

The effect of fibre treatment on water absorption and mechanical properties of buri palm (*Corypha utan*) fibre reinforced epoxy composites

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ABSTRACT – Over the past century, there has been a dramatic increase in natural fibre composites in which natural fibre has served as reinforcement in polymer. However, the existence of moisture and defects in natural fibres has impacted the mechanical and physical properties of natural fibre polymer composites. The main objective of this study is to fabricate the buri palm fibre reinforced epoxy composite and evaluate the effects of fibre treatment on water absorption and tensile properties. The buri palm fibre were treated by using 5 wt.% NaOH for 24 h and the laminated composite of untreated and treated four-layer and five layer fibres were fabricated via hand lay-up process. The tensile specimens are prepared according to the ASTM D638 standard and the water absorption experiment was conducted by immersing the specimen in distilled water at room temperature until it reached the saturated moisture absorption. The results revealed that the percentage of moisture uptake was reduced to 69% and 95% in treated four-layer and five-layer sequences. It is observed that the thickness swelling of the composite increased with the increase of sequence layering, while the thickness swelling decreased with treated fibre. Alkali treatment affected the properties of buri palm fibre which improved the interfacial bonding between the fibre and epoxy matrix for better tensile properties and reduced water absorption. Finally, morphology examinations were carried out to analyse the fracture behaviour and fibre failure on the tensile test specimen by using microscope analysis.

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INTRODUCTION

In recent years, there has been increased interest in environmentally friendly materials that have good properties. In the previous years, natural fibres have attracted much attention in the utilisation and economical use of polymer composites. Natural fibres can be sustainable with appropriate property requirements and handling [1-2]. Natural fibre composites have few advantages over synthetic fibre composites in terms of environmentally friendly nature, sustainability, lightweight, high strength with water, electrical, corrosion and fire-resistant properties [3-7]. Previous studies have reported the many uses of natural fibre composites in manufacturing, automotive, furniture, aircraft, building, and construction industries, which are currently growing rapidly due to the greater mechanical properties and lightweight [3-7]. The mechanical properties of natural fibre depend on chemical compositions, such as cellulose, hemicellulose, lignin, pectin, and moisture content [6-8].

Most natural fibres contain a large amount of cellulose (semi-crystalline polysaccharide) which is responsible for the hydrophilic nature and polymer matrix responsible for the hydrophobic characteristic [8-11]. Hemicellulose, lignin, wax, pectin, and moisture content affect the lack of bonding between fibres and polymer [9]. When the component of natural fibre and resin are combined, it will cause the mechanical properties to expend slightly due to the different fractions of natural fibre chemical composition which affect their interfacial adhesion between fibre and polymer [10]. Composite materials are strongly influenced by the mechanical properties, the matrix and fiber distribution, as well as the efficiency of stress transfer between two components. The mechanical properties of natural fibre reinforced polymer composite depend on a number of factors, which are volume fraction/ weight fraction, geometry, architecture, form of natural fibres (i.e., filler, short fibre, continuous long fibre), aspect ratio, and fabrication method [11,12]. To further increase the mechanical properties of natural fibre reinforced composite, surface modification on natural fibre was carried out by using a specific treatment. The surface modification has the ability to respond to the fibre structure and change the compositions. Fibre treatment reduces the moisture absorption and removes impurities (lignin, pectin, ash content), which resulted in an excellent incompatibility enhancement between the polymer matrix and fibre [7,13-14]. There are many types of surface

modification applied by researchers on natural fibres, such as alkaline treatment, permanganate, silanes, peroxides, benzylation, isocyanate, acetylation, titanate, and zirconate [8,9,15]. Among all, alkaline treatment is the most widely applied in many studies because it is a cheap and simple method to improve adhesion properties [13,15].

The major drawback of natural fibre reinforced composites is the sensitivity to humidity and high-water absorption and poor fibre matrix adhesion. In order to enhance the fibre matrix adhesion and improve the mechanical properties, alkaline treatment has been considered as a better solution [14,16-17]. Sreekumar et al. [12] examined the moisture uptake on the effect of polyester-sisal fibre by using various surface modifications, such as 5 wt.% sodium hydroxide (NaOH), benzylation, silane, thermal, and permanganate treatment. They found that the fibre treated with 5 wt.% NaOH performed better than the other surface modifications, which increased by 36% in tensile strength and 53% in Young's modulus. Mathivanan et al. [16] reported that pineapple leaf fibre (PALF) treated with 5 wt.% NaOH affected the mechanical properties, which increased by 21.3% in tensile strength and 11.1% in flexural strength. Ramadevi et al. [17] noted that the moisture absorbed can be reduced by the NaOH treatment of fibre. A few studies had mentioned the methods available to improve the mechanical properties of natural fibre in order to block the OH group of cellulose molecule in the fibre by using chemical treatment, hybridisation, and appropriate layering as well as adding a compatibilising agent to improve the bonding between matrix and fibre [9,15,19]. Several studies on swelling, moisture absorption, and chemical resistance behaviours of natural fibre composites were conducted as important determinations for their various applications [14, 20-22].

Buri palm is also known as *Corypha utan* lamarck fibre. It is considered as part of the palm species (Arecaceae) is highly available in Indonesia, Philippines, and Malaysia [23]. It has various benefits whereby it is used intensively in house thatches and handcrafted products [24]. However, studies on buri palm fibre are scarce in literature. Accordingly, this study attempts to compare the swelling and the effects of fibre treatment on the moisture uptake and mechanical properties of buri palm fibre reinforced epoxy composite. Buri palm fibres were treated with 5 wt.% NaOH solution to improve the adhesion between the fibre and the matrix. This percentage (5%) was applied based on the previous research done by Rokbi et al. [25] which found the improvement in tensile and flexural properties of the composite. It is hoped that this data can contribute towards future research on the other applications of buri palm fibre reinforced epoxy composite as lightweight and low cost alternative composite material, and can subsequently be used in the industries, such as in the automotive industry.

METHODS AND MATERIALS

Figure 1 shows the plain woven buri fibres investigated. According to the density analysis by electronic densimeter (MD 300S), the density was recorded as 1.04 g/cm³. The buri fibre density is very low and it is of superior stiffness. Epoxy 816A with density of 1.2 g/cm³ and hardener epoxy 651 that were purchased from Impiana Z Enterprise, Kuala Lumpur, Malaysia, were used to fabricate the composite laminate in this study. The buri fibre reinforced epoxy composite was fabricated by uniformly mixing the epoxy resin and hardener by hand with the ratio 3:1 by weight to increase the bonding between resin and hardener. Another material used in this study was sodium hydroxide (NaOH) for surface modification.



Figure 1. Plain woven buri palm fibre

Alkaline Treatment

In order to swell the raw, plain woven buri fibres, the fibres were fully soaked in 5 wt.% concentration of sodium hydroxide (NaOH) for 24 h at room temperature to remove more lignin amount which had covered the fibre surfaces. This is followed by washing with distilled water until a neutral pH value (pH= 7) was obtained. To eliminate the moisture, the fibre was oven dried at 105°C for 24 h.

Composite Preparation

The composite was fabricated through a hand lay-up method to produce two different layer stacking sequences of woven buri palm fibre reinforced epoxy. This work used the four-layer and five-layer of woven buri palm fibre reinforced epoxy composite, as illustrated in Figure 2. Each layer of the buri palm fibre was cut into 300 x 300 mm and laid up one by one in aluminium moulds. A release agent was used for easy removal of the composite from the mould once it was cured. The epoxy was evenly poured over the woven fibre and was continued for the four-layer and five-layer woven i palm fibres. All the samples were performed according to the formulation given in Table 1. To remove the trapped air bubbles, a compression moulding machine was used and cured under 50 bars for 24 hours at room temperature. The composite specimens were cut according to the required dimension by using a laser cutter.

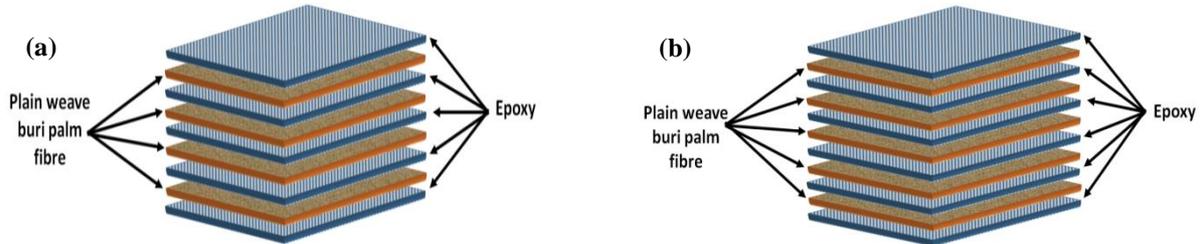


Figure 2. Plain woven buri palm fibre reinforced epoxy: (a) 4 layer and (b) 5 layer stacking sequence

Table 1. Composites sample formulation

Sample Abbreviation	Number of layering	Weight fraction of fibre	Weight fraction of epoxy	Surface treatment condition
Untreated (UT4)	4	11.03	88.97	None
Treated (T4)		10.87	89.13	5 wt.% NaOH
Untreated (UT5)	5	12.66	87.34	None
Treated (T5)		12.08	87.92	5 wt.% NaOH

CHARACTERIZATION

Water Absorption Test

The buri palm fibre reinforced epoxy composite were machined into the required measurement by using a laser cutter for water absorption and mechanical testing. Specimens for the water absorption test were set up according to the ASTM D638 standard for tensile test. The moisture absorption test was conducted in distilled water for various laminate stacking sequences that were prepared according to the tensile test standard. The initial weight of dry composite specimens was weighed by using the digital electronic balance. The sample was immersed in distilled water at different time intervals for 28 days at room temperature. The water was removed from the sample for weight measurement. The amount of moisture absorbed was noted by weighing the specimens. The water on the sample surface was wiped off by using a clean dry fabric before being weighed. The calculation of water absorption was obtained by using Eq. (1):

$$\text{Weight gain, (\%)} = \frac{W_w - W_d}{W_d} \times 100 \quad (1)$$

where W_w represents the sample in wet conditions after the immersion time, while W_d refers to the sample in dry conditions. Graphs were plotted for percentage weight gain against the submersion time frame length of 1 day, 7 days, 14 days, 21 days, and 28 days. The effect of water immersion on the durability of the composite specimen was experimentally investigated and discussed.

Thickness Swelling

One of the drawbacks of natural fibre composite is poor water resistance that will affect thickness swelling. To measure the reliability of dimensional changes in composite specimens when immersed in the water for a specific duration thickness, six samples from each of the two categories for untreated and treated of different layering sequence of buri palm reinforced epoxy composite were produced to examine the composite swelling and water absorption of the specimens (UT4=6, UT5=6, T4=6, T5=6). The specimen thickness was measured before and after the soaking period by using digital Vernier callipers. Thickness swelling can be computed by Eq. (2) and was performed according to the

procedure outlined in ASTM D570. For statistical purpose, the average and standard deviations for the sample test were calculated. Then a gradual change of thickness was observed.

$$\text{Thickness swelling, (\%)} = \frac{T_w - T_d}{T_d} \times 100 \quad (2)$$

where, T_w is the thickness after soaking, while T_d is the thickness before soaking.

Tensile Test

To study the effects of absorption on the mechanical properties of buri palm fibre reinforced epoxy composite, tensile test was performed on the untreated and treated woven four-layer and five-layer stacking sequences of the composite laminates under dry and wet conditions. The tensile strength specimens of plain woven buri palm fibre composites were prepared and machined according to the ASTM D638 procedures. The specimens were placed on a universal testing machine (i.e., Instron-3369 model) with the load of 50 kN. The test applied load at the upper crosshead by using a constant speed of 2 mm/min at room temperature until a fracture occurred on the specimen. These tests were carried out on the samples under dry and wet conditions. The load-displacement curves were obtained by using Bluehill software, and the tensile strength and modulus values were calculated from the curves. For each sample, five replicated specimens were tested for average accuracy.

Microscopic Analysis

Soptop soft imaging system, an optical microscopy machine model was used to capture the images of fracture effect. The effect of fracture surface for all tensile tests was examined under 2x magnification. The fibre-matrix interfacial bonding appearance and the effect of failure mechanism showed the buri palm fibre composite before and after the alkaline treatment, as observed under the microscope.

RESULTS

Moisture Absorption Behaviour

Plain woven buri palm fibre composites were treated with alkali to improve the tensile properties by enhancing the great surface adhesion property between fibre and matrix. The data of this study addressed that the treated fibre was lighter as compared to the untreated fibre. It happened based on the assumptions that alkaline treatment removes a certain amount of wax, lignin, and oil covering the external surface of the fibre [13]. In this case, the composite was fabricated based on four and five layering sequences. The moisture content of each specimen was determined by using Eq. (1). Based on the equation, the percentage of moisture absorption for all specimens was determined by the weight gain relative to the dry weight of samples. It was expected that the samples reached a saturated moisture absorption, while the interval period weight increase of the specimen was not more than 0.01% [26]. Figure 3 presents the moisture absorption percentage for different samples, which were immersed in distilled water at room temperature. From the data, it was observed that the water absorption content increased for a prolonged period of time. The finding was consistent with the findings of past studies by Hazim et al. [27] and Rassmann et al. [28], in which the water absorption reached a saturation point between 28 and 30 days.

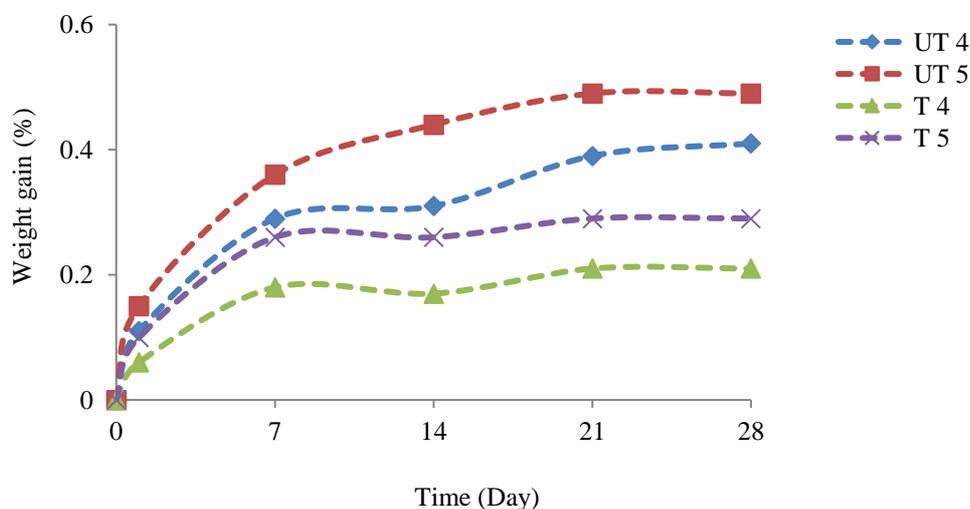


Figure 3. Water absorption curves of plain woven sequence stacking laminate composites

Initially, the water absorption of 4 and 5 layer stacking sequences of untreated and treated specimens were very rapid in the first week due to the water absorption which led to the swelling of the fibre, formation of voids, and micro cracks on the fibre matrix interface region. The water absorption was observed to be slowing down and reached a saturation level after 21 days of immersion, whereby the composite attained no further increase in water retention. In this situation, when the water absorption exceeds the saturation level, the bound and free water remain inside the composite as a reservoir. UT5 and UT4 woven fibre composites absorbed a higher amount of water as compared to T5 and T4, which were 69% and 95%, respectively. It was observed that the water absorption of treated fibre had reduced drastically, which was consistent with the literature [17]. The outcome was in line with a prior study by Maslinda et al. [26] which found that the woven individual kenaf composites absorbed more moisture because the cellulose content of kenaf was 72% as compared to jute and hemp fibres. Similarly, Venkatesh et al.[29] found that treated sisal and bamboo in polyester composites absorbed less water as compared to untreated sisal and bamboo in polyester composites, which were 9.1% and 19.6%, respectively. An investigation by Asim et al. [30] revealed that all untreated hybrid composites of kenaf/pineapple leaf absorbed more water than treated hybrid composites. The purpose of the alkaline treatment is to remove lignin and impurities [31], thus after the lignin is removed, the resin will easily penetrate to the fibre. Due to that, the interfacial adhesion of natural fibre and polymer composite will be improved. Water molecules cannot penetrate the fibre surface easily because it is already covered with polymer matrix. However, the water can still enter the cavity in the composite surface via capillary effect [28, 33].

Thickness Swelling

From the result, it can be seen that UT5 leads the rate of swelling, followed by UT4, which were 0.09% and 0.08%, respectively due to the presence of hydroxyl groups which attracted water molecules over hydrogen bonding [33]. This influenced the dimension changes in natural fibre composites, especially thickness and expansion due to composite swelling. Figure 4 illustrates that the fibres treated with 5 wt.% NaOH concentration significantly reduced the composite thickness swelling by 62.5% and 55.56% for T4 and T5, respectively. T4 and T5 composites reach the same thickness swelled by 0.01% with fibre composite soaking in distilled water for 1 day, 7 days, and 14 days and increased by 0.03% for 21 days, while only T5 increased by 0.04 % for 28 days.

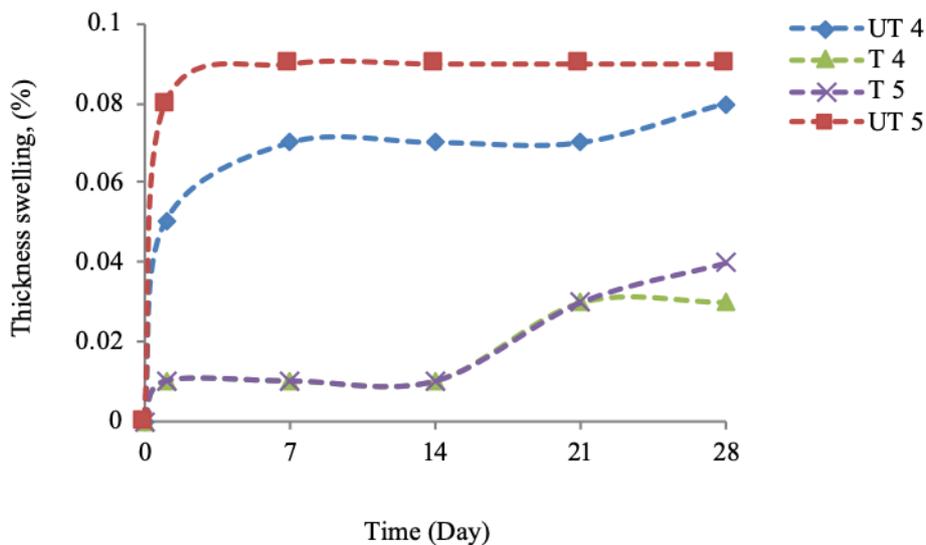


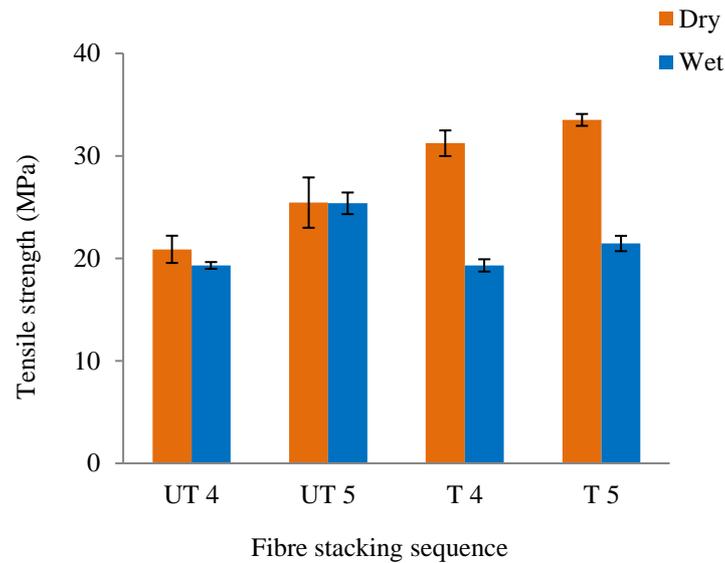
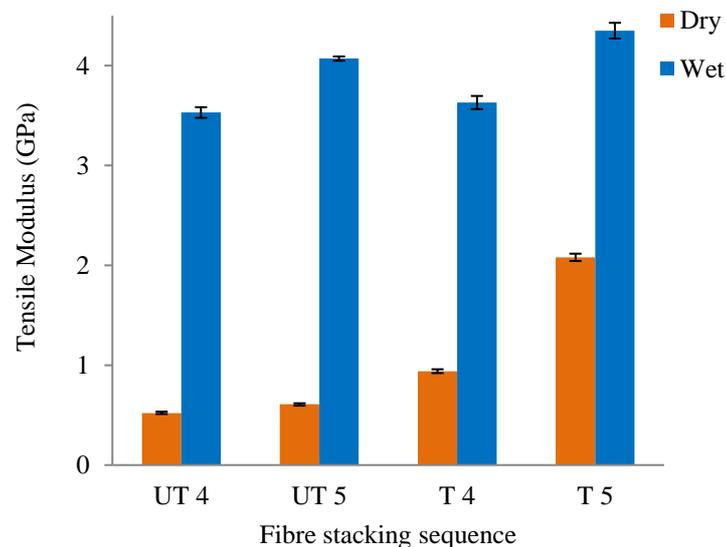
Figure 4. Water absorption curves of plain woven sequence stacking laminate composites

Tensile Properties

The tabulated data at Figure 5a and Figure 5b present the tensile strength and tensile modulus of specimens for various layer stacking sequences of UT4, UT5, T4, and T5 before and after the water absorption for a saturated period. It was demonstrated that the tensile properties of wet composite specimens significantly decreased as compared to dry composite specimens. The water uptake had affected the properties, structure, and interface between the fibre and matrix [34].

Table 2. Summary of the tensile properties of dry and wet buri palm fibre reinforced epoxy composites

Properties		Specimen			
		UT 4	UT 5	T 4	T 5
Tensile strength (MPa)	Dry	20.88	25.44	31.23	33.51
	Saturated	19.31	25.37	19.31	21.45
Tensile modulus (GPa)	Dry	0.52	0.61	1.94	2.08
	Saturated	3.53	4.07	3.63	4.35

**Figure 5a.** Summary of tensile strength of dry and wet buri palm fibre reinforced epoxy composites**Figure 5b.** Summary of tensile modulus of dry and wet buri palm fibre reinforced epoxy composites

The finding revealed that the dry treated composite showed a higher tensile strength as compared to dry untreated composite. It was noticed that treated fibre composites increased the tensile strength under a dry condition. The tensile strengths of dry T4 and T5 specimens were 31.23 MPa and 33.51 MPa, while for UT4 and UT5 were 20.88 MPa and 25.44 MPa, which was an increase of 49.57% for T4 and 31.72% for T5. The finding of this study on increasing the weight fraction of fibre loading and layering sequence on dry composite specimen lead to the increase in tensile strength

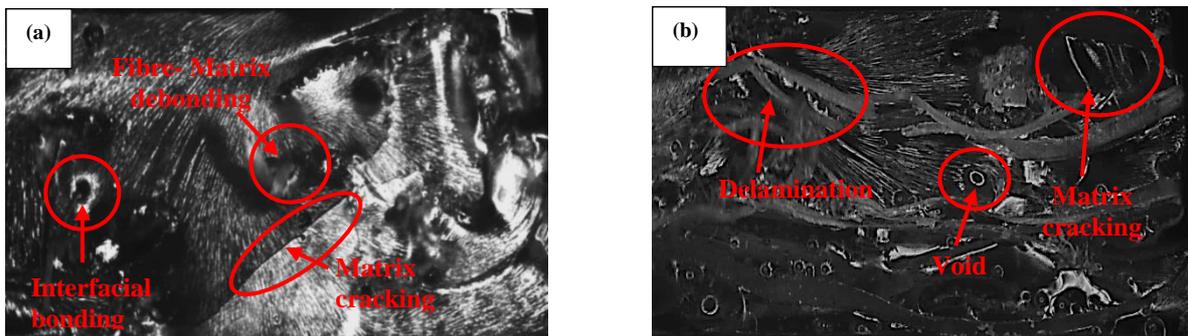
and tensile modulus. This was also because the alkali treatment had altered the surface properties of buri palm fibre which improved the interfacial bonding and mechanical interlocking between the treated fibre and epoxy matrix for better tensile properties [29, 30]. From the data, it is shown that the tensile properties for all the sample were reduced after the immersion process using the distilled water due the weaken of interfacial addition between fibre- matrix in the composite. The test results showed that the UT5 specimens has greatly decrease in tensile strength from 25.44 MPa and 25.37 MPa consecutively before and after the water immersion. However, the tensile strength dropped by 38.2% and 36% for layers T4 and T5 of the buri fibre composites, respectively. The data also revealed a significant decrease in the tensile strength of both conditions in a wet composite due to the degradation of composite. The finding was consistent with the findings of a past study by Dhakal et al. [18], in the tensile strength for three-layer and four-layer sequences of hemp fibre reinforced polyester composite which reduced by 38% and 15%, respectively as compared to dry composites. On the contrary, the tensile modulus result from all the samples has been observed to be increased after the immersion process. The tensile modulus percentage is higher with more number of layering sequence of untreated and treated buri palm fibre reinforced epoxy composite. Therefore, the soaking of fibre composites in the water had affected the strength properties. Generally, it is found that the tensile strength of higher fibre content immersed in the water had reduced. The reduction in strength was probably due to the inclusion of defects, such as pores and poor interface fibre-matrix bonding during the laminate fabrication process [28].

Surface Morphology of Wet Composite Specimens

The moisture surface characterisations of the tensile fractured composite specimens were studied through a microscopic analysis. The micrograph of untreated buri palm fibre reinforced epoxy composite specimens is illustrated in Figure 6. shows notable interface voids which appeared as a result of the poor contact between fibre and matrix in the fabrication process due to the fibre-matrix debonding during tensile loading. The presence of voids in buri palm fibre reinforced epoxy composites indicated a weak interfacial bonding between fibre-matrix adhesions. It had eventually affected the stress transfer from the matrix to buri. When the water absorbing composites reached a saturated level, the bound and free water remained in the composite as a reservoir, thus causing the fibre to swell. The voids had encouraged water absorption into the composite [33].

The water absorption in the polymer composite has effecting the decrease in mechanical properties by looking at the three mechanisms of diffusion of water molecules into the microgaps or voids between matrix chains, water molecules migrates into the flows and gaps at the fibre- matrix interface due to capillary action and the fibre swelled. It has also influenced the emergence of microcracking in the brittle epoxy matrix and contributed to higher volume of water which is absorbed into the interface [21, 31-32].

The water was actively assaulted by the interface and resulted in the debonding of fibre-matrix and fibre delamination, as plotted and clearly shown in Figure 6. However, the alkali treatment enhanced the external surface roughness of buri palm fibre by reducing the hydroxyl groups and providing a better interfacial bonding between hydrophilic fibre and hydrophobic polymer matrix. This study indicated that the treated fibre increased the degree of surface roughness as compared to untreated fibre based on the elimination of hemicellulose, lignin, and wax with NaOH concentration. Therefore, buri palm fibre became softened and helped the fibre-matrix to improve the interfacial bonding thereby increasing the tensile strength of the composite. Figure 7 shows the typical microscopic image whereby matrix cracking can be seen in the treated buri palm fibre and epoxy laminates. Similar morphological studies were conducted in previous research [36].



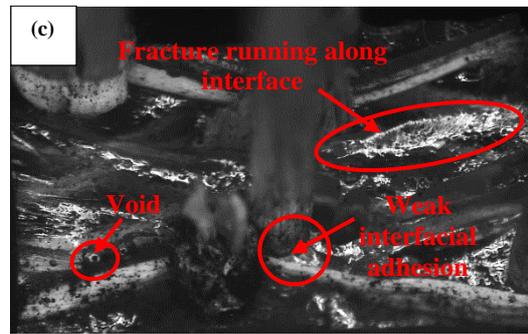


Figure 6. Tensile fracture of moisture untreated specimen under microscopic 2.0x magnifications: (a) and (b) UT 4 and (c) UT 5

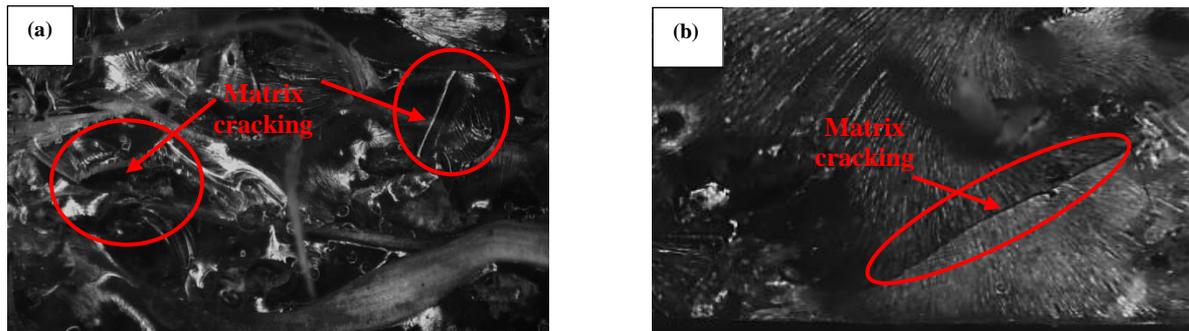


Figure 7. Tensile fracture of moisture treated specimen: (a) T4 and (b) T5

CONCLUSIONS

The purpose of this research was to study the effects of water absorption on the tensile strength of untreated and treated buri fibre composites with different stacking sequences (four-layer and five-layer) in distilled water at room temperature. Results showed that the UT4 and UT5 layer composites increased the water absorption and thickness swelling properties as compared to the treated buri palm fibre reinforced epoxy composites. The study findings indicated that stacking sequence and weight friction of fibre loading play a role in the rate of water absorption. Fibre composites exposed to moisture resulted in a significant decrease of tensile properties due to the fibre-matrix interface degradation. Fibre treatment in the composite increased the tensile strength. Dry specimens and treated fibres had higher tensile strength, which was measured for T4 (31.23 MPa) and T5 (33.51 MPa). In wet untreated or treated composites, the tensile properties showed a great decrease. From the morphological observation, the interfacial characteristic, fibre delamination, fibre debonding, matrix cracking, and voids were clearly shown. In conclusion, it is evident that this study has shown that alkali treatment provides a good improvement in the tensile properties of buri palm composite. Due to this significant improvement in tensile strength, buri palm fibre reinforced epoxy treated with alkaline could be used as the cheapest and lightweight material composite in the automotive, furniture, and building industries.

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