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The Effect of Alkaline Treatment on the Mechanical Properties of Treated Sugar Palm Yarn Fibre Reinforced Unsaturated Polyester Composites Reinforced with Different Fibre Loadings of Sugar Palm Fibre

(Kesan Rawatan Alkali terhadap Sifat Mekanikal Serabut Kabung Yarn Terawat Diperkuat dengan Komposit Poliester tak Tepu Diperkuat dengan Pembebanan Berbeza Serabut Kabung)

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ABSTRACT

The aim of this paper was to describe the effects of treated sugar palm yarn fibre loading on the mechanical properties of reinforced unsaturated polyester composites. Composites with varying fibre loads (10, 20, 30, 40 and 50 wt. %) were prepared using a hand-layup process. The composites were tested for tensile, flexural and impact strength according to ASTM D3930, ASTM D790 and ASTM D256 standards, respectively. The results showed that an increase in fibre loading of up to 30 wt. % increased tensile strength (31.27 MPa), tensile modulus (4.83 GPa), flexural strength (58.14 MPa) and modulus (4.48 GPa). Maximum loading can be attained at 40 wt. % of fibre loading for impact strength (38 kJ/m^2). The effectiveness of stress transfer mechanism through the fibre-matrix interaction, coupled with the optimization of fibre loading in resisting fracture and failure, boosts the overall mechanical performance of sugar palm composite.

Keywords: Alkaline treatment; fibre loadings; mechanical properties; sugar palm; unsaturated polyester

ABSTRAK

Matlamat kajian ini adalah untuk menerangkan kesan muatan serabut kabung terawat terhadap sifat mekanikal komposit poliester tak tepu diperkuat. Komposit dengan muatan serabut yang berbeza (10, 20, 30, 40 dan 50 % bt.) disediakan menggunakan proses layup tangan. Komposit ini diuji untuk kekuatan tegangan, lenturan dan hentaman mengikut piawai ASTM D3930, ASTM D790 dan ASTM D256. Analisis menunjukkan dengan peningkatan muatan serabut sehingga 30 % bt. akan meningkatkan kekuatan tegangan (31.27 MPa), modulus tegangan (4.83 GPa), kekuatan lenturan (58.14 MPa) dan modulus (4.48 GPa). Muatan maksimum dapat diperoleh pada 40 % bt. muatan serabut dengan kekuatan hentaman (38 kJ/m^2). Keberkesanan mekanisme pemindahan tekanan melalui interaksi matriks-serabut, digabungkan dengan pengoptimuman muatan serabut dalam rintangan retakan dan kerosakan, meningkatkan keseluruhan prestasi mekanikal untuk komposit kabung.

Kata kunci: Kabung; muatan serabut; poliester tak tepu; rawatan alkali; sifat mekanikal

INTRODUCTION

In recent years, the use of natural fibres as reinforcement in polymer composites for low-cost engineering materials has attracted much interest. The use of natural fibres as a potential replacement for synthetic fibres such as glass and carbon fibres in composites materials have intensified research recently due to environmental and economic factors. The primary advantages of natural fibres over synthetic fibres are their abundance and relatively low cost, low mass and specific density, high specific strength and renewability and biodegradability (Mohanty et al. 2002). In the meantime, the present use of the term ‘biodegradable’ in natural fibre composites actually refers to the utilization of natural sources in the polymer industry, which could reduce the dependence on petroleum resources and decrease industrial carbon emissions (CO_2) (Sahari et al. 2013).

There are several factors, including fibre length, fibre loading or volume fraction, fibre aspect ratio, fibre orientation and interfacial shear stress (IFSS), which affect the mechanical and thermal properties of composites (Nurazzi et al. 2017a). Furthermore, several studies have been conducted to determine the optimum natural fibre loadings in order to improve the physical, mechanical and thermal properties. However, according to Shalwan and Yousif (2013), there is no single estimate of fibre volume fraction and natural fibre loading universal value for which an optimum tensile strength could be achieved.

According to Bismarck et al. (2005), the presence of natural fibre has strongly influenced the performance of the composite as it changed the chemical structure. In general, increasing the fibre loading in the composites increases the composites stiffness and impact strength significantly (Bledzki et al. 2008). A study by Aji et al.

(2011) on kenaf reinforced unsaturated polyester showed that the tensile strength, strain and modulus were improved as the fibre volume fraction increases up to 45%, while an experimental study by Ramesh et al. (2014) reported that the 50% banana fibre reinforced epoxy composite shows the maximum tensile and flexural strength, while the maximum impact strength was held by the 60% of banana fibre reinforced epoxy composite. In addition, a study reported that the fabricated sisal leaf fibre reinforced polyester shows the maximum tensile strength and young modulus at 43 vol. % of the fibre loading (Sreekumar et al. 2007). In other words, for each fibre type, there is a type-specific optimum loading for exhibiting good tensile strength. This is strongly related to the chemical compositions of the fibres, interfacial adhesion towards the matrix used, environmental conditions and soil fertility factors for growth, among other factors (Shalwan & Yousif 2013). In this light, according to Thomason and Vlug (1996), high-performance composites can only be obtained with high fibre loading and if the reinforcing fibres in the final product have a sufficiently high aspect ratio (length/diameter).

Chemical treatments, such as alkaline treatment, are commonly used to improve the fibre strength. During an alkaline treatment, the hydrogen bonding in the network structure is disrupted and removed certain amounts of lignin, wax and oils covering the external surface of the cell fibre wall, increasing surface roughness, depolymerises cellulose and expose the short length crystallites (Li et al. 2007; Mohanty et al. 2001a). Alkaline treatment also resulted in an increase of amorphous cellulose content at the expense of crystalline cellulose (Garcia et al. 1998). As per Jähn et al. (2002), alkaline treatment influences cellulosic fibril, the degree of polymerization in the extraction of lignin and hemicellulosic compositions. Equation 1 describes a typical reaction of sodium hydroxide with natural fibre (Satyanarayana et al. 2009).

$$\text{Fibre} - \text{OH} + \text{NaOHà Fibre} - \text{O} - \text{Na} + \text{H}_2\text{O} \qquad (1)$$

Bachtiar et al. (2010, 2008) studied the effect of alkaline treatment on sugar palm fibre reinforced epoxy composites on tensile and flexural properties. The results showed that 0.25 M concentration of NaOH solution with 1 h soaking time has the highest tensile and flexural strength at 49.88 and 96.96 MPa, respectively.

In this work, sugar palm fibre was treated at this concentration before being yarned. The objective of this paper was to study the effect of treated sugar palm yarn fibre loading on the mechanical and thermal properties of reinforced unsaturated polyester composites. Hence, a comparison can be made from the previous research on the effect of untreated sugar palm yarn fibre loading reinforced unsaturated polyester composites.

Materials and Methods

Sugar palm fibre (L/D ratio 66.67) was obtained from Kampung Kuala Jempol, Negeri Sembilan, Malaysia.

TABLE 1. Chemical compositions of untreated and treated sugar palm fibre

Chemical constituents	Composition (%)	
	Treated	Untreated
Cellulose	42.52	47.74
Hemicellulose	12.74	5.96
Lignin	35.70	37.68
Others	9.04	8.62

The chemical constituents of sugar palm fibre are shown in Table 1 as determined using an in-house method of the Malaysia Agricultural Research and Development Institute (MARDI). Unsaturated polyester resin (UPE) (RTM grade, 40% styrene content, density of 1.025 g/cm^3), methyl ethyl ketone peroxide (MEKP) (Butanox-M50) as curing initiator and cobalt as reaction accelerator were supplied by CCP Composites Resins Malaysia Sdn. Bhd. Sodium hydroxide (NaOH) pellets was supplied by MERCK (M) Sdn Bhd. The bundles of sugar palm fibre were soaked in a 1% NaOH solution for 1 h. The treated sugar palm fibres were then washed several times with distilled water until obtaining pH 7. Subsequently, the fibres were dried in an oven at 60°C for 24 h. Then, a manual hand spinning machine from SDL ATLAS was used to make sugar palm yarn fibre with 2500tex. Figure 1 shows the sugar palm yarn fibre at 2500tex.

PREPARATION OF COMPOSITE

The sugar palm yarn fibre was horizontally placed in the closed steel mould with dimensions 160 × 120 × 3 mm. Initially, the 1% of MEKP as the initiator was mixed well with UPE resin, followed by mixing it with 0.2% of cobalt. Then, the mixed resin was poured over the fibre and compressed using hot press machine with 70°C and 80 bar for 30 min (Nurazzi et al. 2017b). A mould previously was sprayed with silicon mould release agent to avoid any sticking with the composites. Figure 1(d) shows the arrangement of yarn fibre in the mould.

CHARACTERIZATIONS

The tensile test was performed using Instron 3365 test machine according to ASTM D3039. The dimensions of the samples were 120 × 15 × 3 mm. The gauge length was 60 mm, with a crosshead speed of 5 mm/min applied for the test. The flexural test was performed using the three-point bending method using Instron 3365 test machine according to ASTM D790. The dimensions of the samples were 127 × 13 × 3 mm. The crosshead speed was set at 5 mm/min. An Izod impact test was performed using Instron CEAST 9050 testing machine with the capacity of pendulum 5.5 J according to ASTM D256. The dimensions of the unnotched samples were 65 × 10 × 3 mm. For each sample, seven to eight repetitions were performed and the average then reported.

FIGURE 1. (a) A bundle of sugar palm fibre after combing, (b) yarning process, (c) sugar palm yarn fibre with 2500tex and (d) composite

RESULTS AND DISCUSSION

TENSILE PROPERTIES

Table 2 shows the variation of tensile properties of treated sugar palm fibre reinforced unsaturated polyester composites. The total fibre loading of the composites varies from 10 to 50 wt. %. The tensile strength, tensile modulus and elongation at break linearly increase with an increase in the fibre loading in composites. The composite with 30 wt. % of fibre loading show the highest tensile strength properties among the composites. This might be because that upon reaching the 30 wt. % of fibre loading, the stress transfer mechanism can be effectively transferred uniformly along the fibre yarn parallel to the tensile stress direction (longitudinal).

However, the tensile properties will be decreased with a further increase in fibre loading up to 40 and 50 wt. %, respectively. The deterioration in tensile strength and modulus may be due to de-wetting effect. According to Ismail et al. (2008), the de-wetting effect may occur when the higher fibre loading tends to promote more fibre-fibre interactions as opposed to fibre-matrix interaction. Therefore, the wettability of the fibre by the matrix was significantly reduced (Sreekumar et al. 2007),

TABLE 2. Tensile properties of untreated (Nurazzi et al. 2017b) and treated fibre composite

Fibre loading (wt. %)	Tensile properties					
	Strength (MPa)		Modulus (GPa)		Elongation at break (%)	
	Treated	Untreated	Treated	Untreated	Treated	Untreated
10	19.08 (0.67)	19.12 (2.16)	3.78 (0.12)	3.16 (0.23)	0.66 (0.04)	0.80 (0.14)
20	23.92 (1.08)	29.30 (1.49)	4.72 (0.11)	3.28 (0.11)	0.97 (0.14)	1.13 (0.11)
30	31.27 (2.19)	40.91 (1.57)	4.83 (0.05)	4.43 (0.08)	1.28 (0.14)	1.39 (0.07)
40	28.18 (0.63)	38.00 (1.94)	4.58 (0.04)	4.01 (0.09)	1.22 (0.10)	1.65 (0.12)
50	27.68 (2.98)	36.73 (1.26)	4.30 (0.03)	3.90 (0.14)	1.11 (0.09)	1.73 (0.16)

( ) refer to standard deviation

resulting in poor stress transfer upon application of stress concentrations. Meanwhile, the incompatibility in the interfacial region between sugar palm fibre and unsaturated polyester increased with an increase in fibre loadings. This is due to the highly hydrophilic nature of sugar palm and the hydrophobic nature of unsaturated polyester matrix (Abdul Khalil et al. 2007). Finally, in a composite system, crack initiation and propagation will occur more easily at higher loadings (Sreekumar et al. 2007).

The enhancement of tensile modulus is attributed to the enhancement in the stiffness of the composites. According to Supri and Ismail (2011), the tensile modulus of the composite is dependent on the tensile modulus of the fibre itself. The higher tensile modulus of up to 30 wt. % fibre loading shows the successful restriction of the unsaturated polyester matrix from moving freely, thereby, increasing the rigidity of the composites (Pang et al. 2015). This finding is in good agreement with another study done by Santiagoo et al. (2011) where the tensile modulus increased as the fibre loading increased at a certain amount.

Meanwhile, for the elongation at break, the elongation was increased to up to 30 wt. % fibre loading was due to the rigidity of sugar palm fibre which contributes to the plasticity of the composites system (the elongation at break of sugar palm is 7.83% while UPE is 2.15%). The reduction of both tensile modulus and elongation at break of composites may be related to the stress transfer between the fibre-fibre interactions instead of fibre-matrix interaction.

The Rules of Mixtures (ROM) theory is a theory where the approximate composites modulus can be obtained from the ROM equation as follows:

$$E_c = \eta_0 E_f V_f + E_m V_m \qquad (2)$$

where η_0 refers to the Krenchel factor or efficiency factor and the value differs according to the fibre orientation. E_f and E_m refer to the modulus of fibre and matrix, respectively, while V_f and V_m refer to the volume fraction of fibre and matrix, respectively. According to Aziz and Ansell (2004), the η_0 for unidirectional fibre is equal to 1 and η_0 for randomly oriented fibre is equal to 0.25.

Figure 2 shows the theoretical values from ROM calculation versus tensile modulus from experimental findings. Figure 2 shows that there are differences between the theoretical and experimental values. The factors contributing to the lowered differences could be due to the fibre orientation during the composites preparation. As the action of manually lining up the fibres in a straight manner is almost impossible due to the nature of natural fibre (Aziz & Ansell 2004), the sugar palm fibres used were in coil form. These fibres were not perfectly aligned, causing alignment problems during the yarning process and composite manufacturing. Additionally, the deviation in fibre angles amounts to a maximum 5°-10° instead of straight alignment manifested as slight waviness (Sreekala et al. 2002; Sreekumar et al. 2007).

As calculated by using the theoretical calculation from ROM equation, the modulus values increased as the fibre loading increased compared to the experimental modulus values and decreased after the 40 wt. % fibre loading. In terms of the experimental value, the decreasing percentage of ROM value after the 40 wt. % of fibre loading is about 5.6% and 6.5% up to 50 wt. % of fibre loading. However, for the theoretical value, the increasing percentage after 40 wt. % is 54% and increased another 18% up to 50 wt. % of fibre loading. The differentiation of trend between the theoretical and experimental values of ROM was explained by Mallick (2007) and Mansor et al. (2013). Both studies reported that usually, the theoretical prediction will overestimate the value since the theoretical model does not take into account the effect of interfacial adhesion, dispersion and void content. This is because at very high fibre loadings, the processing is considered as difficult due to their poor compatibility and wettability which may lead to the decrementation of the properties (Aziz & Ansell 2004; Sreekala et al. 2002). Besides that, further packing defects may occur due to high fibre-fibre interaction instead of fibre-matrix interaction and finally may reduce the stress transfer effectively. This may become a noticeable factor and prediction that may contribute to the slight waviness of values from the theoretical and experimental values.

FLEXURAL PROPERTIES

The effect of treated sugar palm yarn fibre loading on flexural strength is shown in Figure 3. In this light, the

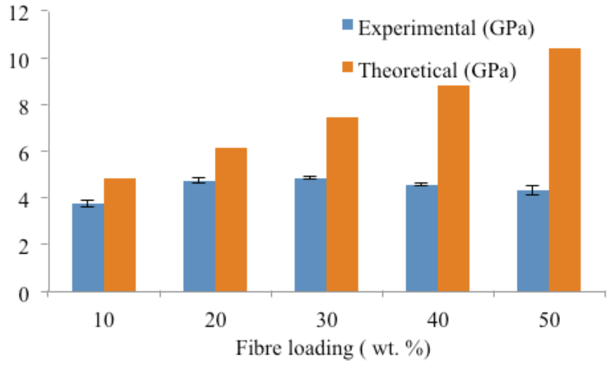

FIGURE 2. Comparison of tensile modulus form experimental value and theoretical value from Rules of Mixture (ROM)

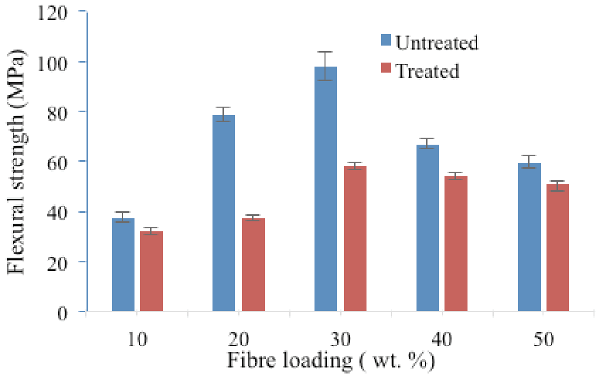

FIGURE 3. Flexural strength of untreated (Nurazzi et al. 2017b) and treated fibre composite

bending stresses can be taken up by sugar palm fibre loadings in different behaviours. From Figure 3, it can be inferred that the flexural strength and flexural modulus increase as the fibre loading increases in composites up to 30 wt. % about 58 and 4.5 GPa, respectively. The lowest flexural strength at 10 wt. % of fibre loading may be attributed to the lower loads transferred from the matrix to the fibres, resulting in a lower load carried by the fibres.

The significant increase in the flexural strength as the fibre loading increase is due to the increases in the bending stress which was transferred to the fibres as a result of the increase in the interfacial shear stress at the fibre-matrix interface (Rajesh et al. 2011). Besides that, the increases in the flexural strength with increases of fibre loading could be due to the ability of the cellulose fibre content to effectively resist the bending force (Alamri & Low 2012). However, due to the very high packing of fibre loading, the stress transfer from the matrix to the fibre becomes inefficient due to the high fibre-fibre interaction (Sreekala et al. 2002).

The flexural modulus versus fibre loading (wt. %) is shown in Figure 3. It demonstrates a trend similar to that of the flexural strength. As the fibre loading increased, the flexural modulus increased up to 30 wt. %. The addition of 30 wt. % of sugar palm yarn fibre loading has increased the flexural modulus by 82% and 57% from 10 to 20 wt. % of fibre loadings, respectively. These increases in certain mechanical properties of natural fibre reinforced polymer composites could be affected by the higher fibre loading due to the increases in fibre pull-out, fibre debonding and fibre bridging. This led to the increase in fibre-fibre interaction, rather than fibre-matrix interaction when the stress was transferred (Mishra et al. 2003). However, the flexural strength and modulus decreased after 30 to 40 wt. % about 6.7% and 10%, respectively.

IMPACT PROPERTIES

The effects of sugar palm yarn fibre reinforced unsaturated polyester composites impact strength at various fibre loadings are shown in Figure 4. According to Alamri and Low (2012), impact strength is an essential data that indicates the overall material toughness, where it is governed by the matrix-fibre interfacial bonding and the properties of matrix and fibres individually (Hatem & Meng 2012).

As depicted in Figure 4, the composite impact strength increased when the yarn fibre loading increased from 10 to 40 wt. %. This could be attributed to the improved toughness conferred by the sugar palm yarn in the composites. 40 wt. % of fibre loading composites which demonstrated the highest impact strength among the set tested. These values were caused by the sufficient presence of fibre in the composites in resisting fractures under high-speed applied stress. Thus, a 40% loading is the most effective loading of fibre composites to absorb energy (Abdul Khalil et al. 2007).

On the other hand, the composite exhibited a light reduction of about 2.7% impact strength compared to 40% loading composites with a fibre loading of over 40 wt. %. This may be due to the agglomeration of chain at high loading which resulted in higher fibre-fibre interaction and lower energy dissipation in resisting the sudden force and energy applied. According to Wambua et al. (2003), when the composites undergo a sudden force applied, the impact energy is dissipated by the combination of fibre pullouts, fibre fracture and matrix deformation. Moreover, according to Mishra et al. (2003), normally in fibre reinforced polymer composites, impact strength increase as fibre content increases due to an increase in fibre pull-out and fibre breakage.

In this work, the highest impact strength was imparted through the packed of sugar palm fibre yarn at 40 wt. % of fibre loading. The yarn involved intermingled individual fibre which enables the fibres to bridge with each other and the pack bundle yarn has helped to transfer the stress to the fibres effectively resulting in higher impact strength. Meanwhile, according to findings by Alamri and Low (2012), an increase in energy dissipation was observed due to an increase in fibre-fracture, fibre debonding, fibre pull-out, fibre-bridging and matrix deformation for composites loaded with higher fibre loading.

EFFECT OF ALKALINE TREATMENT

The result shows that the alkaline treatment has deteriorated the mechanical properties of the composite compared to

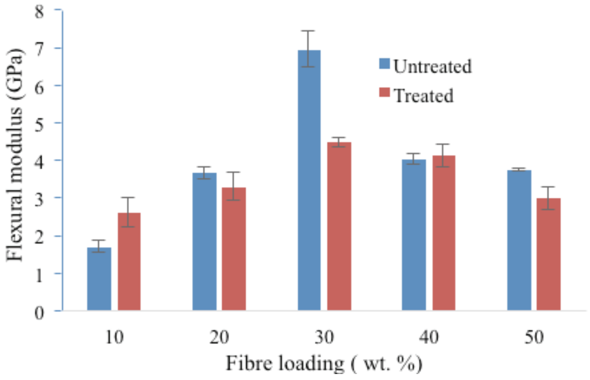

FIGURE 4. Flexural modulus of untreated (Nurazzi et al. 2017b) and treated composite

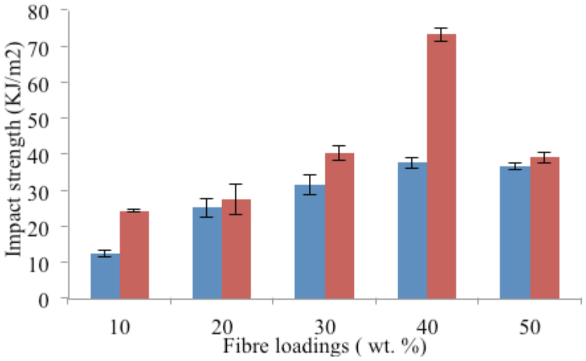

FIGURE 5. Impact properties of untreated (Nurazzi et al. 2017b) and treated fibre composite

the composite reinforced with untreated sugar palm yarn fibre. Theoretically, alkaline treatment is a process of fibre modification to reduce the hydrophilicity property of natural fibre which has increased the interfacial adhesion between the natural fibre and polymer matrix (hydrophobic property). Furthermore, according to Mohanty et al. (2001b), through the optimum alkaline treatment, hemicellulose is removed and the interfibrillar region is likely to be less dense and less rigid and thereby makes the fibrils more capable of rearranging themselves (fibrillation) along the direction of tensile force applied, removed certain amounts of lignin that bind the cellulosic structure, wax and oils that covering the external surface of the fibre cell wall.

This statement is aligned with the findings of Bachtiar et al. (2008). The study examined the effect of alkaline treatment on the tensile properties of sugar palm reinforced epoxy composites and reported that the highest tensile strength and modulus of the composite was shown by 1% or 0.25 M for 1 h soaking time in NaOH solution compared to other concentrations and soaking time. However, the result of this current study shows an opposite findings where the untreated composite has a much higher result compared to the treated composites. As shown in Table 1, the percentage of cellulose content of the treated fibre is lesser than the untreated fibre about 12.3%. The reduction of cellulose content may due to the polymeric chains in the microfibrils were broken. Hence, it is believed that the reduction of cellulose content will decrease the tensile strength of single fibres (Ishak et al. 2011). This is because cellulose is the main structural component which provides mechanical strength to the fibres (Reddy & Yang 2005).

Besides that, it is possible that the inconsistency of sources from the bulk of sugar palm supplies from the villagers have affected the performance of the fibres and finally to the composites. In this light, as reported by a pilot plant study done by Ishak et al. (2011) on the strength of a single sugar palm fibre, sugars palm fibre harvested from different heights (1 to 15 m) will have different strengths. The study found that degradation may alter the chemical compositions of fibres since the fibres are obtained from the matured sugar palm tree. Thus, the fibre may undergo several kinds of degradations especially at the bottom part of the tree.

Ishak et al. (2011) also concluded that the properties of sugar palm fibre are not dependant on the positions of fibres obtained, but they also depend on the condition of the fibre. This is because the fibres harvested from green palm fronds are considered to be at the matured stage and have the optimum cellulose content, providing maximum fibre strength. Meanwhile, fibres obtained from the upper parts of a tree are assumed to be at the juvenile stage where they are still in the growing process. They have poor dimensional stability and as a result, a lower cellulose content. For fibres obtained from a matured tree (tree is in the flowering stage), portions of dead palm fronds are often seen and it is assumed that the fibres are weak when they are degraded biologically.

CONCLUSION

The mechanical properties of composites increased with the increases in fibre loading. The highest tensile strength, tensile modulus, flexural strength and flexural modulus were observed at 30 wt. % of fibre loading, while superior impact strength was observed at 40 wt. % of fibre loading. This result could be due to the high effectiveness of stress transfer mechanism through the fibre-matrix interaction compared to fibre-fibre interaction, interfacial shear stress between fibre and matrix, fibre packing and optimization of fibre loading in resisting fractures and failures. The reduction of the cellulose content after the alkaline treatment is associated with the main structural components that provide mechanical strength to the fibre structure, and this has affected the final mechanical properties of the composites. The possibilities of the harvesting sugar from different tree heights might cause the inconsistency in the properties of the sugar palm supplies.

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