

Effect of Alkaline Treatment on Structural Characterisation, Thermal Degradation and Water Absorption Ability of Coir Fibre Polymer Composites

(Kesan Rawatan Alkali terhadap Pencirian Struktur, Degradasi Termal dan Keupayaan Serapan Air bagi Komposit Polimer Serabut Sabut Kelapa)

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ABSTRACT

The alkaline treatment with 5 wt. % sodium hydroxide (NaOH) solution at room temperature for 24 and 48 h was performed on coir fibre. The structural characterisation, thermal degradation and water absorption ability of the untreated and NaOH-treated coir fibre polymer composites have been studied. Scanning electron microscope (SEM) images showed that coir fibres treated with NaOH have rough surface texture and the roughness of the fibre surface becomes significant as the duration of the NaOH treatment increased. Fourier transform infrared (FTIR) spectra confirmed that NaOH treatment removed hemicelluloses as evidenced by the absence of absorption bands at 1724.36 cm⁻¹ and changes the absorption intensity at bands 1244.09 cm⁻¹ and 1249.87 cm⁻¹ due to the loss of lignin. NaOH-treated coir fibre composites demonstrated better thermal stability at low temperature degradation. At high temperature, the thermal stability was reduced due to the decreased of residual lignin content. The water absorption of the NaOH-treated coir fibre composites was lower than untreated coir composite contributed by better interfacial adhesion between the NaOH-treated coir fibre to epoxy resin.

Keywords: Coir fibre; NaOH; polymer

ABSTRAK

Rawatan alkali dengan kepekatan 5 % bt. larutan natrium hidroksida (NaOH) pada suhu bilik selama 24 dan 48 jam telah dibuat terhadap serabut kelapa. Pencirian struktur, degradasi termal dan keupayaan serapan air terhadap komposit polimer serabut kelapa tidak dirawat dan serabut kelapa dirawat-NaOH telah dikaji. Imej daripada mikroskop elektron imbasan (SEM) menunjukkan serabut kelapa dirawat-NaOH mempunyai permukaan yang kasar dan kekesatan permukaan serabut kelapa menjadi ketara dengan penambahan tempoh rawatan alkali. Spektra transformasi Fourier inframerah (FTIR) mengesahkan bahawa rawatan NaOH menyingkirkan kandungan hemiselulosa yang dibuktikan oleh ketiadaan jalur 1724.36 cm⁻¹ serta mengubah keamatan jalur 1244.09 dan 1249.87 cm⁻¹ yang disebabkan oleh kehilangan lignin. Komposit polimer serabut kelapa dirawat-NaOH menunjukkan kestabilan termal pada degradasi suhu rendah. Pada degradasi suhu yang tinggi, kestabilan termal berkurang disebabkan oleh penurunan kandungan sisa lignin. Serapan air oleh komposit polimer serabut kelapa dirawat-NaOH adalah lebih rendah daripada komposit polimer serabut kelapa tidak dirawat disebabkan oleh lekatan yang lebih baik antara serabut kelapa terawat dan matriks polimer.

Kata kunci: NaOH; polimer; serabut serabut kelapa

INTRODUCTION

In recent years, there has been intensive use of natural fibres as alternative reinforcement filler in the polymers as it provides the benefits of being cost effective, biodegradability, strong, low density and light weight composites (Al-Oqla et al. 2015; Lee et al. 2014; Li et al. 2007; Pickering et al. 2016; Raghavendra et al. 2014; Summerscales et al. 2010).

Natural fibres such as coir, palm, flax, hemp, kenaf, and jute has gain extensive research interest (Akhtar et al. 2016; Amiri et al. 2015; Oushabi et al. 2017; Sair et al. 2018; Zhang et al. 2017). Among different types of natural fibres, coir fibre is widely used as it is abundantly available, cheap, and possesses good mechanical and thermal properties (Brígida et al. 2010; Haque et al.

2012; Zaman & Beg 2014). Thermoset and thermoplastic polymers are the most common matrices use in natural fibre composites as they are lightweight and can be processed at low temperature. The selection of the polymer matrix is normally limited by the temperature as natural fibres are thermally unstable above 200°C (Summerscales et al. 2010). Due to this limitation, thermosets which can be cured and thermoplastics that soften below this temperature are useable as a matrix (Pickering et al. 2016).

Natural fibres are lignocellulosic material composed of crystalline cellulose and amorphous non-cellulose constituents consisting of lignin, hemicelluloses and surface impurities such as waxes, pectin, and greases. These amorphous non-cellulose constituents bind and protect the inner structure of crystalline cellulose fibre. The crystalline

cellulose is responsible for assigning strength, stiffness, and dimensional stability of the natural fibres due to its crystalline structure whereas the amorphous non-cellulose constituents are rich in hydroxyl groups that contribute to its hydrophilic nature and prompt to absorb moisture (Costa et al. 2006). Due to the hydrophilic nature of natural fibres and hydrophobic nature of polymer matrices, low compatibility between the fibre-matrix leads to poor wettability of the natural fibres to the polymer matrices. Poor interfacial adhesion between the natural fibres and polymer matrices degrades the mechanical properties and the moisture resistance of the composites, thus limits the further application of natural fibre composites (Akhtar et al. 2016; Punyamurthy et al. 2017; Qian et al. 2015; Zhang et al. 2018).

Surface modification using chemical treatments is usually performed on natural fibres to change its hydrophilic nature, mainly to remove the hydroxyl groups (Akhtar et al. 2016; Kabir et al. 2012; Li et al. 2007). Alkaline treatment using sodium hydroxide (NaOH) is one of the most applied chemical treatments for natural fibres as it removes the hemicelluloses, lignin and surface impurities such as pectin, wax, and grease that are covering the internal cellulose components (Gomes et al. 2007). There have been numerous studies that investigate the effect of alkaline treatment on the natural fibres and the findings showed that alkaline treatment improves the compatibility of the natural fibres to the polymer matrix and results in good interfacial adhesion of the fibre-matrix. A study conducted by Amiri et al. (2015) showed that the alkaline treatment improved the fibre-to-matrix adhesion, as defined by increasing shear, tensile and flexural strength, tensile but this in turns decreased the tensile and flexural modulus. Similarly, Fiore et al. (2015) had reported that the alkaline treatment improves fibre-matrix interfacial adhesion which enhanced the mechanical properties of the kenaf fibres reinforced epoxy composites. Oushabi et al. (2017) had investigated the effect of alkaline treatment on the structural, mechanical and thermal properties of natural fibres and the results showed that the removal of the non-cellulose constituents inclusive of hemicelluloses and lignin promotes the tensile strength, interfacial adhesion of the fibre-matrix and thermal resistance of the natural fibres. Meanwhile, a technique that integrates the alkaline and microwave treatments had been introduced by Liu et al. (2018) and the findings showed that the microwave treatment significantly ruptured the natural fibre structure and promotes the alkaline solution penetration into the inner natural fibre structure that removes and separates hemicelluloses from cellulose.

It becomes crucial to determine the most feasible approach to remove the amorphous non-cellulose constituents by varying the concentration of the alkaline solution, the duration of the treatment or any integrated methods in order to remove the amorphous non-cellulose constituents without deteriorating the cellulose structure of the natural fibres. Thus, in this paper, alkaline treatment with 5 wt. % NaOH solution at room temperature for 24

and 48 h was performed on coir fibres. Epoxy resin was used as the polymer matrix as it has excellent mechanical properties, chemical resistance and electrical insulation (Zhang et al. 2018). The structural properties (SEM and FTIR analyses), thermal degradation and water absorption ability of the coir fibres composites was further investigated.

MATERIALS AND METHODS

RAW MATERIAL PREPARATION

Locally available raw coir fibres in Kuala Terengganu were collected and cleaned from the coconut shell and coco peat using distilled water. Thermoset Epocast PT100 based epoxy resin and Epoharden PT100S amine based epoxy hardener, were supplied by Portal Trading, Penang, Malaysia. Sodium hydroxide (NaOH) pellet with molecular weight of 40.00 g/mol was supplied by R&M Chemicals.

ALKALINE TREATMENT ON COIR FIBERS

Coir fibres were treated with NaOH solution that was prepared by adding 5 wt. % of NaOH into 95 wt. % of distilled water and soaked at room temperature for 24 and 48 h. The treated fibres were washed thoroughly with distilled water to neutralise NaOH and put under sunshades until completely dried. The treated fibres were kept in desiccator with drying agent to remove excess moisture.

COMPOSITE PREPARATION

Untreated and NaOH-treated coir fibres were pulverised into fine powder and used as filler in the polymer composites. The pulverised coir fibres were further sieved using 75 µm test sieve in order to minimize the agglomeration degree of coir fibre in the epoxy resin. The composites were prepared at room temperature by homogeneously dispersed the coir fibre filler in epoxy resin until thoroughly mixed. The composites were cured at room temperature (30~35°C) for approximately 1 to 2 h. The formulation of composites is shown in Table 1.

SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

The effect of NaOH treatment on the surface morphologies of coir fibres were studied using JEOL JSM-6360L scanning electron microscope (SEM). The specimens under test were prepared by lightly pressed the specimen on the carbon conductive adhesive double-sided tape that was mounted on the SEM stub. The specimens were sputter coated with a thin layer of platinum to avoid electrostatic charges during the imaging process.

FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY ANALYSIS

The effect of NaOH treatment on the chemical structure of coir fibres was analysed using Shimadzu/IRTracer-100.

TABLE 1. Composition weight percentage (wt. %)

Material	Polymer with untreated coir fibre (weight, g)	Polymer with NaOH-treated coir fibre (weight, g)
Epocast PT100	100	100
Epo Harden PT100s	50	50
Untreated Coir fibre	45	-
NaOH-treated Coir fibres	-	45

The FTIR spectra with percentage transmittance (%T) versus wavelength (cm^{-1}) were recorded within the scanning range of 650-4000 cm^{-1} .

THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermal stability of the polymer composites reinforced with untreated and NaOH-treated fibres were analysed through thermogravimetric analysis (TGA) curves using Mettler Toledo/TGADCS 1 analyser. Samples of 20 mg were scanned from 30 to 600°C. The heating rate temperature was 10°C/min with constant argon flow of 50 mL/min to avoid thermal oxidation.

WATER ABSORPTION TEST

The water absorption test of the polymer composites reinforced with untreated and NaOH-treated fibres was carried out according to ASTM D570. Five samples with a diameter of 51 mm and thickness of 3 mm were used for each composition. The samples under test were totally immersed in distilled water at room temperature for 24 h. The samples under test were taken out from the distilled water and all surface water was removed with a clean dry cloth and weighed using analytical balance of 0.1 mg resolution. The water absorption is determined by the increase in weight percentage (%) of the specimens after 24 h of immersion was calculated with the following formula (Sinha & Rout 2009):

$$\text{Increasing in weight percentage (\%)} = \frac{\text{Weight after} - \text{Weight before}}{\text{Weight before}} \times 100$$

RESULTS AND DISCUSSION

SEM MORPHOLOGIES OF THE COIR FIBRES

Figure 1 shows the morphologies of coir fibres at 1500× magnification. In Figure 1(a), it can be observed that the outer surface texture of the untreated coir fibre was uniform, smooth and there was absence of destructive surface. This was due to the presence of surface impurities such as wax, pectin, and greases that were covering the coir fibres. In Figure 1(b) and 1(c), there was presence of destructive surfaces on both NaOH-treated coir fibres. The destructive degree or roughness of the NaOH-treated coir fibres becomes more significant as the duration of the NaOH treatment increases from 24 to 48 h. This was attributed by the penetration of NaOH solution into

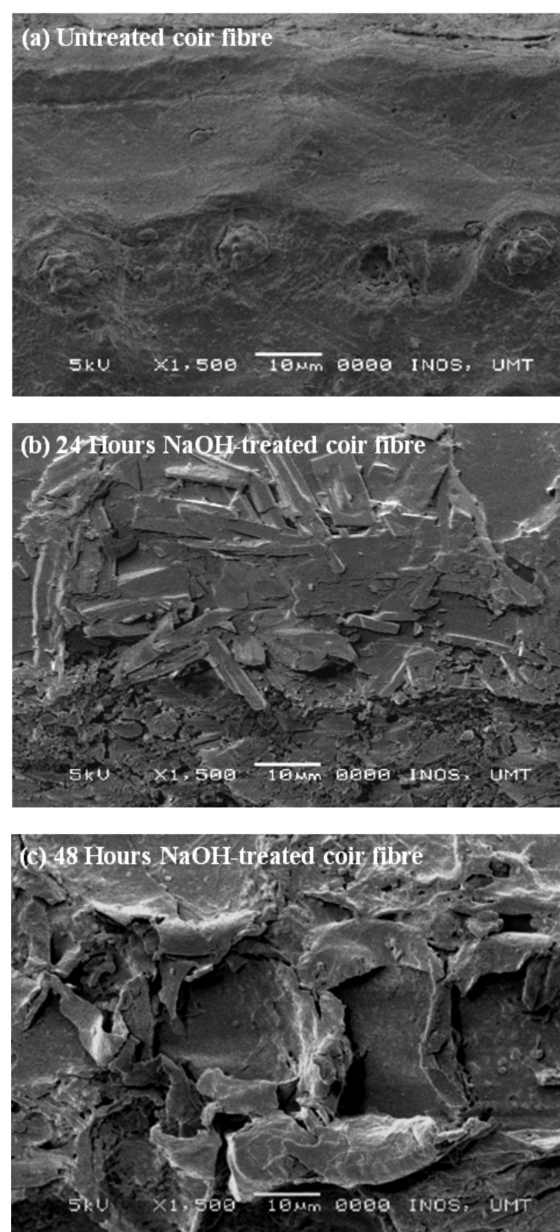


FIGURE 1. SEM morphology of (a) untreated coir fibre (b) 24 h NaOH-treated coir fibre (c) 48 h NaOH-treated coir fibre

the coir fibres. NaOH breaks and removes the hydroxyl groups as well as the surface impurities in the amorphous non-cellulose constituents which increases the number the crystalline cellulose exposed on the fibre surface. Mechanical interlocking of the exposed crystalline

cellulose in the NaOH-treated coir fibre to the epoxy resin led to better fibre-matrix interfacial adhesion in the NaOH-treated coir fibre composites. Moreover, the removal of the hydroxyl groups in the NaOH-treated coir fibres reduces its hydrophilic nature and provides better compatibility of fibre to the matrix (Akhtar et al. 2016; Brígida et al. 2010; Kabir et al. 2012; Li et al. 2007; Punyamurthy et al. 2017; Zaman & Beg 2014). In order to confirm the removal of the amorphous non-cellulose constituents from the coir fibres, FTIR spectroscopy was used to investigate the structural changes in the coir fibres surface before and after alkaline treatment.

FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY ANALYSIS OF COIR FIBRES

Figure 2 shows the comparison of FTIR spectra of untreated, 24 h NaOH-treated and 48 h NaOH-treated coir fibres within the 650-4000 cm^{-1} wavelength region. It was found that the absorption bands at 3340.71, 3344.57 and 3350.35 cm^{-1} correspond to the hydroxyl group (OH) in untreated coir fibre, 24 h NaOH-treated and 48 h NaOH-treated coir fibres, respectively (Akhtar et al. 2016; Nascimento et al. 2018). It can be observed that the intensity of OH group in the coir fibre was significantly reduced after both 24 and 48 h alkaline treatment, indicating that the alkaline treatment breaks the hydrogen bonding in the OH group, caused the reduction of the OH groups in the NaOH-treated coir fibres. Meanwhile, it was noticed that the decrease in intensity of the OH group for 48 h NaOH-treated coir fibre was insignificant compared to 24 h NaOH-treated coir fibre as during this stage, the alkaline treatment just removed the remaining OH group in the coir fibre.

The absorption bands at 2908.65 and 2910.58 cm^{-1} for the NaOH treated coir fibres and untreated coir fibre, respectively, may be attributed to C-H stretching in methyl and methylene groups (Ikhuoria et al. 2017). The absorption band at 1724.36 cm^{-1} in untreated coir fibre corresponds to the carbonyl (C=O) stretching vibrations of carboxyl and acetyl groups in hemicelluloses. It can be observed that the absorption band at 1724.36 cm^{-1} no longer existed in NaOH-treated coir fibres for both 24 and 48 h. The carboxyl and acetyl groups in the hemicelluloses were soluble in aqueous alkaline solution, thus causing the band to disappear in NaOH-treated coir fibres for both 24 and 48 h. This finding is in agreement with previous study which reported that alkali treatment removed hemicelluloses (Abraham et al. 2013; Akhtar et al. 2016; Ikhuoria et al. 2017; Sinha & Rout 2009; Valadez-Gonzalez et al. 1999).

The absorption bands at 1247.94, 1244.09 and 1249.87 cm^{-1} correspond to the C-O stretching of the acetyl group of lignin. It can be observed that for both 24 and 48 h NaOH-treated coir fibres, the intensity of the bands at 1244.09 and 1249.87 cm^{-1} , respectively, decreased with increasing alkaline treatment duration but these bands do not completely disappear after both 24 and 48 h alkaline treatment. This implies that there was a decrease in lignin content of the NaOH-treated coir fibres but lignin is much

more difficult to be completely removed by alkalization compared to hemicelluloses (Oushabi et al. 2017; Sinha & Rout 2009). The absorption bands at 1031.92, 1035.77 and 1029.99 cm^{-1} for the untreated coir fibre, 24 and 48 h NaOH treated-coir fibres, respectively, were attributed to C-O stretching in cellulose, hemicelluloses and lignin (Nascimento et al. 2018).

THERMAL DEGRADATION

The thermal decomposition of natural fibres involves the

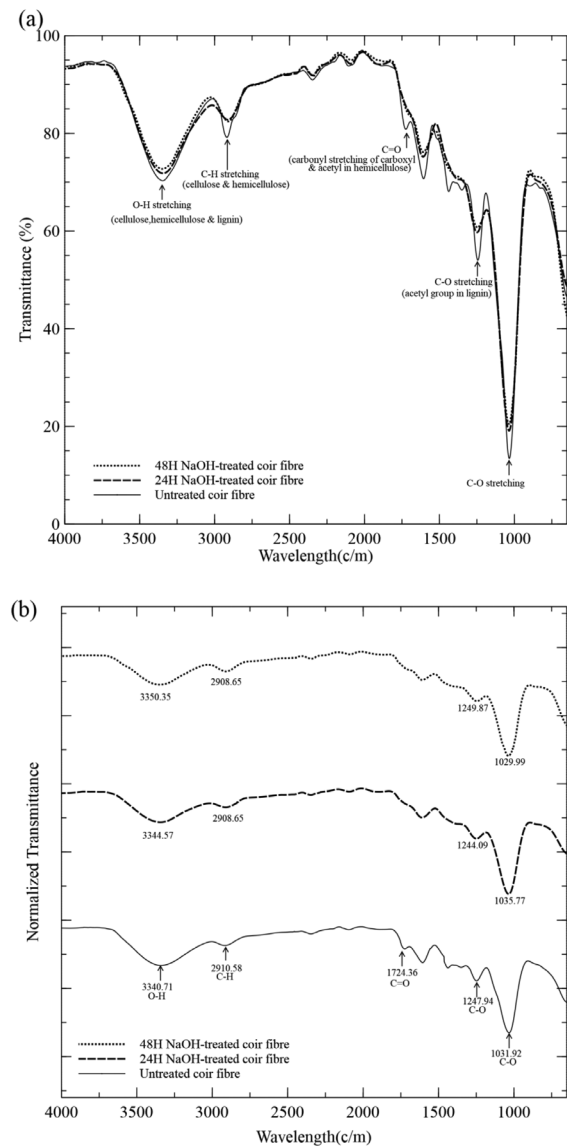


FIGURE 2. FTIR spectra of the coir fibres (a) Transmittance versus wavelength (b) Normalized transmittance versus wavelength

dehydration, emission of volatile components, weight loss due to hemicelluloses and lignin degradations (Azwa et al. 2013). The polymer composites reinforced with natural fibres normally decompose at lower temperature than the pure polymer, indicating less thermal stability (Methacanon et al. 2010).

Figure 3 presents the TGA curves of the untreated and NaOH-treated coir fibre composites. The curves showed the untreated and NaOH-treated coir fibres composites degrade through three main stages in the following ranges: (A) from 50°C to 120°C, (B) 260°C to 370°C, (C) 400°C to 600°C. The decomposition at range A was due to the evaporation of the moisture and volatile contents in the coir fibres. The second decomposition at range B involved the hemicelluloses degradation, whereas during the third decomposition at range C, the coir fibres started to decompose due to cellulose and lignin degradations.

It was found that the untreated coir fibre composite showed early decomposition than 24 and 48 h NaOH-treated coir fibre composites. During the first decomposition stage that occurs at lower temperature, there was significant residue weight loss in untreated coir fibre composite indicating that greater amount of moisture and volatile contents evaporate in untreated coir fibres. The untreated coir fibre has higher moisture content due to the presence of hemicelluloses and lignin (Azwa et al. 2013). The higher the amount of hemicelluloses and lignin, the greater is the moisture absorption in coir fibres and thus, weight loss due to evaporation of moisture occurs more in untreated coir fibre when it was heated. Moreover, weight loss of the untreated coir fibre composite at lower temperature was also contributed by the evaporation of moisture that was trapped in void caused by the poor interface of the untreated coir fibre and the epoxy resin (Costa et al. 2006). On the other hand, both 24 and 48 h NaOH-treated coir fibre composites showed better thermal stability at lower temperature and higher amount of residue due to the removal of hemicelluloses and lignin that effectively reduces the weight loss due to moisture evaporation.

During the second decomposition stage that occurs at range B, decomposition of hemicelluloses took place. Similarly for both 24 and 48 h NaOH-treated coir fibre composites, since there was removal of hemicelluloses content in the NaOH-treated coir fibres, thermal degradation of those composites occurs at higher temperature than the untreated coir fibre composite thus, supports the increased of thermal stability. Moreover, 48 h NaOH-treated coir fibre composite showed better thermal stability compared to 24 h NaOH-treated coir fibre composite as the hemicelluloses content was less, implies that more hemicelluloses were removed with the increased duration of coir fibres surface modification process through alkaline treatment.

During the third decomposition stage that takes place at higher temperature, untreated coir fibre composite started to decompose earlier than both 24 and 48 h NaOH-treated coir fibre composites. However, in untreated coir composite, the amount of residual weight was greater than NaOH-treated coir fibre composites, attributed by the higher lignin content in untreated coir fibre that produces more char. The formation of char in the untreated coir composite insulates the composite from further thermal

degradation thus increased the fire resistance ability of the composite. Meanwhile, the NaOH treatment reduces lignin content in the coir fibres and the removal of the lignin content increased with the increase of treatment duration. When the NaOH-treated coir fibre composites were subjected to heat, less chars were formed due to the lower lignin content in the NaOH-treated coir fibre, resulting in lower amount of residual weight (Beg & Pickering 2008).

It can be summarised that the surface modification on the coir fibre by using NaOH treatment led to better thermal stability but reduces the fire resistance ability or insulation to thermal degradation due to lower amount of char formation. Thus, there is a need to yields optimum removal of lignin and hemicelluloses contents in the coir fibre in order to improve chars formation at high temperature degradation as well as to increase its thermal stability at low temperature degradation.

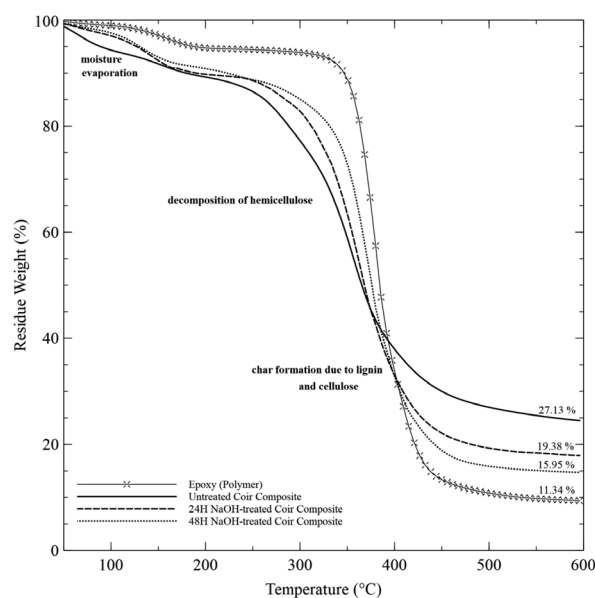


FIGURE 3. TGA curves of untreated and NaOH-treated coir fibres composites

WATER ABSORPTION

The water absorption percentage of the composites is presented in Figure 4. It was noticed that polymer absorbed the least water or moisture due to its hydrophobic nature. On the other hand, it was found that there was significant increase in water absorption percentage in untreated coir fibre composite. This was due to the presence of high hydrophilic hemicelluloses content in untreated coir fibres which facilitates its water absorption tendency and caused the coir fibre to swell when it is immersed in water or exposed to moisture. The hydrophilic nature of the coir fibre were reduced through NaOH treatment, as evidenced by the water absorption percentage of NaOH-treated coir fibre composites for 24 and 48 h that were

lower compared to untreated coir fibre composite. NaOH solution penetrated through lignocellulosic structure in the coir fibre to effectively remove hemicelluloses, caused the NaOH-treated coir fibres to be less hydrophilic, which in turn promotes the compatibility of the NaOH-treated coir fibre to the epoxy resin. The NaOH-treated coir fibre was easily wetted to epoxy resin than untreated coir fibre, thus resulting in better mechanical interlocking between the coir fibre and the epoxy and minimises the void spaces around fibre particles (Li et al. 2007). Note that in untreated coir fibre composite, void spaces are formed in between fibre particles and the matrix that is resulted by poor wettability of the fibre to the matrix. Water or moisture as well as air were easily trapped in the void spaces, resulting in higher water absorption or water uptake (Azwa et al. 2013; Costa et al. 2006).

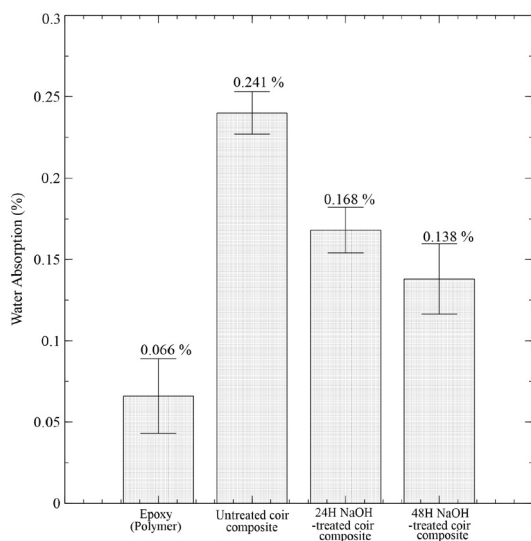


FIGURE 4. Water absorption of the composites for 24 h immersion

CONCLUSION

Coir fibres were treated with 5 wt. % NaOH solution for 24 and 48 h at room temperature. SEM analysis showed that the surface morphologies of the NaOH-treated coir fibres became rough as NaOH solution penetrated through the coir fibre surface to remove amorphous non-cellulose constituents consisting of hemicelluloses, lignin, and surface impurities. The coir fibre surface roughness became significant with the increase of the alkaline treatment duration. The rough surface of NaOH-treated coir fibres improved the interfacial adhesion of the fibre-matrix thus supports the increase in moisture resistance ability, as evidenced in water absorption test of the NaOH-treated coir fibres composites. FTIR results confirmed that the hemicelluloses in the NaOH-treated coir fibres were removed due to the absence of absorption bands at 1724 cm^{-1} . However, lignin content in the coir fibre is unable to be fully removed by alkaline treatment that was performed

in this study. Thus, it is recommended to increase the concentration of NaOH solution used for alkaline treatment on the coir fibres. Meanwhile, the alkaline treatment on the coir fibres effectively increased the thermal stability of the composites but have lower amount of char formation at high temperature degradation. Furthermore, the NaOH-treated coir fibres were less hydrophilic compared to untreated coir fibre, as evidenced by intensity decreased in OH group in the NaOH-treated coir fibres and lower water absorption percentage in the NaOH-treated coir fibres composites.

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