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Curing behaviour of unsaturated polyester resin and interfacial shear stress of sugar palm fibre

N. Mohd Nurazzi[1], A. Khalina[1,2*], S. M. Sapuan[1,2], A. M. Dayang Laila[1] and M. Rahmah[3]

[1]Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
[2]Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
[3]Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM), 40450 Shah Alam, Selangor, Malaysia
* Email: khalina.upm@gmail.com
Phone: +60389466420

ABSTRACT

Studies on the effect of cobalt of unsaturated polyester resin and the effect of treated sugar palm fibre with sodium hydroxide on single fibre strength and interfacial shear strength (IFSS) are presented in this paper. 1% of methyl ethyl ketone peroxide was used as the initiator, while cobalt of variable percentages (0.05%, 0.1%, 0.2%, 0.4%, 0.6%, 0.8% and 1%) was used as the hardener. The effects on glass transition and exothermic reaction of unsaturated polyester were studied for post curing temperature determination using differential scanning calorimetry by heating the samples at 10ºC/min heating rate from 30ºC to 120ºC with flowing of purge nitrogen gas atmosphere. For the single fibre test and IFSS, the treatment was carried out using sodium hydroxide solution with 1% concentration for one hour soaking time. Based on the optimisation percentage of cobalt, it was found that the higher the percentage of cobalt, the faster the sample tested to gel and cured. Treated sugar palm fibre exhibited better single fibre strength and IFSS between the matrices compared to untreated fibre due to the effectiveness of the alkali treatment. This can be attributed to the rearrangement of fibrils along the direction of tensile force and the removal of the coating layer and impurities after the alkaline treatment.

Keywords: Unsaturated polyester; sugar palm; curing characteristic; IFSS.

INTRODUCTION

Unsaturated polyester resins are an important class of high-performance engineering polymers used in numerous applications primarily in compression moulding (sheet moulding compounds), injection moulding (bulk moulding compounds), resin transfer moulding (RTM), pultrusion, filament winding and hand lay-up process [1]. 85% of the fibre reinforced polymer (FRP) products such as boats, car and aircraft components and chairs were manufactured using polyesters [2]. To produce a composite product, the determination of gel time and curing time is a very important stage in the processing of unsaturated polyester resins. In order to achieve a good quality product, the curing reaction should occur in a controllable way [3]. Other than that, it is also important to take

precautionary steps, especially in the estimation of mixing time; from the start of the initial mix until the resin is fully injected into the mould. This step is crucial to determine the right formulation mix of hardener required to optimise the time needed before the resin gels and hardens. This is especially vital when RTM or any other complex shape is to be employed for future application, as resin must be in working fluidic flow and unwanted fast cure must be observed in the equipment. Besides, the curing reaction is a very complicated process that is affected by many different things, such as weather, humidity, storage conditions and so on [4]. Cook et al. [5] stated that, decreasing gel time in a reciprocal fashion and increased rate of polymerisation of an unsaturated polyester resin may be due to increasing concentrations of initiator (either MEKP or acetyl acetone peroxide)

Cellulosic fibres are abundantly available in nature from a variety of plant species. Cellulose is the main component of natural fibres, however, the amount of pure cellulose, hemicellulose, pectin, lignin and other extractives will vary from fibre to fibre. Nowadays, the most prominent natural fibres applied in research and used commercially are flax, kenaf, hemp, bamboo, pineapple leaf fibre (PALF), cotton, ramie, sisal, coir, and sugar palm due to their promising fibre strength. Faruk et al. [6] listed the chemical compositions of cellulose, hemicellulose, lignin and wax content as having some common natural fibres presence. The interphase properties of the natural fibre reinforced polymer composites may exhibit very different mechanical performance and environmental ageing resistance. The interphase of fibre is a major consideration to facilitate the transfer of stress from fibre to fibre across the matric [7, 8]. Since there is a limitation of interface issue between the natural fibres (hydrophilic) and the polymer matric (hydrophobic), the interfacial bonding and interaction between the reinforcing fibres and the resin have become important elements to be considered in the manufacturing of composites [9, 10]. As an alternative, the surface modification of the fibres by chemical treatment has become one of the main interests of researchers nowadays. Instead of listing alkaline treatment with sodium hydroxide (NaOH), Sreekala et al. [11] listed the present modification methods for natural fibre such as acetylation, peroxide and permanganate, and isocyanate and acrylation treatments. These modification methods may give certain contributions either on structural, tensile strength and modulus, strength flexural and modulus and thermal properties [12-15]. The structural performance of natural fibre composites after the modification of fibres either increases or decreases, hence, the understanding and revelation of the morphology of fibre are of great importance as references [16, 17].

The adhesion between the fibre and matric is a major factor in determining the interface strength under stress. Therefore, micromechanical techniques such as fibre pull-out, single fibre push out and microdroplet test are extensively used to evaluate and quantify the interfacial bonding behaviour and the IFSS [18]. However, the microdroplet testing developed by Miller et al. [19] became one of the widely used single fibre-matric interfacial bond test methods to determine by Miller et al. [19]. Hence, the IFSS properties results can be computed using Eq. (1) [20-22], where τ is the interfacial shear strength (MPa), F is the load at maximum stress (N), D is the single fibre diameter in metre unit and L is the embedded length of droplet resin (m).

$$\textit{Formula of IFSS, } \tau = F/\pi DL \tag{1}$$

According to Kang et al. [23], the IFSS equation is only derived for differential cylindrical model which consists of a single fibre and surrounded by polymer resins. The sugar palm tree, *Arenga pinnata,* comes from a forest plant that can be found abundantly

in Southeast Asian countries such as Malaysia and Indonesia. It has been the topic of many research as a reinforcement in various polymer composites either in the form of short, long, random orientation and also in other types of fibre treatments [24-38]. Bachtiar et al. [[39, 40] treated sugar palm with various concentrations of NaOH and different soaking times. They found that, 0.25M solution with one hour soaking time to be the optimised concentrations and time, respectively, for tensile strength and flexural modulus, while 0.5M solution with four hours soaking time to be the optimised concentrations and time, respectively, for tensile modulus and flexural modulus. In this paper, studies on the gel and curing behaviours of unsaturated polyester using different percentages of cobalt as hardener were conducted. The structural analysis of untreated and treated sugar palm fibres, the strength of the single fibre and IFSS of sugar palm fibre embedded with unsaturated polyester resin were also investigated.

METHODS AND MATERIALS

Materials

The sugar palm fibre was obtained from Kampung Kuala Jempol, Negeri Sembilan, Malaysia. The unsaturated polyester resin (UPE)(RTM grad-40% of styrene content, density of 1.025g/cm^3), methyl ethyl ketone peroxide (MEKP) (Butanox-M50) as an initiator and cobaltnapthanate as an accelerator, Cobaltnapthanate is a product of CCP Composites Resins Malaysia Sdn. Bhd., while NaOH is by MERCK (M) Sdn. Bhd. Sodium hydroxide (NaOH) pallets was supplied by MERCK (M) Sdn. Bhd.

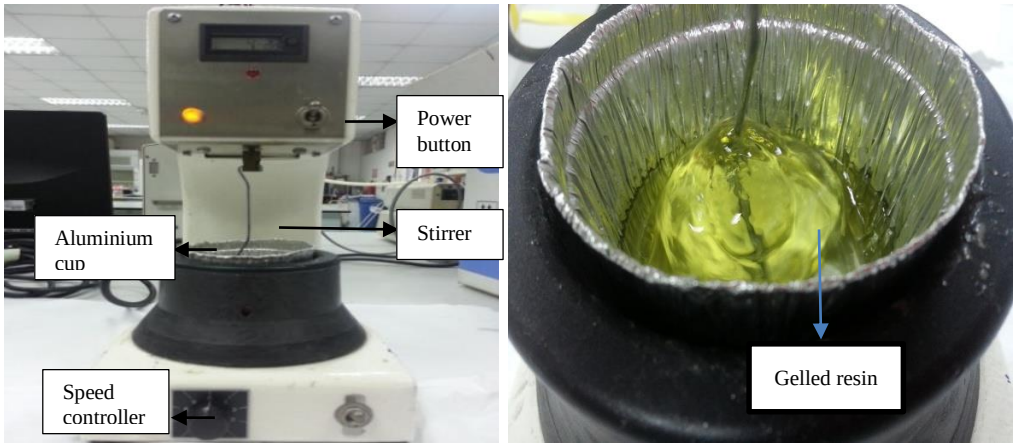


Figure 1. (a) Gel timer machine, and (b) UPE starts to gel after a certain processing time.

Treatment of Sugar Palm Fibre

The fibres were soaked in 1%/0.25M of sodium hydroxide (NaOH) alkaline solutions for one hour. Then, the treated fibre was washed with distilled water until it reached pH7 and dried at a temperature of 60ºC for 24 hours in the oven.

Determination of Gel and Curing Time

The purposes of the analysis are to get the optimum processing time and to estimate the changes of curing mixing resin with variable amount of cobalt loading and time. The gel time and curing time of UPE were investigated using a gel timer machine (Gardcomodel).

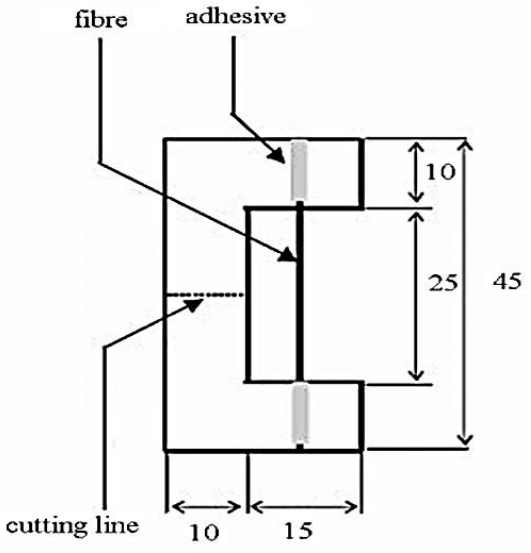
The curing time of UPE was analysed by physical observation under room temperature. The UPE resin was mixed with a constant percentage of MEKP (1%) and different percentages of cobalt (0%, 0.05%, 0.1%, 0.2%, 0.3%, 0.4%, 0.6%, 0.8% and 1.0%). The mixed resin (UPE:MEKP:Cobalt) was stirred at a speed of 0.05ms^{-1} until it turned to gel (Figure 1).

Determination of Tg of Unsaturated Polyester Resin

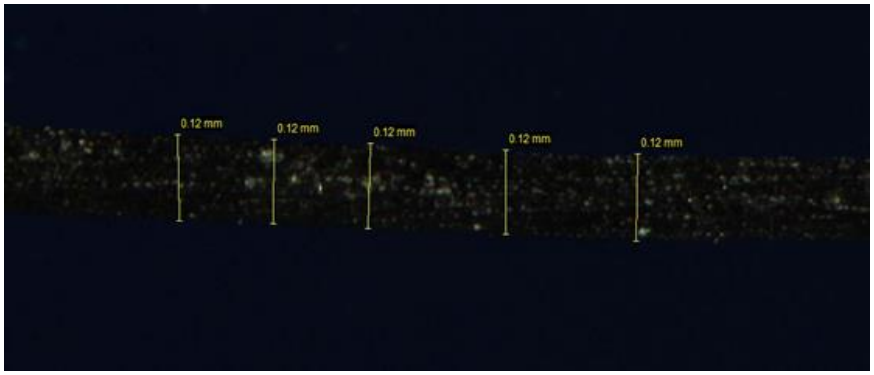
The glass transition (T_g) and change of exothermic reaction of unsaturated polyester resins upon the addition of different percentages of cobalt was analysed using DuPont DSC 7 by Perkin Elmer. 8mg to 10mg of samples were weighted and crimped in an aluminium pan. The samples were heated from 30ºC to 120ºC at a rate of 10ºC/ with flowing of purge nitrogen gas atmosphere.

FTIR Analysis of Sugar Palm Fibre

The structural changes of sugar palm fibre were analysed using Perkin Elmer Fourier Transform Infrared (FTIR) spectrometer. The FTIR test was run using transmission mode in the range of 4000cm^{-1} to 600cm^{-1} with 16 scans. The fibre particles were mixed with potassium bromide (KBr) and pressed into a small disc of about 1mm thick [41].



(a)



(b)

Figure 2. (a) Schematic diagram of specimens for single fibre test mounted on the cardboard [26] and (b) image of diameters measured using optical microscope.

Single Fibre Test of Sugar Palm Fibre

A single fibre tensile test was conducted in accordance with ASTM D3379 for single fibres. Single fibres from each untreated and treated sugar palm fibres were attached to a cardboard frame as shown in Figure 2. The fibres were mounted and glued on the tab, designed with a gauge length of 20mm, and the diameters were measured using an optical microscope model Olympus SZX12. The specimens were tested using universal testing machine model Instron 3366 with 5kN load capacity with a crosshead speed of 1mm/min. For each sample, five repetitions were performed and the average of the five tests was reported. The surface morphology of untreated and treated sugar palm fibres were examined using Polarise Optical Microscope (POM).

Microdroplet test

A microdroplet test was carried out to determine the interfacial adhesion or IFSS characteristics between untreated and treated sugar palm fibres embedded with unsaturated polyester resins. The fibres were mounted and glued on the tab which was designed with a gauge length of 20mm. The diameters of the single fibres (D) and embedded length of the droplets (L) were measured using an optical microscope model Olympus SZX12. The microdroplet test was done by firstly applying a resin drop onto the surface of a single fibre, curing the fibre-resin to form the droplet and then applying a shearing force to pull the fibre out of the droplet. The schematic diagram of the microdroplet test is shown in Figure 3. The analysis of IFSS was performed using Eq. (1). The specimens were then tested with 5 kN load capacity with a cross head speed of 1mm/min using the universal testing machine model Instron 3366. For each sample, five repetitions were performed and the average of the five tests was reported.

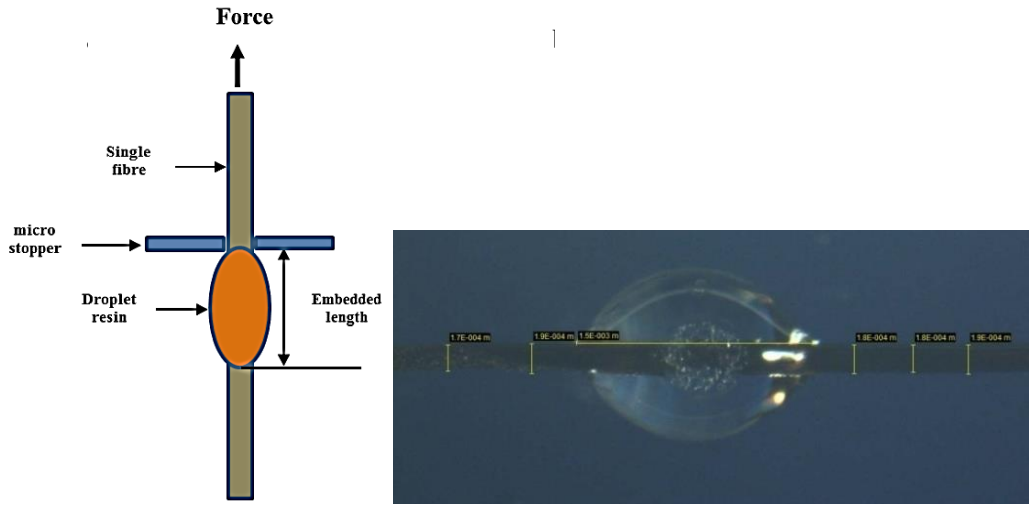


Figure 3. (a) Schematic diagram of a micro droplet [22], (b) Current studies of unsaturated polyester microdroplets adhered on a single fibre under microscope.

RESULTS AND DISCUSSION

Gel Time and Curing Time Analysis

Figure 4 shows the effect of the addition of cobalt content between 0.05% and 1% on gel time and curing time of polyester resins. A downward trend of the gel time and curing time was observed. The patent showed that as the percentage of cobalt increased, both the

gel time and curing time decreased. Initially, without the addition of the hardener, the unsaturated polyester recorded curing time was about 50 minutes. After the addition of 0.05% of cobalt, the gel time decreased 56%, which was from 50 to 32 minutes. As the MEKP became a reactive compound, the peroxide-free radical of MEKP started the polymerisation reaction by attacking the styrene that was present in the resin, which allowed the formation to cross-link with another polyester chain [42].

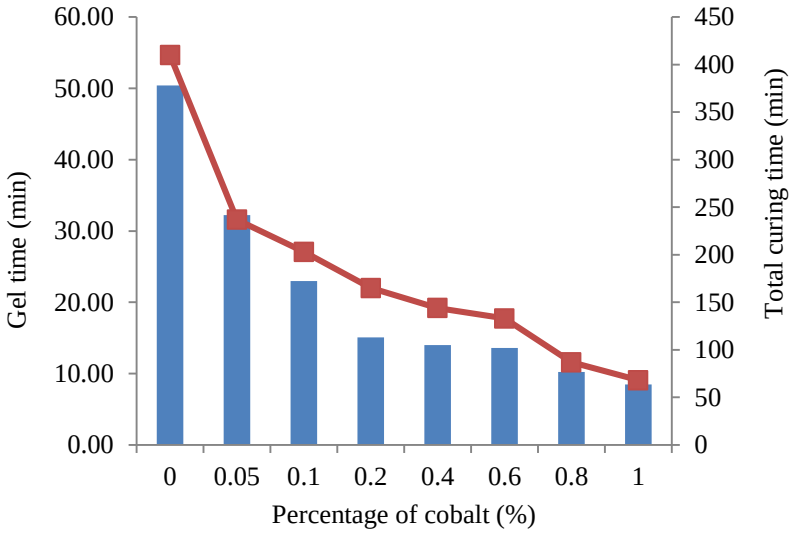


Figure 4. Effect of gel time and curing time of unsaturated polyester.

Initially, the gelation rate was determined by MEKP, which initiated polymerisation and cross-link. With the addition of cobalt as the accelerator, the polymerisation started to cross-link faster [43]. However, the effect of cobalt was limited to the increase in rate only, not the number of cross-links. The reactions occurred at room temperature, but the exothermic reaction took place upon curing. According to Bharat et al. [43], the processing temperature and the amount of catalyst can control the rate of polymerisation. The higher the temperature, or the more the catalyst, the faster the reaction will be. According to Nabil et al. [44], the processing of fabrication cost may be reduced with a shorter curing time. The determination of gelation and curing time are important in the manufacturing of composites using the RTM. Hence, sufficient processing time can be estimated starting from the mixing of UPE:MEKP:cobalt, the flowing in the tubing, as well as the time needed for the resin to penetrate into the fibre before it becomes a gel and a fully cured composite product.

Glass Transition and Exothermic Reaction of Unsaturated Polyester Resin

The glass transition (Tg) indicates material changes from glass or amorphous into a rubbery state. Table 1 and the DSC thermogram in Figure 5 show that the resin is not fully cured. A secondary heating was needed to fully cure the resin. Partially cured resin was observed with exothermic peak and glass transition of precured resin. It is clearly shown that there a few differences of Tg with different loadings of cobalt. The Tg values for all samples were identified to be around 68°C to 73°C. The curing temperature was around 68ºC to 73ºC. The lowest curing temperature was indicated by the 0.6% of cobalt (blue thermogram), while the highest was by the 0.8% of cobalt (red thermogram). The uncured or partially cured resin was observed with exothermic enthalpies. The highest

exothermic was observed on the control sample (without cobalt) with the highest enthalpy value of 2.39J/g. The high amount of exothermic corresponded to the greater amount of uncured resin.

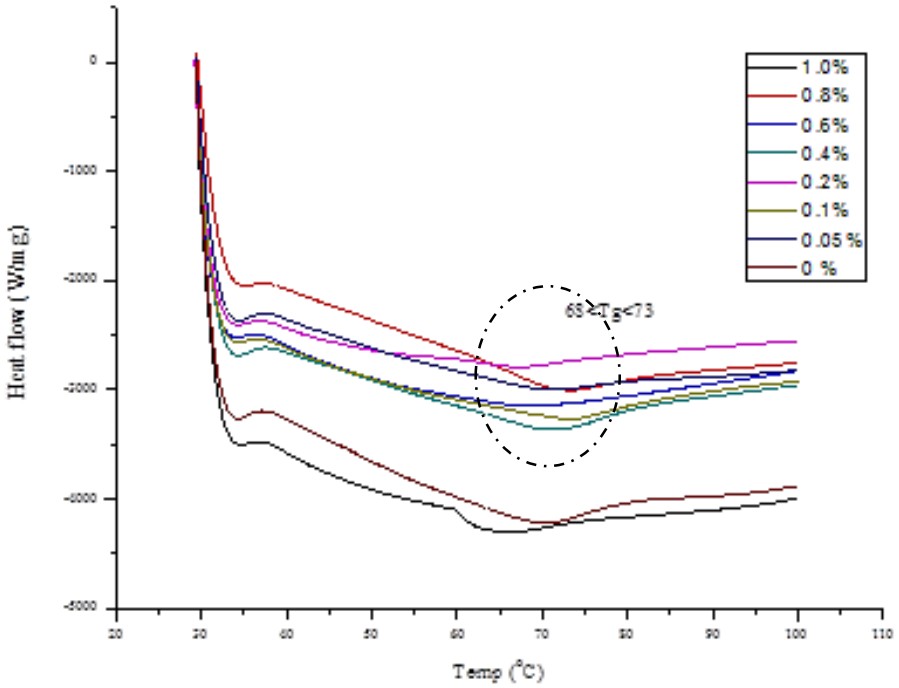


Figure 5. Glass transition analysis with different percentages of hardener.

Table 1. Effect of total curing time (min) on Tg with different percentages of cobalt.

	Samples							
Cobalt (%)	0	0.05	0.1	0.2	0.4	0.6	0.8	1.0
Tg (ºC)	70.2	71.3	72.9	73.1	72	68.8	73.4	71.8
Total curing time (min)	410	237	203	165	144	133	87	68

The rigidity of network UPE upon cross-linking was similar to the initiation site of cross-linking provided by the MEKP, which cross-linked with styrenic groups and unsaturated vinyl UPE. The addition of hardener did not affect the cross-link density of UPE; it only fastened the resin's gel time and curing time. The Tg values varied slightly around ~ 4°C for lowest Tg with 0.06% of cobalt and highest Tg with 0.02% of cobalt as shown in Table 1. The slight changes on some of the Tg values upon the addition of cobalt may be due to the decrease of mobility of the chains and the inability of the initiator to form a regular cross-linking network [45]. Therefore, from the results of the exothermic reaction and the formation of an unstable cross-linking which was still in the pre-cure stage, full curing was required for a complete cure. Hence, post-curing of the sample was done within the temperature of between 70°C to 80°C for at least 90 minutes instead of curing under room temperature, as previously done by Varma et al. [46]. In order for a complete cure to take place, the internal morphology and the cross-linked reaction of the UPE have to reach 99% or 100% for the UPE resin to transform from a rubbery state to an amorphous state.

Structural and Morphology Analysis of Sugar Palm Fibre

Figure 6 shows the FTIR spectra of chemical changes in sugar palm fibre before and after the treatment. It is well known that natural fibre mainly consists of cellulose, hemicellulose and lignin. Hemicellulose is composed of different sugars and other various substituents which are water soluble because it is easy to hydrolyse. Then, a part of lignin is soluble in alkali solution. Therefore, it was possible that a part of hemicellulose and lignin were dissolved and decreased during the alkali solution treatment [47]. The trend showed the changes, which were almost the same for the sugar palm fibre, before and after the treatment. The difference was only found in the slight shifting of the wave numbers. Firstly, the vibration peak, which was around 1760cm^{-1} to 1665cm^{-1} associated with carbonyl (C=O) groups of hemicellulose, was not clearly present even in the untreated sugar palm fibre. This may be due to a relatively low hemicellulose content in the sugar palm ranging between 4.71% to 7.93% [48, 49].

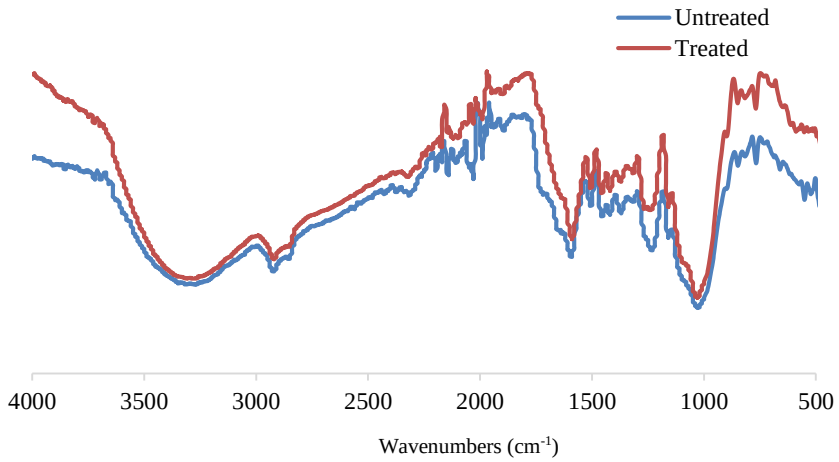


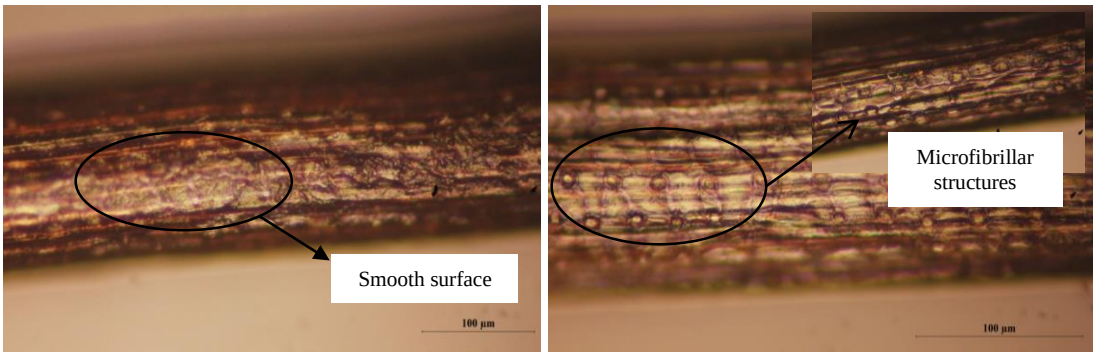
Figure 6. FTIR spectra of untreated and treated sugar palm fibres with 1% NaOH.

The peak intensity reduction found at around 1230cm^{-1} (C-O) of the acetyl group in lignin component for 1% and 2% of treated sugar palm fibre was associated with the mercerisation of lignocellulosic fibres [50]. Mercerisation removed the waxy layer, adhesive pectins and hemicelluloses that bind the fibre bundles to each other and to the pectin and hemicellulose rich sheats of the core [41]. The vibration peak at around 1580cm^{-1} to 1590cm^{-1} (C-C) associated with the benzene ring of lignin reduced as the alkali treated concentration increased. In addition, the vibration peak of C-H stretched at 2900cm^{-1} in cellulose and hemicellulose that was present in the untreated fibre became weak, indicating that the part of the hemicellulose was removed [47]. Besides cleaning and modifying the fibre surface, alkali treatment reduces the hydrogen bonding due to the removal of the hydroxyl groups (-OH) by reacting with sodium hydroxide [51, 52]. The hydroxyl groups are also involved in hydrogen bonding with the carboxyl groups, perhaps also with the fatty acids that are available on the fibre surface of sugar palm fibres. Thus, for this test, it was indicated by the reduction and broadening of the peaks around 3200cm^{-1}. Figure 7 shows a typical reaction of sodium hydroxide with a natural fibre [53].

$$\text{Fibre-OH} + \text{NaOH} \longrightarrow \text{Fibre-O}^{-}\text{Na}^{+} + \text{H}_2\text{O} + \text{surface impurities}$$

Figure 7. Chemical reaction of natural fibre with NaOH solution.

Figure 8 shows the morphological surface of sugar palm fibre before and after it was soaked in 1% of NaOH for one hour. Based on observations of its microstructure, there was a strong evidence that NaOH treatment changed the physical surface appearance of the sugar palm fibre compared to the untreated sugar palm fibre. It showed that the physical changes with slight fibrillation can be clearly observed on the outer surface of the fibre and it was due to the removal of the waxy layer on the outer surface (Figure 8(b)).



(a) (b)

Figure 8. (a) Surface morphology of untreated and (b) treated sugar palm fibres.

Moreover, the alkali treatment may lead to fibre fibrillation, which is the breaking down of fibre bundles into smaller fibres, that increases the effective surface available for contact with the matric. This resulted in the single fibre and IFSS properties embedded with polyester resin to increased tremendously after the treatment. This was also thought to be caused by the treated sugar palm fibres having good interfacial bonding with the matric, therefore, resulting in a larger mechanical interlocking force between the fibre and the matric [54, 55].

Single Fibre Strength of Sugar Palm Fibre

Table 2 shows the comparison of tensile strength, modulus and elongation of untreated 1% treated sugar palm fibre with some other fibres. It was reported by Bachtiar et al. [23], that the treated sugar palm reinforced epoxy composite using 1% or 0.25M of NaOH concentration, with one hour soaking time showed higher tensile strength compared to four hours and eight hours of soaking time [39]. The tensile strength of treated sugar palm fibre was approximately 110% greater compared to the value of untreated sugar palm fibre. This increase can be attributed to the removal of lignin and hemicellulose, which facilitated the rearrangement of fibrils along the direction of tensile deformation, hence caused higher tensile strength [56, 57].

In comparing the performance of sugar palm with other fibres on Table 2, the tensile strength of sugar palm showed moderate value and was almost similar with the strength of bamboo, kenaf and coir within the range of between 140MPa to 215.4MPa. On the other hand, the tensile strength of the treated sugar palm fibre was 332.28MPa, which was higher than bagasse, bamboo, kenaf and coir fibres and was almost similar to flax and jute. It means that there is a potential of using sugar palm fibres as reinforcement to polymer composites research and applications. The tensile modulus of treated sugar palm fibre increased from 4.96GPa to 17.27GPa. The improvement of modulus may indicate an improvement in the crystallinity index of cellulose due to the removal of the hemicellulose and lignin content, leading to better packing of cellulose chains, and therefore, causing a decrease in the spiral angle and an increase in the degree of molecular

orientation. In other words, the fibres become relatively ductile after the removal of some hemicellulose and lignin content which results in the improvement of stiffness due to the increase in crystallinity of hard cellulose [58, 59].

Table 2. Comparison of mechanical properties of untreated and 1% treated sugar palm with other fibres [60-63].

Fibres	Density (g/cm^3)	Tensile strength (MPa)	Elongation at break (%)	Tensile Modulus (GPa)
*Sugar Palm**	*1.292*	*156.96*	*7.98*	*4.96*
*Treated sugar palm (1% NaOH)**	*1.193*	*332.28*	*5.3*	*17.27*
Bagasse	1.5	290	-	17
Bamboo	1.25	140-230	-	11-17
Flax	0.6-1.1	345-1035	2.7-3.2	27.6
Hemp	1.48	690	1.6-4	70
Jute	1.3	393-773	1.5-1.8	26.5
Kenaf	1.45	215.4	1.6	53
Sisal	1.5	511-535	2.0-2.5	9.4-22
Ramie	1.5	560	2.5-3.8	24.5
Pineapple	0.8-1.6	400-627	14.5	1.44
Coir	1.2	138.7	30	4-6
E-Glass	2.5	2000-3500	0.5	70
S-Glass	2.5	4570	2.8	86
Aramid	1.4	3000-3150	3.3-3.7	63.0-67.0

Note: * indicates the current study done by researcher.

Interfacial Shear Strength (IFSS) of Sugar Palm Fibre with Unsaturated Polyester Resin

Figure 9 demonstrates the effect of alkaline treatment on the IFSS of sugar palm fibre. The IFSS showed an increase after the 1% alkaline treatment (from 2.59 MPa to 3.58 MPa). Recently published studies done by [64-67] showed that the chemical treatments may enhance the mechanical properties of the treated fibres as compared to the untreated fibres. The increase in the IFSS of the treated single fibre compared to the untreated fibre was attributed to the removal of impurities and waxy substances from the fibre surface and the creation of a rougher surface after the modification. Alkali treatment cleaned the fibres' surface from impurities, which in turn increased the disruption of the moisture absorption process by removing the coating of the -OH groups in the fibre, thus, increased interface quality. Conclusively, the waxy layer contributes towards ineffective fibre matric bonding and poor surface wet out. The enhancement in the IFSS of the treated sugar palm fibre after NaOH treatment in the studies may be attributed to the phenomenon called fibrillation. Untreated fibres are packed together in a bundle. After the fibre treatment, the packed alignments of the fibres are broken into smaller ones through the dissolution of hemicellulose. This phenomenon increases the effectiveness of the surface area available for contact and matric penetration inside the fibre cell, thus improving interfacial adhesion [68, 69]. However, Bachtiar et al. [39] reported that tensile strength is expected to improve when the alkali concentration is raised, but from the experiment, high concentration after 0.25M of NaOH may decrease the strength due to fibre structure damage and consequently reduce the tensile strength of the fibre [39].

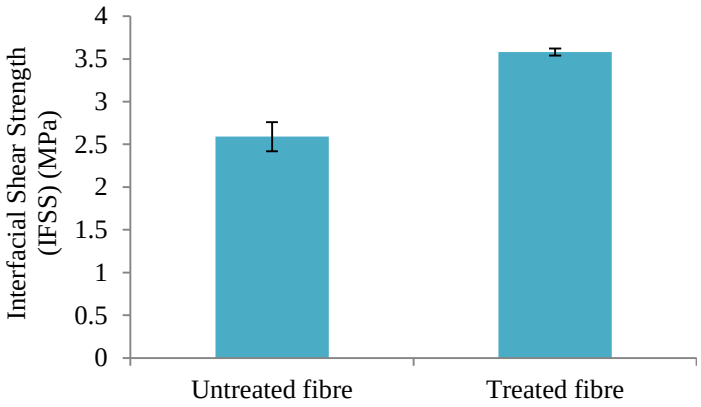


Figure 9. IFFS of untreated and treated sugar palm fibres.

CONCLUSIONS

In conclusion, other than using the MEKP as the initiator, cobalt as the hardener did assist in the formation of the crosslinking process as the curing time proportionally increased with the increase in the amount of hardener. Cure time was halved when 0.02% of cobalt solution was used. It was observed that the Tg varied slightly around ~4°C which revealed that the cross-linked structure density and resin rigidity were not structurally affected as shown by the DSC thermogram. Post curing of unsaturated polyester was needed to complete the crosslinking formation even after 24 hours of curing time under room temperature, as a result of high exothermic peaks for some compositions of the hardener. The differences in structural features were found to be affected by the treated and untreated sugar palm fibres. Alkali treatment using NaOH led to the enhancement in IFSS due to internal morphological changes of sugar palm fibres.

ACKNOWLEDGEMENTS

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