysis (DTG)

Figure 5 illustrates the derivative thermogravimetric (DTG) analysis curves obtained for the various composites. The decomposition temperature of each specimen was between 300 and 650 °C; this corresponds to the peak of the respective curve. The woven hemp (HH) composites exhibited a two-step decomposition process, where decomposition occurred at 363 °C and 615 °C; this corresponds to maximum decomposition rates of 1.98 and 0.07%/°C, respectively. The decomposition peak observed at 615 °C could be attributed to the thermal degradation of the cellulose, which led to the hemp fibre exhibiting a 20% weight loss. A high temperature was required for further degradation of the resulting residue to occur. The degradation behaviour of the hemp fibre is similar to that of cynocell, cynowood flour, and other natural fibres studied by Girija[34].

Similarly, as shown by the DTG curves obtained for the woven PET (PP) composite, the decomposition commences at 171 °C, with a maximum decomposition rate of 0.04%/°C. The second deformation stage occurs at 363 °C, with a maximum decomposition rate of 0.74%/°C. Meanwhile, in the case of the DTG curve obtained for the interwoven hybrid (HP/PH) composite, only one peak was observed, which had a greater intensity than those of other composites; here, a maximum decomposition rate of 2.38 %/°C was achieved at 363 °C. This indicates that, in the case of the interwoven composites, the hybridisation of the hemp with the PET fibre influenced the rate of decomposition; this was due to the change in the degree of stability of the cellulose. The peaks of the DTG curves also reveal changes in the degradation behaviours of the woven hemp (HH) and interwoven hybrid (HP/PH) composites. These results show that thermal degradation can occur at accidental extreme temperatures such as those generated by a fire. The degradation temperatures were all above 60 °C. In addition, the natural fibre-reinforced polymers were expected to undergo moisture evaporation at temperatures of between 60 and 100 °C, which was indicated by the initial peak of each curve. Such peaks were not observed in the cases of the specimens consisting of the woven and interwoven fabrics that were initially dried in an oven at 100 °C for 30 min, which was conducted to remove any moisture present within the fabric. This action could explain the absence of the aforementioned initial peak.

3.2 Dynamic Mechanical Analysis (DMA)

3.2.1 Storage Modulus

The storage modulus (E') is used to measure the ability of a material to store applied energy. It is related to Young's modulus, which determines the stiffness of a composite material[22]. **Figure 6** shows the effect of temperature on the storage moduli of the woven and interwoven hybrid composites. These thermal stabilities were studied over a temperature range of 40–160 °C. As shown by the curves, for each specimen, the storage modulus decreases as the temperature increases. The plot also indicates that, the interwoven hemp/PET hybrid (HP) composite had the greatest storage modulus value. This was followed by the woven hemp (HH), woven PET (PP), and the interwoven PET/hemp hybrid (PH) composites.

Significant differences in the storage moduli of the specimens were observed between 56 and 80 °C. The curves of the storage moduli for the woven PET (PP) and interwoven PET/hemp hybrid (PH) composite specimens began to decrease sooner than those of the woven hemp (HH) and interwoven hemp/PET hybrid (HP). Increasing the temperature beyond the transition glass temperature, T_g , causes a material transition, from the glassy to the rubbery state, to occur. The components of a composite in the glassy state are closely and tightly packed, highly immobile, and exhibit strong intermolecular forces that contribute to a higher storage modulus[35–37]. As the temperature increases, the molecular movement would increase and the molecular bonding would relax, resulting in the loss of stiffness and hence reducing the value of the storage modulus. In addition, in the rubber region that appears at 100 °C and above, the maximum storage modulus of the composite is attained, because the epoxy resin becomes unstable. However, the interwoven PET/hemp hybrid (PH) composite had a lower storage modulus value than that of the woven and interwoven hemp/PET hybrid (HP). This is due to the modulus of elasticity (Young's modulus) of the hemp fibre being greater than that of the PET fibre. Therefore, a higher storage modulus is associated with a material being more elastic. The storage modulus values of the interwoven hybrid composites vary as a result of the presence of the PET fibres within the composite. Thus, amongst the composite materials studied, the interwoven hemp/PET hybrid (HP) composite had the highest maximum storage modulus. This is influenced by the degree of interfacial adhesion between the fibre and matrix. In addition, the incorporation of the PET fibre imposed a high degree of restriction which supports efficient stress transfer throughout the fibre/matrix interface. This phenomenon has also been demonstrated by the DMA results for intralaminar Curaua/glass, where the incorporation of synthetic fibre results in an increase in the storage modulus. The storage modulus can also be increased via the stiffening effect that which occurs between the fibre and matrix, and which supports efficient stress transfer[38]. In addition, a previous study of woven fabric glass/carbon fibre-reinforced epoxy composites has shown that the storage modulus increases as the hybridization of the carbon-layer laminates is increased[37].

3.2.2 Loss Modulus

During a deformation cycle, the energy dissipated as heat by the system, as a result of viscous motion within the material, is represented by the loss modulus, E'' . **Figure 7** is a chart showing the loss moduli as a function of temperature for a range of 40–160 °C, determined for the woven and interwoven hybrid composites. As shown in the curves, in the case of each specimen, the value of the loss modulus increased until the highest peak and subsequently decreased as the temperature increased. Initially, it can be seen that the interwoven hemp/PET hybrid (HP) composite exhibits the greatest loss modulus, followed by the woven hemp (HH), woven PET (PP), and interwoven PET/hemp hybrid (PH) composites. The maximum value of the loss modulus can be used to determine the maximum rate of heat dissipation, which represents the glass transition temperature (T_g). The glass transition temperature is associated with the shift from the glassy to the rubbery state. The behaviour represents the free movement of the polymer chain at high temperatures[22,35,39]. Conversely, previous researchers have stated that, compared with the damping factor, the use of the loss modulus value is more appropriate for determining T_g [39,40].

Table 4 presents the T_g values determined for the woven and interwoven hybrid composites. The woven hemp (HH) composite had a T_g value of 68 °C, while the interwoven PET/hemp hybrid (HP) composite had a greater T_g value of 69 °C. This shift in T_g to a higher temperature is associated with the lower mobility of the matrix molecules of the interwoven PET/hemp hybrid (HP) composite[35,38]. This change could be a result of the changes in the crosslinking density of the network. Besides, the stress fields surrounding the preforms resulted in a shift in T_g . In the present study, the lowest loss modulus value was determined for the interwoven PET/hemp hybrid (PH) composite. At the glass transition temperature, the interwoven hemp/PET hybrid (HP) composite exhibited the highest loss modulus value. Thus, the hybridisation of the hemp and PET fibre results in a tight arrangement between the components of the composite. This improves the interfacial adhesion that exists between the fibre and the epoxy matrix, which consequently enhances the structural mobility of the polymer chains of the composite. Yakubu stated that the hybridisation and tight arrangement between the Kenaf and PET fibres improves the fibre/matrix interfacial adhesion of the composites[14]. A previous study reported that frictional resistance to molecular movement could result in the dissipation of heat to the interface[21,41]. With an increase in the rate of the heat dissipation, the intensity of the internal forces within the composite also increases. The fibre/matrix interface of the interwoven hemp/PET hybrid (HP) composite exhibited the greatest loss modulus. This was due to the incorporation of the PET fibres, which resulted in an increase in the amount of energy dissipated. This also causes the transition peak to shift to a higher temperature and greatly increases the loss modulus value corresponding to T_g [37]. In addition, the physical state of the polymer matrix surrounding the fibres was altered, which restricted the molecular movement that leads to the energy dissipation[42].

3.2.3 Damping Factor

Damping refers to the dissipation of energy or the absorption of energy by a material under a cyclic load. It can be expressed as a dimensionless number in terms of the phase angle ($\tan \delta$). It can also be defined as the ratio of the loss modulus to the storage modulus ($\tan \delta = E''/E'$). The damping factors of the woven and interwoven hybrid composites, measured over a range of temperatures, are shown in **Figure 8**. The damping factor can be seen to increase with the temperature. The woven hemp (HH) composite exhibited greater damping factors than the woven PET (PP) composite. A previous study found that the reinforcement fabric of the composite acts as a barrier, limiting the movement of the polymer chain. This reduces the flexibility, molecular motion, and hence the damping factor of the composites[42]. A number of other factors also affect the damping behaviour, such as the interphase zones, fibre distribution, void content, frictional resistance, fibre breakage, matrix cracking, and fibre/matrix interfaces associated with the composites[43]. In the present study, the highest identified phase angle was that for the interwoven hemp/PET hybrid (HP) composite because it exhibited the greatest degree of molecular mobility. Meanwhile, the lowest phase angle was identified for the interwoven PET/hemp hybrid (PH) composite; this was a result of the reduction in the energy-loss rate. A material with a low phase angle is likely to possess enhanced load-bearing properties. Therefore, when the fibre/matrix interfacial bonding is enhanced, the molecular mobility of the polymer decreases; this consequently results in a reduction in the damping factor. When the degree of stiffness of a composite is increased, the degree of freedom of the polymer chain is limited; this restricts the molecular mobility, resulting in a decrease in the damping value of the material[35,44].

3.3 Water Uptake

The percentage weight gain was determined by calculating the difference between the weights of the dry specimens, and those of the corresponding wet specimens, which had been

immersed in distilled water at room temperature (25 °C); this is defined by Eq. 2.1. The composites were assumed to attain a moisture equilibrium state when the weight gain of the specimen was lower than 0.01% [45]. The water absorption curves of the woven and interwoven hybrid composites are shown in **Figure 9**, in which the water uptake percentage is plotted against the square root of the immersion period. Each data point in the graph represents the average of the results obtained from five specimens of each type of composite. The water uptake of the specimen increased as the immersion period was extended, as indicated by the curves. Other studies have found that the water absorption of fibre-reinforced polymers increases as the immersion period is extended [25,32,46].

The rate of water absorption was initially linear and rapid. The rate eventually decreased as the immersion period was extended, until an equilibrium state was attained. The behaviour of the tested composites followed a Fickian diffusion process [24,47]. This can be explained by considering the water absorption characteristics of the hemp and PET fibres. Amongst the composites tested, the greatest water uptake percentage, 6.18%, was recorded for the woven hemp (HH) composite. This is due to the hydrophilic nature of the hemp fibre, which allows the absorption of water molecules. In addition, the hemp fibre contains cellulose, hemicellulose, and lignin. Cellulose is a hydrophilic polymer that consists of hydroxyl groups, which react with water molecules to form hydrogen bonds (-OH). When exposed to water, these bonds allow the woven hemp (HH) composite to absorb a relatively large quantity of water. This weakens the fibre/matrix interfacial adhesion of the composites, and reduces their strength. Hemp fibre is a highly permeable fibre, and thus can absorb a greater amount of water than the resin itself [32].

The hybridisation of the hemp with the PET fibres results in a reduction in the water uptake of the interwoven hybrid composites, to less than half that of the woven hemp (HH) composites. The moisture content of the composite decreases as the synthetic fibre content increases, as reported by Salleh [48]. This demonstrates that, with the incorporation of the synthetic PET fibre, the water penetrates the composite at a lower rate. In addition, since the PET fibre is water-resistant, the interwoven hybrid (HP/PH) composite was partially damaged by the water molecules that penetrated the cracks; this could be illustrated by observing the surface morphology of the fractured portion of the specimen. The composite contained hemp fibre, which swelled when it was exposed to moisture; this initiated microcracks in the surface of the brittle, thermoset epoxy resin. The cellulose content of the hemp fibre promotes the penetration of water through the interfaces with the cracks initiated due to the swelling of the fibre. Thus, the composite is subjected to more external stress, which leads to failure. As a result of the penetration of the water molecules, these cracks were continually propagated. This further weakened the bonds between the fibres and the matrix.

The woven PET (PP) composite has hydrophobic characteristics. The PET fibre does not contain cellulose that allows the absorption of water. Thus, compared with the woven hemp (HH) and interwoven hybrid (HP/PH) composites, the woven PET (PP) composite exhibits the lowest water uptake percentage of 1.91%. The hydrophobic nature of the epoxy resin matrix also plays a vital role in resisting water absorption. It acts as a binder for the reinforcing material, thus enabling load transfer and protecting the composite from environmental attack [17]. The water content of the composite was almost negligible because of the hydrophobic nature of the epoxy resin [49]. However, if the specimen was continuously exposed to water, the resin matrix would become more brittle. Consequently, water would enter the matrix and attack the composite material, resulting in the debonding of the fibre/matrix interface [50].

The diffusion coefficient represents the ability of the water to penetrate the composite. It is calculated from the slope of the plot of the water uptake percentage against the square root of the immersion period. The water uptake percentage of the composite reflects the value of the diffusion coefficient. Among the tested composites, the woven hemp (HH) composites exhibited the highest diffusion coefficients. This is due to the high cellulose content of the composite, which results in its absorbing large quantities of water. The diffusion coefficient is influenced by the fibre type and fibre content of the composite, as well as the level of humidity, and the presence of microcracks in the surface of the composite. Thus, the presence of the PET fibres within the interwoven hybrid composites results in a reduction in the diffusion coefficient. The presence of voids, which are created during fabrication, also accelerates the diffusion process. This consequently increases the water uptake percentage[25].

3.4 Tensile Properties

The woven and interwoven hybrid composites, in the dry and wet states, were subjected to tensile testing to determine their strengths and elastic moduli. To obtain wet samples, the specimens were immersed in water for various durations; subsequently, the wet specimens were tested to observe their water-degradation response. **Figure 10** displays the tensile properties of the woven and interwoven composites, as measured after their immersion in water for a period of up to 1400 h. The tensile strength and elastic modulus values of the dry woven hemp (HH) composite were 39 and 33% greater than those of the woven PET (PP) composite, respectively. However, the benefits of hybridisation can be observed in the case of the interwoven hemp/PET hybrid (HP) composites, which, amongst the composites tested in this study, exhibited the greatest tensile strengths in both their wet and dry states. The dry interwoven hemp/PET hybrid (HP) composite yielded a strength of almost 74 MPa, which is 4 and 42% greater than those of the woven hemp (HH) and PET (PP) composites, respectively. The wet interwoven hemp/PET hybrid (HP) composites exhibited strength of 56 MPa, which is 27 and 38% greater than those of the wet, woven hemp (HH) and PET (PP) composites, respectively; this further demonstrates the effect of the hybridisation.

The interwoven hybrid composite was able to withstand a greater amount of applied stress than the woven hemp (HH) and woven PET (PP) composites. Several factors influenced the strength of the composites, such the strength of the fibre, the fibre content of the composite, and the degree of interfacial bonding between the fibre and the matrix[51]. In the case of this study, the incorporation of the hemp and PET fibre resulted in an enhancement of the tensile properties of the composite. The interlacing of the fibres of the hemp and PET within the matrix results in the development of a taut structure, which makes it difficult for the fibres to be pulled out under applied loads. Thus, the interfacial bonding between the fibre and matrix is improved, and consequently, the strength of the interwoven hybrid composite increases. This can be verified by the morphological images of the composites, which reveal that the fibre bundles are attached to the matrix. This will be discussed further, below.

However, the interwoven PET/hemp hybrid (PH) composite, in the dry state, was determined to have tensile strength and elastic modulus values that were 47% and 36% lower than those of the woven hemp (HH) composite, and 14% and 4% lower than those of the woven PET (PP) composite, respectively. This can be attributed to the complex load-sharing behaviour that occurs between the hemp and PET fibres along the transverse and longitudinal directions, which results in a greater stress capacity[15,52]. The fibre acts as a load within the matrix of the composite. The tensile strength is greatly dependent on effective and uniform

stress distribution[53]. In the present study, the increased strength of the interwoven hybrid composite was primarily attributed to the high strength of the hemp fibre rather than the low strength of the PET fibre[54]. This is indicated by the strength properties shown in **Table 1**; the strength of the hemp fibre (250 MPa) was double that of the PET fibre (100 MPa). The fibre was orientated in the direction of the applied load, and this influenced its strength[55]. This explains why the tensile strength and elastic modulus of the interwoven hemp/PET hybrid (HP) composite were greater than those of the interwoven PET/hemp hybrid (PH) composite.

Figure 10 also shows that the tensile strength and elastic modulus values of the wet specimens were lower than those of the dry specimens. It can be seen that, following their immersion in water for 1400 h, the strengths of the woven hemp (HH), woven PET (PP), interwoven hemp/PET hybrid (HP), and interwoven PET/hemp hybrid (PH) composites decreased by 42, 20, 24, and 32%, respectively; the respective Young's modulus values also decreased by 54, 12, 45, and 44%. This reduction in strength is probably a result of the reduction in the degree of interfacial bonding between the fibre and matrix[25,46,49]. The tensile properties were influenced by the amount of water that was absorbed by the specimens, which was governed by the length of the immersion period. These water-ageing effects have also been observed by previous researchers with regards to polymer composites[32,45,56]. Among all the composites tested, the woven PET (PP) composite was least affected by water exposure; this was followed by the interwoven hemp/PET hybrid (HP) composite.

It is suspected that the hemp fibre began to swell when it was exposed to water. This changed the dimensions of the composites and initiated microcracks within the matrix. The cellulose present within the fibre caused the water molecules to penetrate the interfaces of the microcracks[24]. This consequently reduced the stiffness of the fibre; in addition, shear stress was generated at the fibre/matrix interface, which resulted in the debonding of the composite. In addition, the ability of the specimen to bear external stress is influenced, to some extent, by the hydrogen bonds and van der Waal bonds that exist between the molecules of the composite[57]. When the specimen is exposed to water for an extended period of time, hydrogen bonds form between the macromolecules of the fibre and the matrix of the composite. The water molecules are attracted to the hydroxyl groups (-OH) of the hemp fibre, and form a large number of hydrogen bonds[58]. The adhesion of the polymer chains becomes impaired, and the interface becomes weaker. This reduces the degree of interfacial adhesion between the fibre and matrix, which results in the degradation of the tensile properties. This indicates that the hybridisation with the PET fibres helps to reduce the degradation rate of the hybrid composites[17].

3.5 Flexural Properties

A flexural test was used to determine the ability of the specimen to withstand bending prior to breaking. The bending strength of a composite is determined by both its compression strength and shear strength[16]. The flexural modulus is a measure of the resistance to deformation as caused by deflection[42]. The flexural properties determined for the woven and interwoven composites, in the dry and wet states, are presented in **Figure 11**. In the dry state, the woven hemp (HH) composites achieved relatively high flexural strength and modulus values of 80 MPa and 2.9 GPa, respectively; these values were 27 and 14% greater than those of the woven PET (PP) composites. The superior flexural behaviour of the woven hemp (HH) composite was primarily due to the high strength of the hemp fibre, as shown in **Table 1**.

Figure 11 indicates that among the dry specimens, the highest flexural strength (102 MPa) and modulus (4.9 GPa) values were determined for the interwoven hemp/PET hybrid (HP) composites; these values were 22% and 41% greater than those for the woven hemp (HH) composite, and 43% and 49% greater than those of the woven PET (PP) composite. The enhancement of the flexural properties shows that the interwoven hemp/PET hybrid (HP) composites can withstand a larger bending load than the woven hemp (HH) and woven PET (PP) composites. Prior to failure, the stress initially advances along the longitudinal dimension of the fibre before proceeding along the transverse dimension. The fibre bundles are removed when a load is applied, resulting in failure. This failure is a result of various mechanisms, such as tension, compressive stress, and shear stress[32]. Alavudeen reported that, in the case of woven hybrid composites, the elongation of the fibre yarn along the longitudinal and transverse directions depends on the types of fibre used in the warp and weft directions of the materials[16]. In our study, the interwoven hemp/PET hybrid (HP) composites exhibited superior flexural behaviour to those of the woven hemp (HH) and woven PET (PP) composites, which verifies the aforementioned theory.

Meanwhile, the dry specimens exhibited greater flexural strength and modulus values than the wet specimens (those which had been exposed to water for 1400 h). The strengths of the woven hemp (HH), woven PET (PP), and interwoven hemp/PET hybrid (HP) composites in the wet condition were 49, 23, and 23% lower than those in the dry condition, respectively. The flexural modulus values also exhibited a similar response, with reductions of 55, 36, and 45% recorded for the woven hemp (HH), woven PET (PP), and interwoven hemp/PET hybrid (HP) composites, respectively. Even though they also exhibited degradation, the hybrid composites still yielded a strength value that was 49% and 43% greater than those of the woven hemp (HH) and woven PET (PP) composites. In addition, the flexural modulus value of the hybrid composite was 52 and 41% greater than those of the woven hemp (HH) and PET (PP) composites, respectively. The fibres and matrix of the composites were adversely affected by the exposure to water, which affected the flexural properties. This indicates that the presence of the PET fibres decelerated the rate at which the moisture was absorbed by the composite. The hybridization of hemp fibres with PET fibres thus has a significant effect with regard to the reduction of the mechanical degradation rate of the fibre-matrix interphase. The incorporation of the hemp and PET fibres within the interwoven hybrid composite also resulted in the provision of an excellent interlocking arrangement, which further promoted the bending capacity of the material. Thus, the interwoven hemp/PET hybrid (HP) composite could tolerate a higher load before failure occurred.

It is also suspected that the moisture absorbed by the composite attacks the cellulose of the hemp fibre, and fills the gaps between the fibre and matrix, causing swelling. The initiation of the microcracks was caused by the swelling resulting from the prolonged exposure to water. This led to a reduction in the fibre stiffness. In addition, shear stress was generated at the interfaces of the composite, which resulted in the ultimate debonding of the fibres, as well as delamination, and a loss of structural integrity[49]. The weak bonding of the fibre/matrix interface eventually resulted in the impairment of the flexural properties of the composite. Besides, the poor wettability of the material, and the presence of microgaps within the polymer, also led to a reduction in the flexural strength and modulus values of the composite[24,57]. A previous study also found that the presence of moisture within the fibres and matrix of a composite decreases its ability to withstand stress[32]. This is in good agreement with the SEM observations of the fractured surfaces of the specimens.

3.6 Surface Morphologies of Fractured Specimens

In the present study, SEM was used to examine the fracture-surface morphologies of the woven and interwoven composites that were subjected to tensile and flexural tests. **Figure 12**, **Figure 13**, and **Figure 14** show the surface morphologies of the dry and wet specimens. These specimens were selected to elucidate the effect of the water absorption on the fibre/matrix interfaces of the composites. Effective stress transfer between the fibre and the matrix, resulting from good interfacial bonding, was promoted by maximising the fibre strength[59,60]. **Figure 12(a)** shows that the specimen that was tested when dry exhibited fibre breakage; this suggests that good interfacial bonding existed at the fibre-matrix interphase. Hollow areas, or voids, which are formed during fabrication, can also be observed in **Figure 12(a-b)**. In addition, **Figure 12(b)** shows the bending of the fibre, which is due to flexural failure. Similar observations were also reported by Atiqah[13].

However, the samples that were tested while wet exhibited surface failures, which can be attributed to fibre pull-outs, voids, fibre/matrix debonding, and matrix cracking. These are illustrated in **Figure 13** and **Figure 14** for the tensile and flexural test samples, respectively. The absorption of water by the composites is thought to result in swelling and plasticization of the polymer matrix, which impairs the fibre/matrix interfacial bonding[57]. **Figure 13(a)** shows the fracture surface of the composite, which has a cracked matrix. **Figure 13(c)** shows a reduction in the epoxy content of the composite. This is due to the plasticization effect, which leads to leaching of the resin. In addition, **Figure 13(d)** represents the effect of water molecules that have been observed on the fractured matrix surfaces. This phenomenon has resulted in the significant degradation of the tensile strength and decreases the stress-transfer capability of the composite. Other than that, voids, which can be attributed to fibre pull-out, and debonding of the fibre/matrix interface, could be clearly observed at the fracture surface, as illustrated in **Figure 13(b)**. Water molecules penetrate the voids and microcracks in the epoxy resin, even though it offers superior water resistance. The voids act as stress concentrators, which result in the debonding failure of the fibre/matrix interface. As illustrated by the experimental results, the presence of the PET fibre within the interwoven hemp/PET hybrid (HP) composite obstructs the ingress of water into the composites, and thus promotes interfacial bonding between the matrices.

Figure 14 shows the fracture-surface morphologies of the woven and interwoven hybrid composites that were subjected to the flexural tests. Voids due to fibre pullout, fibre breakage, fibre/matrix interfacial debonding, matrix cracking, and a non-uniform matrix surface texture due to the effect of water molecules, can all be observed, as shown in **Figure 14(a-d)**; these observations are similar to those for the samples that were subjected to tensile testing. These observations indicate that the weakened interfacial strength of the interwoven hybrid composite is due to poor fibre/matrix adhesion. The moisture exposure greatly influenced the debonding of the fibre/matrix interface, which resulted in the gradual decline of the flexural properties. This explanation is supported by **Figure 14(b)**. This figure shows that the fibres and epoxy resin were impaired because of the plasticization effect, which resulted in interfacial failure[23]. In addition, **Figure 14(d)** shows the effect of the water molecules that form on the fractured matrix surfaces. This phenomenon results in a significant degradation in the flexural strength of the composite. Voids due to fibre pullout and debonding of the fibre/matrix interface can also be observed, as shown in **Figure 14(c)**. This suggests that the fibre pullouts result in further detachments of the fibre and matrix, which consequently increases the stress concentration within the composite, accelerating the failure[25].

Conclusion

The effects of hybridisation on the thermal behaviour and moisture degradation of woven hemp (HH), woven PET (PP), and interwoven hemp/PET hybrid (HP) composites were investigated. Hybrid composites were fabricated, using different fibres, which were interwoven between the hemp and PET yarns. The effects of the hybridisation were investigated using TGA, DMA, tensile tests, flexural tests, and morphological observations of the prepared composite specimens. The following conclusions were drawn based on the extensive experimental study:

- a) The results of the TGA performed on the interwoven hybrid composites showed that the final residue percentage decreased following the hybridisation of the PET fibre.
- b) Regarding the DMA testing, the maximum storage modulus and loss modulus values were recorded for the interwoven hemp/PET hybrid (HP) composite. In contrast, the highest damping factor was recorded for the interwoven PET/hemp hybrid (HP).
- c) The interwoven hemp/PET hybrid (HP) composites had tensile and flexural properties that were superior to those of the woven hemp (HH) and PET (PP) composites.
- d) The moisture absorption plot shows that the highest moisture-uptake percentage was recorded for the woven hemp (HH) composite.
- e) The presence of the PET fibres was found to reduce the water uptake of the interwoven hybrid composites. Voids, fibre pullouts, matrix cracking, and breakage, caused by the presence of moisture, could be observed within the SEM images.
- f) Thermal tests (such as the DMA method) that generate data, define the dynamic mechanical properties of the woven and interwoven hybrid composites in order to support development, such as is the construction and automotive industries, where these composites can be used as replacements for concrete, steel, and wood reinforcements.
- g) Finally, hybrid composites could be used as construction materials in the housing industry, in which, as an engineered material, they could be used to fabricate strong, lightweight, and thermal insulating building components.

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Figure Captions

Figure 1: (a) Weaving process, (b) Fabric

Figure 2: Warp and weft fibre directions used for the interwoven hybrid composite

Figure 3: Vacuum infusion system

Figure 4: Thermogravimetric analysis (TGA) curves obtained for woven and interwoven hybrid composites

Figure 5: Derivative thermogravimetric analysis (DTG) curves obtained for woven and interwoven hybrid composites

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Figure 7: Loss modulus curves of woven and interwoven hybrid composites

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Figure 14: SEM images of woven and interwoven hybrid composites subjected to flexural testing when wet

Tables

Table 1: Mechanical properties of fibres and epoxy resin

Mechanical properties	PET	Hemp	Epoxy
Tensile strength (MPa)	100	250	55
Elastic Modulus (GPa)	0.9	6.9	1.75
Tensile Strain (%)	1.5-1.8	2-4	6
Absorption Rate (%)	-	6.5	-

Table 2: Symbols used for various types of prepared composites

Type of composite	Symbol
Woven hemp	HH
Woven PET	PP
Interwoven hemp/PET hybrid along hemp direction	HP
Interwoven PET/hemp hybrid along PET direction	PH

Table 3: Thermogravimetric analysis (TGA) results obtained for woven and interwoven hybrid composites

Composites	Degradation temperature (°C)		Char residue (%)
	Initial	Final	
HH	92.5	470	1.7
PP	100	499	2.4
HP/PH	98.8	486	9.9

Table 4: T_g values obtained from loss modulus and damping curve

Composite	T_g obtained from loss modulus curve (°C)	T_g obtained from damping curve (°C)
HH	68	74
PP	67	73
HP	69	75
PH	63	73

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Figure 20: Storage modulus curves of the woven and interwoven hybrid composites

Figure 21: Loss modulus curves of woven and interwoven hybrid composites

Figure 22: Damping curves of woven and interwoven hybrid composites

Figure 23: Water absorption curves of woven and interwoven hybrid composites

a) Tensile strength of specimen

b) Elastic modulus of specimen

Figure 24: Tensile behaviour of woven and interwoven hybrid composites

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a) Specimen following tensile failure

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a) Matrix cracking

b) Void and debonding of the fibre/matrix

c) Reduction in the epoxy content due to plasticization effect

d) Effect of water molecules

Figure 27: SEM images of woven and interwoven hybrid composites subjected to tensile tests when wet

a) Matrix cracking

b) Reduction in the epoxy content due to plasticization effect

c) Void and debonding of the fibre/matrix interface

d) Effect of water molecules

Figure 28: SEM images of woven and interwoven hybrid composites subjected to flexural testing when wet