Effect of Alkali Treatment on the Physical, Mechanical, and Morphological Properties of Waste Betel Nut (*Areca catechu*) Husk Fibre

Yusriah Lazim,[a,]* Sapuan Mohd Salit,[b] Edi Syams Zainudin,[c] Mariatti Mustapha,[d] and Mohammad Jawaid [b]

This study aims to determine the properties of waste betel nut husk (BNH) fiber as a potential alternative for reinforcement in polymer composites. The BNH fibres were subjected to alkali treatment using 5% sodium hydroxide. In this work, husk fibres extracted from betel nut fruit were characterized for its chemical composition, tensile properties, morphology, and interfacial shear strength. The cellulose content was increased with alkali treatment. Tensile strength and Young's modulus of BNH fibre dropped drastically with alkali treatment but with improvement in elongation at break of the fibre due to extraction of cementing materials of microfibrils in natural fibre, *i.e.* lignin and hemicellulose. SEM observations revealed that poor tensile strength and modulus were related to the cell wall thinning and deep pores in BNH fibre due to alkali treatment. Interfacial shear strength (IFSS) of alkali treated fibre was higher as compared to untreated BNH fibre due to the increase in fibre surface roughness with alkali treatment.

Keywords: Natural fibre; Alkali treatment; Fibre microstructure; Mechanical properties

Contact information: a: Department of Mechanical and Manufacturing Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia; b: Laboratory of Biocomposite Technology, Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia; c: School of Engineering & Design, Brunel University, Uxbridge Middlesex, UB8 3PH, United Kingdom; d: School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia Engineering Campus, 14300 NibongTebal, Penang, Malaysia;
** Corresponding author: yusriahlazim@gmail.com; Tel.: +603-89466318; Fax:+603-86567122*

INTRODUCTION

Nowadays, the urge to replace synthetic fibre with natural fibre is increasing due to environmental concerns. Synthetic fibres are commonly used in composites fabrication, owing to their excellent mechanical properties. However, synthetic fibres are non-biodegradable; hence many attempts are made to find alternatives for synthetic fibre. Natural fibres are available everywhere, and researchers are attracted towards them as promising alternative material to traditional glass fibres due to their good specific strength, low cost, renewability, market appeal, fully biodegradable nature, and non-abrasive character (Jawaid and Khalil 2011).

Natural fibres used as reinforcement can be classified into many categories, *i.e.* bast, seed, fruit, straw fibre, leaf, and grass fibres (Gon *et al.* 2012). There is a wide selection of natural fibre used as reinforcement in polymer composites such as kenaf, hemp, jute, and coir. These fibres exhibit good reinforcing properties in polymer composites; they have low density and are cost effective. They are less hazardous to humans during fabrication and handling as compared to synthetic fibre; hence it is a good option to employ natural fibre as reinforcement in polymer composites.

Agricultural crop byproducts are a good source of natural fibre. They are in abundance, renewable, and also an inexpensive alternative source for natural fibre (Reddy and Yang 2009). Utilizing byproducts as reinforcement in polymer composites also helps in recycling agricultural waste and contributes to a better agricultural waste management. Byproducts of food crops such as wheat stalks, corn stalk, rice husk, sugar cane bagasse, fruit peels, and pineapple leaves have been widely used as reinforcement in polymer composites (Panthapulakkal and Sain 2007; Reddy and Yang 2009). Research on using fibre from by-products of commercial crops have also been carried out by several workers. Among them, incorporation of fibres from oil palm empty fruit bunch, coir, jute, and banana fibres in polymer composites have been studied, and it is reported that the use of these crop by-products had improved the mechanical properties of polymer composites (Lai *et al.* 2005; Jawaid and Abdul Khalil 2012).

Betel nut husk (BNH) fibre is a type of agro-waste from commercial crops that appears to be a good alternative to synthetic fibre. Betel nut crops are cultivated in tropical climate countries, and it is categorized as an important economic crop in India. According to statistics reported by Food and Agriculture of United Nation in Table 1 (Food and Agriculture Organization of the United Nations, 2013), India, China, and Indonesia are among major producers of betel nut. Other smaller tropical countries such as Mynmar, Bangladesh, Nepal, and Malaysia also contribute to the global production of betel nut. BNH fibres are obtained from the husk of the betel nut fruit. The BNH fibres are traditionally used as housing insulation material and fabrication of value-added products such as cushion, handcrafts, and non-woven fabrics (Srinivasa and Bharath 2011). Many studies on the properties of polymer composites reinforced betel nut husk fibre have been carried out (Hassan *et al.* 2010; Yousif *et al.* 2010; Nirmal *et al.* 2011).

Table 1: Betel Nut Growing Countries and the Production of Betel Nut in 2010-2011 (Food and Agriculture Organization of the United Nations 2013).

Country	**Production (Tonnes)**	
	2010	**2011**
India	478000	478000
Indonesia	63100	183100
China	201000	129316
Myanmar	126200	121003
Bangladesh	91681	92589
Nepal	4266	7620
Malaysia	650	646
Kenya	92	110
Maldives	9	10

The major downside of utilizing natural fibres in polymer composites is the incompatibility between the hydrophilic nature of natural fibre and hydrophobic properties of polymer matrix, which generally leads to poor fibre-matrix interfacial adhesion. Poor interfacial adhesion between fibre and matrix affects the mechanical properties of the composites due to interruption of good stress transfer at the fibre-matrix interface. Many methods have been investigated to alter the natural fibre surface in order to improve the compatibility of natural fibre with most types of polymer matrix. Common surface treatments for natural fibres are alkali and silane treatment, acetylation, grafting methods, and uses of chemical agents to improve the fibre-matrix bonding (Cyras *et al.* 2004; Stocchi *et al.* 2007). Among these treatments, alkali treatment is often chosen to improve the adhesion properties of natural fibre, as it is considered more economical compared to other types of chemical treatment (Ramadevi *et al.* 2012). Alkalization process involves physical reaction such as removal of pectin, wax, and lignin from the fibre surface; alkaline treatment also chemically changes the structure of cellulose type I to cellulose type II (Mwaikambo and Ansell 2002).

Many authors have reported that alkali treatment has a positive effect on the mechanical properties and interfacial adhesion of various types of natural fibres such as hemp, jute, bamboo, and kenaf (Mwaikambo and Ansell 2002; El-Shekeil *et al.* 2012; Yan *et al.* 2012). According to Sampathkumar *et al.* (2012), BNH fibre has a good potential to be used as reinforcing material in composites after the fibre is subjected to surface modification. They reported that surface modification reduced the moisture absorption and improved the interfacial adhesion of BNH fibre. Thus, in this study, betel nut husk (BNH) fibres of three different degrees of maturity were subjected to 5% sodium hydroxide treatment at room temperature, to investigate the effect of alkali treatment on the chemical content, tensile properties, interfacial shear strength, and morphology of these BNH fibres after the alkalization process.

EXPERIMENTAL

Materials

Betel nut husk fibres were obtained from a local betel nut plantation in Banting, Selangor, Malaysia. The resin used as matrix for fabrication of interfacial shear strength (IFSS) samples was EPOVIA RF-1001; a vinyl ester resin supplied by CCP Composites. 1% methyl ethyl ketone peroxide (MEKP) was used to cure the resin at room temperature. Sodium hydroxide (NaOH) was procured from Sigma-Aldrich, USA.

Extraction of Fibres

The betel nut husks were soaked in water at room temperature for 5 days to loosen the fibre from the husk. The BNH fibres were separated manually from the nut portion by a hand stripping method and washed thoroughly with distilled water before being matured in an oven at 70 °C for 24 h. The oven-dried BNH fibres were kept in a desiccator to protect the fibres from absorbing the moisture from the atmosphere. The physical properties of the BNH fibres were as listed in Table 2.

Table 2. Physical Properties of BNH Fibres (Yusriah *et al.* 2012)

Type of fibre	Fibre diameter (mm)	Fibre length (mm)	Fibre aspect ratio	Density (g/cm^3)
Unripe BNH fibre	0.46-0.47	57-60	121.27	0.19
Ripe BNH fibre	0.42-0.45	53-55	122.22	0.34
Matured BNH fibre	0.37-0.41	45-49	119.51	0.38

Methods

Alkali treatment

Known quantities of sodium hydroxide (NaOH) pellets were dissolved in distilled water at room temperature to prepare a 5 wt.% of NaOH solution (Solid:liquid, 5:100). BNH fibres were immersed in the 5 wt.% NaOH solution for 30 min. The BNH fibres were later washed thoroughly in distilled water to remove residual NaOH solution. Removal of moisture in alkali-treated BNH fibres was done *via* oven-drying at 70 °C for 24 h.

Characterization of Fibre

Chemical composition of betel nut husk fibre

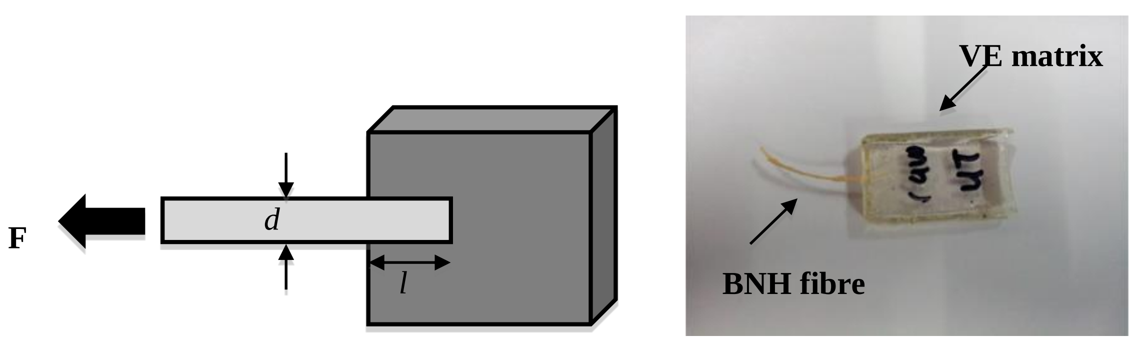
The process to determine the chemical composition of BNH fibre was conducted by Agriculture Chemistry Analysis Laboratory at MARDI, Selangor, Malaysia. Acid detergent fibre (ADF) and neutral detergent fibre (NDF) methods were used to determine the cellulose and hemicellulose content. The cellulose and hemicellulose content were calculated from the ADF, ADL and NDF results using methods reported by Rinne *et al.* (1997).

Tensile properties of betel nut husk fibre

The specimens of betel nut husk fibres for tensile testing were randomly selected from the strand bundle of betel nut husk. The selected fibre was carefully observed using an optical microscope to measure the diameter of the fibre. The fibre was mounted and glued onto a tab-shaped piece of cardboard, with the gauge length of 20 mm and tested according to the standard test method for the single fibre tensile test ASTM D 3379. The tab-shaped cardboard was cut at the mid-gauge length prior to the tensile testing. The specimen was tested with a crosshead speed of 1 mm/min with replication of 25 specimens using a universal testing machine, model Instron 3366, with a load cell of 5 kN.

Interfacial shear strength (IFSS) of BNH fibre

The samples were prepared by embedding the BNH fibres end tip of 3 mm length in a mixture of a known quantity of vinyl ester resin and 1.5 wt% MEKP. The fabricated sample and schematic diagram of IFSS samples are shown in Fig. 1.



(a) (b)

Fig. 1. The IFSS sample in (a) schematic illustration and (b) after fabrication

The samples were tested using a 5 kN cell load Universal Testing Machine (UTM) Instron model 3365 with a cross-head speed rate of 0.1 mm per minute. The gauge length was fixed at 10 mm and 15 samples were tested for each type of BNH fibres. The debonding force required to pull the fibre out of the matrix was determined during the testing and the corresponding IFSS values was calculated using Eq. 1,

$$\sigma = \frac{F}{\pi d l} \tag{1}$$

where σ is the interfacial shear strength (Pa), *F* is the maximum load before fibre pull out (N), *d* is the diameter of BNH fibre (m), and *l* is the length of embedded fiber in matrix (m).

Morphological observation

A scanning electron microscope (SEM) instrument model ZEISS SUPRA 35VP was used to observe the structural changes on the surface and cross-sectional parts of the fibres. The fibres were mounted on aluminum stubs and sputter coated with a thin layer of gold to avoid electron charge accumulation.

RESULTS AND DISCUSSION

Chemical Composition

The chemical compositions of BNH fibres and BNH fibre after alkali treatment are presented in Table 3. The data in Table 3 revealed that cellulose was the major component in BNH fibre, followed by lignin and hemicellulose, respectively. Moon and co-workers (Moon *et al.* 2011) have reported that cellulose is an important component in natural fibre as cellulose fibrils are the main reinforcement phase of plants that provides strength to the plants. Cellulose is known to have a good mechanical strength due to the numbers of hydrogen bonds existing between the molecular chains and also influenced by the crystalline nature of cellulose structures (Moon *et al.* 2011).

Matured BNH fibre was found to have highest cellulose and lignin content as compared to unripe and ripe BNH fibres. The high lignin content in matured BNH fibre is due to the increase in lignin deposition in the fibre cell wall with the increasing maturity of the plant (Kitaba and Tamir 2007). The low hemicellulose content of BNH fibres lessens the moisture uptake of BNH fibres. This is due to the fact that hemicellulose contains many hydroxyl groups, which are responsible for attracting water molecules (Agu *et al.* 2012). Decrease in moisture uptake will benefit good dimensional stability, and reduces moisture related problems during processing, storage, and application of BNH fibre and its corresponding polymer composites. However, it is worth noting that not much difference was observed in the cellulose, hemicellulose, and lignin content of the unripe, ripe, and matured BNH fibre.

BNH fibres were chemically treated using sodium hydroxide (NaOH). Alkalization of plant fibres using sodium hydroxide is one of the common chemical treatments used to remove lignin, oil, and waxy components and pectin from the natural fibre surface. The presence of these substances on natural fibre surfaces interrupts the bonding between natural fibre and polymer matrix in composites (Mwaikambo and Ansell 2002).

Table 3. Chemical Composition of Untreated and Alkali Treated BNH Fibres

Type of BNH fibre		Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)
Untreated	Unripe	43.49	17.71	26.85	1.69
	Ripe	43.99	12.07	26.88	3.13
	Matured	50.21	13.16	29.34	3.48
Treated	Unripe	49.26	8.49	22.17	4.54
	Ripe	52.09	12.04	22.34	5.94
	Matured	50.51	12.37	26.1	2.53

Based on the chemical composition of the BNH fibres in Table 3, it can be observed that lignin was most affected by the alkali treatment. The decrease in lignin content by 10.8%, 16.88%, and 2.79% in unripe, ripe, and matured BNH fibres, respectively, shows that lignin was partially removed from the BNH fibre during the alkali treatment. With the partial removal of lignin from the natural fibre surface, more cellulose components of natural fibre are exposed (Sun 2008). This was evident in the increase of cellulose in BNH fibre after alkali treatment, as shown in Table 3. The cellulose content increased by 13.26%, 18.41, and 0.6% in unripe, ripe, and matured BNH fibres respectively, following the alkaline treatment.

Hemicellulose was the least affected component in the alkali-treated ripe and matured BNH fibre. However, an opposite trend was observed for unripe BNH fibre, where the hemicellulose content dropped by 52.06% after alkali treatment. Based on the observation on the data shown in Table 3, it is interesting to point out that the cellulose, hemicellulose, and lignin content of BNH fibre are comparable to other common lignocellulosic fibres such as coir (Mahato *et al.* 2009). According to Fidelis *et al.* (2013), the chemical composition of natural fibre influenced the tensile properties of natural fibre. Based on observations in their studies, it was found that sisal fibre with high cellulose content (73%) exhibits higher tensile properties than that of jute fibre with lower cellulose content (65%). They also conclude that the good tensile characteristic of the fibre attributed to the high cellulose content and also the morphology of the fibre (Fidelis *et al.* 2013). Similar observations were also reported by Reddy and Yang (2005) in their studies on biofibres derived from agricultural by-products. According to their report, the strength of the fibre such as tensile properties are closely related to the presence of cellulosic (cellulose, hemicellulose), non-cellulosic components (lignin, wax), and also the variations in the fibre structure (dimension of unit cell). These various findings could be an indication that BNH fibre has the potential as reinforcement in polymer matrix owing to its high cellulose content and interesting fibre structures, which will be further discussed in the morphological study part.

Tensile Properties

Single fibre tensile tests were performed to determine the tensile properties of alkali-treated BNH fibres. The results from this testing were analyzed and recorded in Table 4. The tensile properties of alkali-treated BNH fibres were compared to the tensile properties of untreated BNH fibres reported in our previous works (Yusriah *et al.* 2012). In the case of untreated BNH fibre, matured BNH fibre shows higher Young's modulus than those of unripe and ripe BNH fibre. Ripe BNH fibre shows high tensile strength and a slightly lower Young's modulus value than matured BNH fibre. The good stiffness properties of matured BNH fibre is to be expected, since the highest cellulose and lignin content was confirmed in matured BNH fibre, based on the chemical composition data presented in Table 3. This finding is also in accordance with works by Rajan *et al.* (2005), where the matured plant fibres were reported to have more lignin than the plant fiber from an earlier stage of maturity, which influenced the Young's modulus of BNH fibre.

Table 4. Mechanical Properties of Untreated and Alkali-Treated BNH Fibres

Type of BNH fibre		Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Untreated [23]	Unripe	123.92±49.6	1285.66±710.2	22.49±6.5
	Ripe	166.03±55.1	1381.31±551.9	23.21±5.0
	Matured	128.79±43.8	2569.03±637.3	23.13±6.7
Treated	Unripe	32.67±9.4	375.3±85.5	46.42±12.4
	Ripe	44.73±9.5	616.03±136	32.5±6.3
	Matured	30.74±6.0	363.79±77.2	37.25±8.47

The comparison data in Table 4 shows that the alkali-treated BNH fibres exhibited lower tensile strength and Young's modulus values compared to the untreated BNH fibres. The decrease in the tensile strength and Young's modulus of alkali-treated fibre could be related to the changes in the cellulose crystallinity during alkali treatment (Gomes *et al.* 2007). The changes in the crystallinity of the cellulosic molecular structures of the alkali-treated natural fibre is due to the partial conversion of cellulose I into cellulose II, producing more amorphous cellulose structures in the fibre, which reduces the tensile properties of the alkali-treated natural fibre (Okano *et al.* 1998; Gomes *et al.* 2007).

The lowering of the tensile strength and Young's modulus of the alkali-treated natural fibre were also reported by Nitta *et al.* (2013) in their studies on the effect of alkali treatment on the tensile properties of kenaf fibre. They reported that tensile strength and Young's modulus of the alkali-treated kenaf fibre decreased following alkali treatment. According to their report, the decrease in the tensile strength and Young's modulus was attributed to the changes in the cellulose crystallinity index of the fibre, due to the formation of more cellulose type II. The less ability of cellulose type II to deform has influenced the decrease in the tensile strength and modulus of the alkali treated fibre (Gassan and Bledzki 1999). Besides, poor tensile strength of alkali treated BNH fibre could also be attributed to the damages in the fibre structure such as partial rupture of the ultimate cell wall, and increase in deep pores and thinning of the BNH fibre cell walls during alkali treatment (Rodriguez and Vazquez 2006). The details on the changes of the alkali treated BNH fibre structure will be discussed in the morphological study section.

It can be observed from Table 4 that the elongation at break of BNH fibres exhibited positive improvement with alkali treatment. The alkali-treated BNH fibres showed an increase in elongation at break relative to that of untreated BNH fibres. The improvement in elongation at break of alkali treated BNH fibre also may be related to the changes of cellulose structure as a result of the alkali treatment. This is owing to the low modulus of cellulose II structures, which can deform more easily than the cellulose I (Gassan and Bledzki 1999). In addition, the alkali treatment is also reported to result in less dense and less rigid fibre due to the partial removal of the hemicellulose and lignin as binder substances that cement the microfibrils together (Rong *et al.* 2001). The partial removal of these substances decreases the resistance of the microfibrils of natural fibre relative to stretching, causing the loose microfibrils to rearrange along the tensile deformation direction, enhancing the elongation at break of natural fibre (Rong *et al.* 2001; Sampathkumar *et al.* 2012).

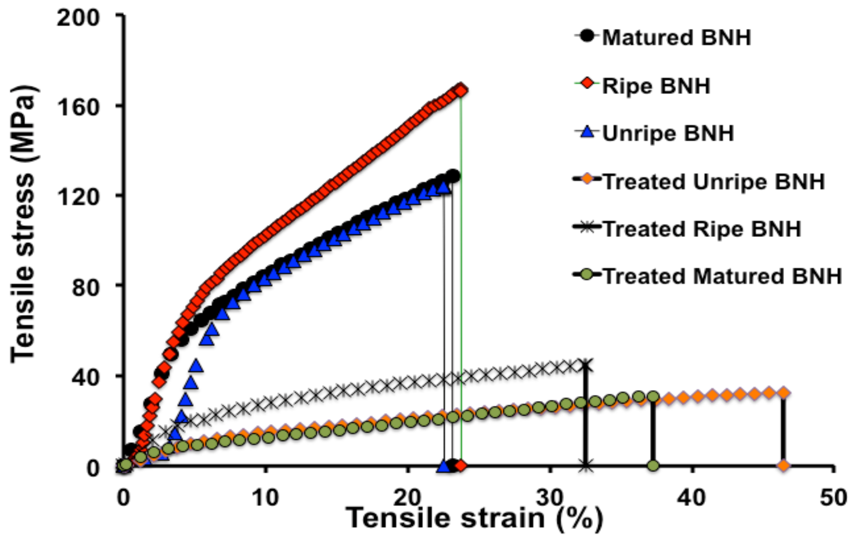


Fig. 2. Stress-strain curves of untreated and alkali treated unripe, ripe, and matured BNH fibres

Figure 2 depicts the stress-strain curves of alkali-treated BNH fibres. These stress-strain curves explained the failure behaviour of BNH fibres during tensile testing and the influence of alkali treatment on the tensile properties of BNH fibres. Based on Fig. 2, stress-strain curves for alkali-treated unripe, ripe, and matured BNH fibres were observed to have lower slope than those of untreated BNH fibres. These findings confirmed that alkali-treated BNH fibres exhibited lower tensile strength compared to untreated BNH fibres. Steeper slopes of untreated BNH fibre shows that untreated BNH possessed higher Young's modulus than that of alkali-treated BNH fibre. This is in accordance with the tensile properties results presented in Table 4. However, it is interesting to note that alkali treatment led to an approximately 20 to 24% increase in tensile strain-to-failure of BNH fibres. Stress-strain curves for both untreated and alkali-treated exhibit non-linear region indicating that BNH fibres undergo plastic deformation and exhibit ductile fracture.

Morphology

SEM observations were conducted on the BNH fibre surface and cross-section of fractured fibre from tensile testing to provide a better understanding on the effect of sodium hydroxide on BNH fibres. The SEM micrographs are presented in Figs. 3 and 4, revealing the morphologies of alkali-treated unripe, ripe, and matured fibre surfaces and fibre cross-section, respectively. It can be observed from Fig. 3 that the alkali treatment had caused an apparent effect on the unripe, ripe, and matured BNH fibre surfaces. The alkali-treated unripe, ripe, and matured BNH fibres showed rougher surfaces as compared to the untreated BNH fibres.

The removal of trichomes following alkali treatment was also evident on the surface of alkali-treated BNH fibres, leaving pit-like pores on BNH fibre surface. This is due to the removal of waxy layer on BNH fibre surface, creating deeper pores and rougher surface. Thus, more possible anchoring points for mechanical interlocking between BNH fibre and polymer in BNH fibre reinforced polymer composites were created. Plus, the waxy layer naturally present on the surface of natural fibre prohibits effective interfacial bonding between natural fibre and polymer matrix (Mwaikambo and Ansell 2002). Hence, removal of wax, pectin, and low-molecular weight components on the natural fibre surface assists in better matrix resin wetting on the fibre surface.

The SEM micrographs of untreated and alkali-treated BNH fibres cross-sections in Fig. 4 revealed that the alkali treatment resulted in thinning of BNH fibre cell walls. Fidelis *et al.* (2013) highlighted similar issue in their works of the effect of fibre morphology on tensile properties of natural fibres. They have reported that natural fibre with thicker secondary cell wall and smaller lumen size area yields high tensile strength and Young's modulus. The reduction in cell wall thickness is more prominent in unripe and ripe BNH fibre. However, there is no sign of cell wall thinning in matured BNH fibre cross-section. Instead, formation of elongated structures and fibrillated microfibrils due to alkali treatment were quite evident in matured BNH fibre. Decrease in wall thickness also contributes to the reduction of density of the fibre, increasing specific properties and thermal conductivity of the BNH fibre. Thus, alkali treatment is a good option for improving the values and properties of BNH fibres as reinforcement in polymer composites.

Interfacial Shear Strength (IFSS)

Interfacial shear strength (IFSS) is very important to determine the interfacial properties of fibre reinforced polymer composites. According to Khalil *et al.* (2001), the interface in polymer composites is described as the region where matrix and fibre are in contact. The interaction that occurs between fibre and matrix at the interface region can be determined via IFSS testing. IFSS depicts the fracture resistance behaviour of fibre reinforced polymer composites (Quek 1998). In this study, the IFSS was evaluated using single fibre pull out test method. The IFSS of untreated and alkali treated unripe, ripe, and matured BNH fibres are listed in Table 5. In the case of untreated fibre, matured BNH fibre showed superior IFSS relative to unripe and ripe BNH fibres. This is attributed to the fact that matured BNH fibres have a higher percentage of lignin content (Rajan *et al.* 2005). The results were in line with studies reported by Khalil *et al.* (2001), where the higher lignin content in coir fibre was found to be the major factor that contributed to the increase in IFSS values of untreated coir fibre in comparison to untreated empty fruit bunch (EFB) fibre.

Based on the IFSS values listed in Table 5, it can be concluded that the alkali-treated BNH fibres exhibited better IFSS than those of untreated BNH fibres ,which reflects that good compatibility was achieved between BNH fibre and polymer matrix after alkalization process. The positive improvement in IFSS of the treated BNH fibres could be related to the increase in surface roughness of the BNH fibres due to removal of waxy layers and other components during alkali treatment of the BNH fibres. This finding is in accordance with the results documented by Nirmal *et al.* (2011) in their studies of the IFSS properties of betel nut fibre embedded in polyester and epoxy resin.

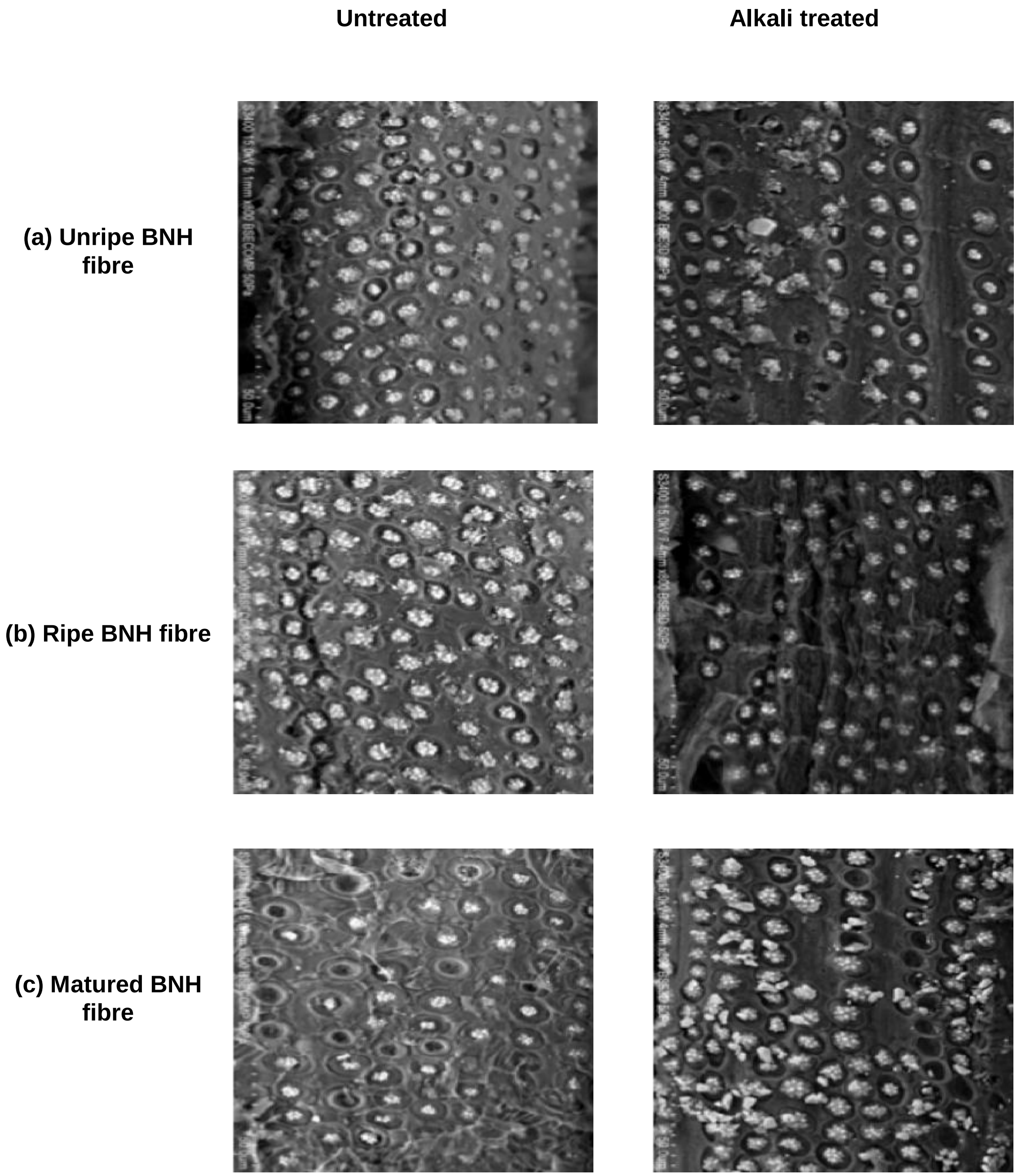


Fig. 3. SEM micrographs of unripe, ripe, and matured fibre surface (untreated and alkali treated)

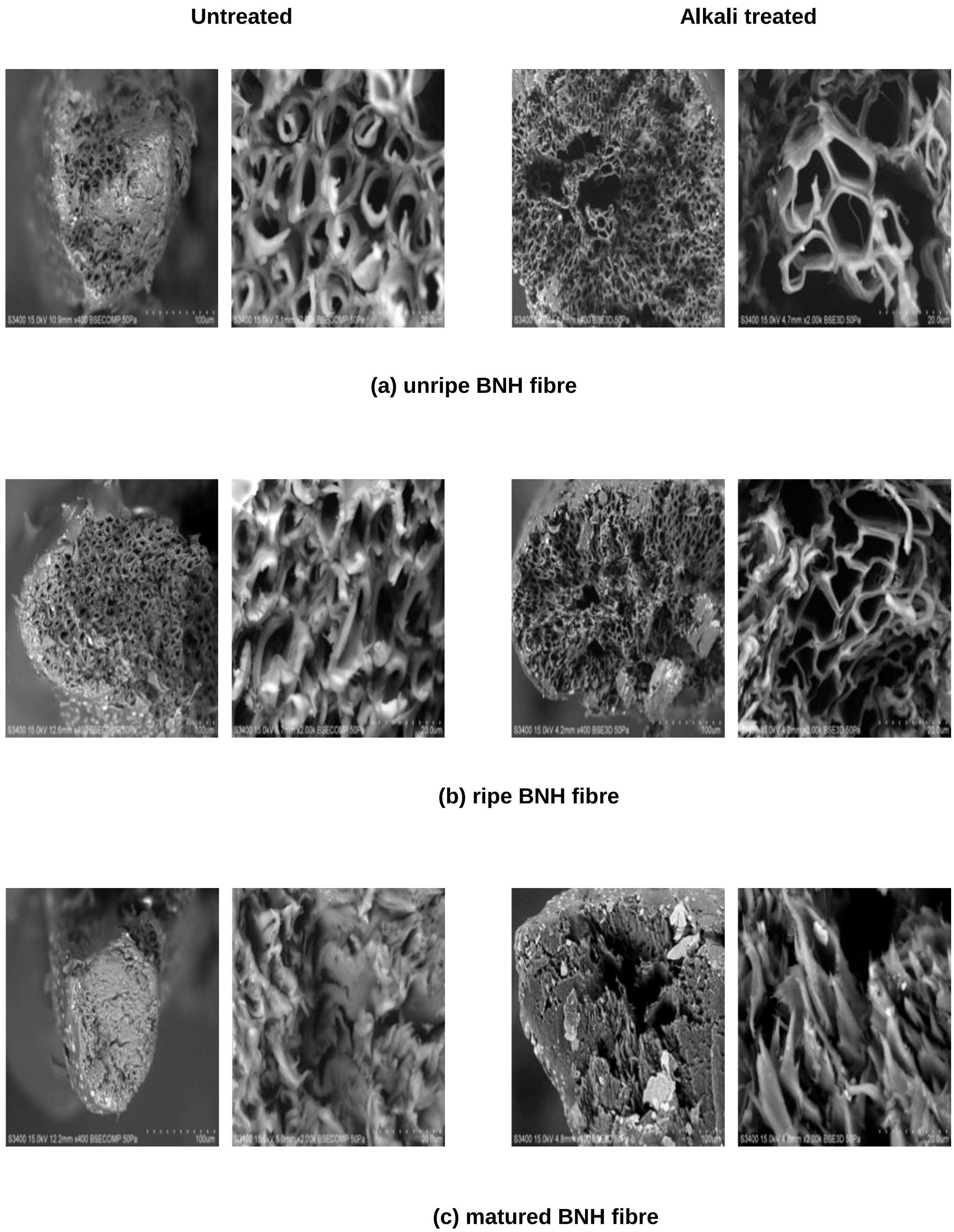


Fig. 4. SEM micrographs of unripe, ripe, and matured BNH fibre cross-sections (untreated and alkali treated)

Table 5. IFSS and Debonding Force of Untreated and Alkali-Treated BNH Fibres

Type of fibre		Debonding force (N)	IFSS (MPa)
Untreated	Unripe	5.95±0.27	1.34 ±0.03
	Ripe	5.22±0.06	1.23±0.11
	Matured	6.08±0.05	1.57±0.02
Alkali treated	Unripe	8.71±0.47	1.96±0.08
	Ripe	14.16±0.39	3.34±0.14
	Matured	7.14±0.21	1.84±0.06

It has been reported that alkali treatment using 6% sodium hydroxide increased the interfacial adhesion strength of the fibre and matrix by 115% (Nirmal *et al.* 2011). The improvement in the IFSS was attributed to the enhanced surface roughness of betel nut fibre due to removal of waxy layer on the fibre surface during alkali treatment, and also low-viscosity of matrix resin for ease of wetting on fibre surface.

According to Thamae and Baillie (2007), alkali treatment assists in the removal of waxy layer and impurities on fibre surfaces; and at the same time exposes small-size fibres and also lignin on the surface of the fibres. Fibrillation is one of the phenomena that occurs during alkali treatment, causing the fibre bundle to split into finer fibres (Joseph *et al.* 1996). Separation of the fibre into smaller fractions has exposed these fine fibres with increased aspect ratio and rougher structures on the fibre surfaces, offering a good fibre-matrix adhesion (Goud and Rao 2011). Exposure of more reactive cellulose components on the fibre surface will promote more effective surface area for better bonding between fibre and polymer matrix via mechanical interlocking mechanism, improving the IFSS of the fibre reinforced composites (Thamae and Bailie 2007; Williams *et al.* 2011). Similar findings were observed in this study, where all alkali-treated BNH fibres showed improved IFSS compared to those of untreated BNH fibres. This can be further understood from SEM micrographs previously shown in Fig. 3, where the micropores on the BNH fibre surface were more prominent after alkali treatment.

Ripe alkali-treated BNH fibre showed the highest IFSS value. The increase in IFSS values for ripe BNH fibre could be related to the tremendous increase in surface roughness of the ripe BNH fibre surface, as shown previously in the SEM micrographs in Fig. 3(b). The changes in ripe BNH fibre structures, such as deep pores, fibrillated surfaces, and removal of waxy layer and trichomes after the fibre was subjected to alkali treatment, aids in providing more anchoring points for interfacial bonding between fibre and polymer matrix *via* mechanical interlocking mechanisms (Sampathkumar *et al.* 2012). A study on the effect of various surface treatments on sisal fibre carried out by Li *et al.* (2005), reported that etched and fibrillated fibre surface, due to surface treatment of sisal fibre, contributes to the increase in the effective surface area which is possible for bonding with the matrix resin.

Furthermore, the samples from the IFSS testing showed that alkali-treated unripe, ripe, and matured BNH fibres exhibited fibre breakage rather than fibre pull-out during IFSS testing. The non-pullout fibre reflects that the BNH fibre adhered well to the matrix, indicating that good bonding between alkali-treated BNH fibre and vinyl ester resin had been achieved.

CONCLUSIONS

1. Cellulose was the component observed in BNH with the highest percentage, followed by lignin and hemicellulose. The chemical composition of BNH fibre was the least affected by plant maturity, as there were only slight differences in the percentage of cellulose, hemicellulose, and lignin content of unripe, ripe, and matured BNH fibre.
2. The lignin and hemicellulose percentages dropped following alkali treatment, yielding a higher proportion of cellulose in alkali-treated BNH fibre.
3. A positive increase in elongation at break of BNH fibre with alkali treatment was observed with deterioration in tensile strength and Young's modulus of BNH fibre after alkali treatment.
4. SEM micrographs of BNH fibre surface and fibre cross-section revealed that the drop in tensile strength and Young's modulus could be related to secondary cell wall thinning and loss of binding materials such as lignin and hemicellulose during alkali treatment.
5. IFSS was found improved with alkali treatment. This is attributed to the improvement in fibre-matrix bonding owing to increase in surface roughness and exposure of more reactive cellulose fibrils on BNH fibre surface due to alkali treatment.
6. Despite the reduction in tensile strength and Young's modulus of single BNH fibre with alkali treatment using 5% sodium hydroxide (NaOH), the treatment successfully removed lignin and unwanted waxy layer in BNH fibre, exposing more cellulose in BNH fibre.

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