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Investigation of Fiber Surface Treatment on Mechanical, Acoustical and Thermal Properties of Betelnut Fiber Polyester Composites

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Abstract

This research work is concerned with the development of unsaturated polyester composites reinforced with betelnut fibers. Moreover, the fibers were chemically modified by sodium hydroxide and its effects on the fiber/matrix interaction were also evaluated. Raw and chemically modified fibers have been used in different proportions for the preparation of these composites. The optimization of fiber proportions has been done in term of their tensile strength. The sodium hydroxide treatment effect of fibers was verified by FTIR analysis and the fibers morphological aspects of fibers by SEM. Likewise, the sound absorption coefficient of composites were studied using impedance tube method. The thermal analyses of composites were made using Thermogravimetric analysis. Compared with untreated fiber composites, there was an improvement in the tensile strength of the treated fiber composites. SEM micrographs of betelnut fiber surface revealed the rough and perforated surface of fibers. Whereas, the cross sectional of the betelnut fibers showed the bigger lumen structures. The sound absorption coefficients of composites increased as the frequency increased and thicker composites appeared to perform better than those of thinner ones. The thermal stability of the treated fiber composites was found to be higher than that of untreated fiber composites.

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Keywords: sound absorption, Thermal stability, Natural fiber, composites, Chemical treatment

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1. Introduction

Polymer based composites are playing a vital role in important applications for the last few decade. These materials are emerging rapidly as the prospective substitute to the metal or ceramic based materials in a number of applications including automotive, aerospace, nautical, sporting goods and electrical and electronic industries. Amongst various composite materials, natural fibers reinforced polymer composites are dwindling under increasing scrutiny due to their easy process and massive eco-friendly advantages [1]. Natural fibers are used as potential reinforcements in polymer matrix in place of glass and other synthetic fibers due to the advantages such as its adequate specific properties, low cost, acceptable biodegradability, low density, and high aspect ratio for efficient stress transfer [2]. Incompatibility between hydrophilic natural fibers and hydrophobic matrix, adversely affects the adhesion of the fiber with the matrix. Weak fiber/matrix interface reduces the reinforcing efficiency of the fiber due to lack of stress transfer from the matrix to load bearing fibers. The mechanical properties of natural fiber-reinforced composite depend extremely on the degree of adhesion between the natural fiber and the matrix [3]. Several strategies have been investigated to improve the fiber properties and to increase the bondability and wettability between the cellulosic materials and the matrix. Generally researchers used fiber surface modification by physical, chemical or mechanical means. Physical methods involve treatments by ionized gas (plasma or corona), laser or steam explosion [4]. Several chemical treatments have been investigated by a number of researchers having potential to remove impurities, oils from the surfaces of the fibers, and make it rough, activate readily available hydroxyl groups or the introduction of new reactive sites/groups; and to stop water uptake [5]. These treatments aimed at improving fiber–matrix adhesion have achieved various levels of success in improving fiber strength, alleviating water absorption and enhancing fiber–matrix adhesion in natural fiber reinforced composites. Many researchers in the past have developed composites using natural fibers such as bamboo [6], coir [7], sisal [8] and banana [9]. The mechanical properties of the composites depend on fiber length, weight ratio, fiber orientation and interfacial adhesion between fiber and matrix [10]. Due to noise pollution in many places in the world, there is a great need to find new sound absorbing materials that are capable of reducing the noise level at various frequency ranges [11]. Traditionally, noise is controlled by using expensive and non-biodegradable sound absorbing materials such as glass wool, asbestos, polymer foams, fabric filler and polymer fibers, posing an added damage to the environment. As alternate, natural fibres like jute, cotton, kenaf, bamboo, flax, ramie, sisal, coir, luffa and hemp obtained from renewable resource can be used as a cheap, biodegradable and recyclable sound absorbing materials [12]. The thermal stability of any natural fiber composite may also impose limitations in applications at temperatures that cause degradation of the fiber organic structure. In principle, the temperature not only degrades the structure, but also affects most properties of the natural fiber composites. A complete understanding of these effects requires a review on the composite basic thermo-gravimetric, i.e., weight loss with increasing temperature characterization [13]. In this study, we have developed novel composite material using betelnut fiber reinforced with unsaturated polyester. The effect of chemical treatment onto betelnut fibers on mechanical, sound absorption and thermal properties of composites has been investigated. The reinforcing property of the alkali treated fiber was also compared with that untreated fiber.

2. Experimental

2.1. Materials

Betelnut is the fruit of Areca palm tree (Areca catechu), a species of palm. The fruits of betelnut are in form of round or oval in shape, with the color of golden yellow to orange. The betel nut husk is the fibrous part of the fruit, which equals to approximately 60 to 80 % of total volume and weight of betelnut.

Table 1 Average main chemical constitution of Betelnut fiber [14]

Fiber	Alpha Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Ash (%)	Other materials (%)
Betelnut	53.20	32.98	7.20	9.2-15.4	1.05	3.12

Matured betelnut fruits were obtained from a local market, Kuching, Sarawak, Malaysia. Betelnut fruits were soaked in water at room temperature for 5 days to loosen the fibre from the husk. The Betelnut fibers were separated manually from the nut portion by a hand stripping method and washed thoroughly with distilled water before dried in an oven at 70^{o}C for 24 h. virgin fiber extracted from betelnut fruit shell was socked in 5% detergent solution for 1 h [15]. The detergent washed fibers was also washed with distilled water for several times. The detergent washed fibers was soaked in 5% of NaOH solution for 48 h at room temperature then washed for 5 times with distilled water and then fibers were oven dried at 80^{o}C for 24 hours. In this research commercially available unsaturated polyester resin with the trade name of "Reversol P9509" supplied by Revertex Malaysia Sdn. Bhd. Company was used as the matrix material. This type of resin has a rigid, low reactivity, and thixotropic general purpose orthophthalic characteristics. For curing the matrix needs to be mixed with a curing catalyst, namely methyl ethyl ketone peroxide (MEKP) at a concentration of 1% by weight ratio of the matrix

Fig.1. Step of betel nut fiber preparation a) Raw betelnuts, b) Betelnuts soaked in water for ripening, c) Extracted fiber, d) Random size fibers

2.2. Preparation of the composite material

Betelnut fibers and Unsaturated polyerster composites were prepared in the different ratio of 5:95, 10:90, 15:85, 20:80 (Betelnut fiber wt%:Unsaturated polyester wt%) using the cold press moulding technique. A circular mould with a diameter of 25 mm and thickness of 2, 4 and 6 mm was used to fabricate the sound absorption specimens. For the tensile test, a mould with a thickness of 5 mm and cross-sectional area of 72.5 mm^2 was used. The inner surfaces of the mould were greased with a thin layer of wax as a release agent. The known weight of unsaturated polyester resin is mixed with 1% of MEKP catalyst to initiate polymerization of polyester resin and stirred properly. The known weights of betelnut fibers were mixed with a solution of catalyst and polyester resin. The mixture is then placed in the mould, making sure that the mould cavity is properly filled. A steel roller was used to distribute the fibers uniformly and to release the air bubbles from the composites. This procedure was repeated until a required thickness was achieved. A pressure of about 50 kPa was applied on the top of the mould to ensure that the bubbles

were forced out then the composite is then left to cure for about 24 hours at room temperature. The mould was opened to remove the composites.

2.3. Testing of Composite Material

Tensile testing was performed with a LS-28011-50 Universal Testing Machine T-machine Technology Co., LTD, Taiwan using ASTM D638 as the control specimen. The sound absorption properties of the composites were assessed using a locally fabricated and calibrated two-microphone transfer-function method according to ASTM E1050-10 as shown in Fig. 2.

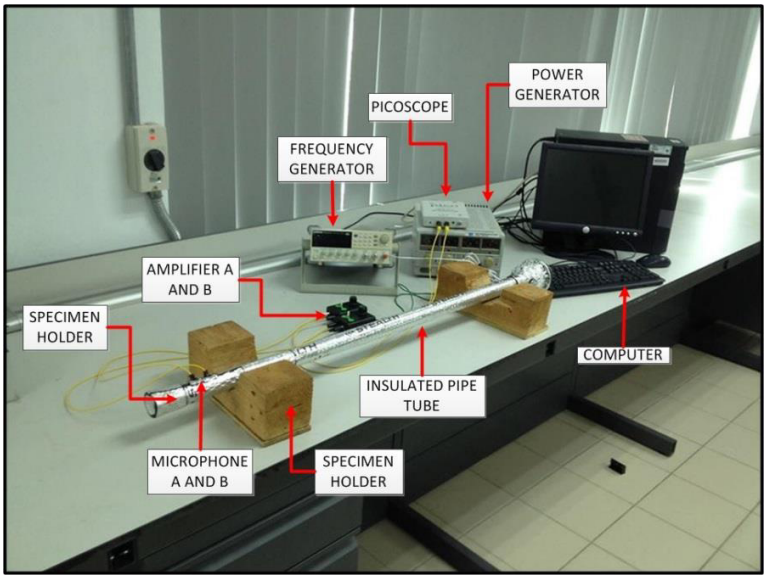

Fig.2. Impedance tube – Two microphone transfer function method

Two different diameter impedance tubes were used to cover the full frequency range. Tube with 80 mm diameter was used to cover measurements over the frequency range from 150 Hz to 1800 Hz and a 50 mm diameter tube are used to cover measurements in the frequency range 400 to 6000 Hz. The TGA was performed on a TA-60WS workstation analyser (Shimadzu Corp.; Kyoto, Japan) at a heating rate of 10 °C/min. Specimens were examined under flowing nitrogen (80 mL/min) over a temperature range of 30 to 900 °C. The morphological studies of the chemically treated betelnut fibres were observed using a JEOL JSM-6390LA SEM (Tokyo Japan) with a field emission gun and an accelerating voltage of 5 kV to collect images of the surface of composites. The test specimens were sliced and mounted on aluminium stubs with double sided adhesive tape and sputter coated with gold for 5 min to a thickness of approximately 10 nm under 0.1 torr and 18 mA to make the sample conductive. The FTIR spectroscopy was performed on a Shimadzu FTIR-8101 spectrometer in the range from 4000 cm^{-1} to 400 cm^{-1}. The FTIR was used to collect and understand the functional groups of the composite materials.

3. Results and Discussion

3.1. Effect of fiber content on sound absorption of composites

The sound absorption data were obtained with an impedance tube in the frequency range of 300 to 6000 Hz. All measurements were performed in accordance with ASTM E1050-10 using the standard setup depicted in Fig. 2. Figure 3 shows the frequency dependence of the sound absorption obtained for the composites tested. The performance of sound absorbing materials in particularly is evaluated by the sound absorption coefficient (α) [16].

The sound absorption coefficient was defined as the ratio between the non-reflected (absorbed) and the initial (incident) sound intensities, with values varying from zero to one, where zero represents full reflection, or no sound absorption by the composites tested, and one represents full absorption [17]. The composites are known to exhibit better sound absorption coefficients compared with the homogenous materials such as pure metals. The fact results from the additive of all kinds of acoustic energy losses [18]. The sound wave propagated through the inhomogeneous medium interacts with a great number of suspended particles, which differ by the density, compressibility and thermo-physical parameters from the matrix. This leads to the additional acoustic energy losses compared to that in the matrix.

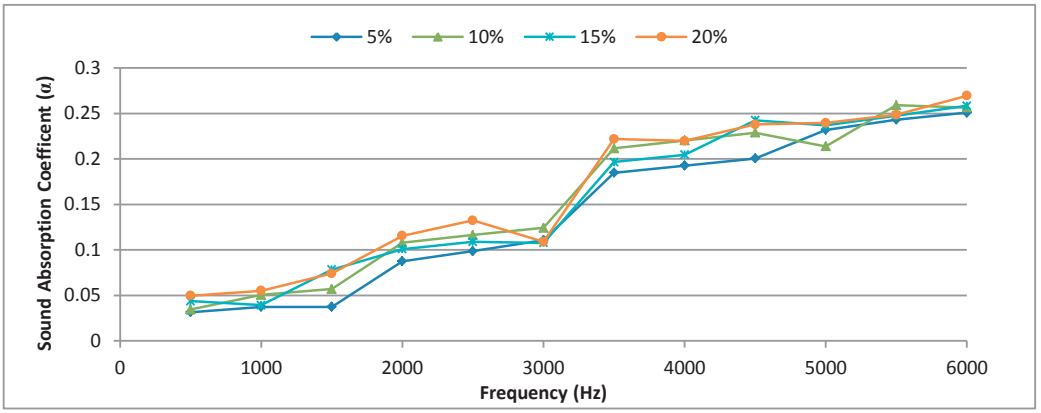

Fig.3. Effect of betelnut fiber content on sound absorption coefficients of composites

The composites with 5 wt.% of betelnut fibers was characterized by the relatively weak sound absorption. The values of sound absorption coefficient α vary between 0.03 to 0.20 with the tendency of increasing with increasing frequency. However, with increasing fiber content, an increase in sound absorption coefficient can be observed, especially at higher concentrations [19]. The composites with 20 wt% of betel nut was characterized by high sound absorption, an absorption peak up to 0.27 were measured.

3.2. Effect of thickness on sound absorption of composites

In figure 4, the variation of sound absorption coefficient against frequency is presented, for 3 different thicknesses of composites. A sound absorption coefficient was improved by increasing thickness of composites [20]. The sound absorption coefficients did not indicate significant differences within the frequency range of 300 to 1500 Hz for 2, 4 and 6 mm thickness.

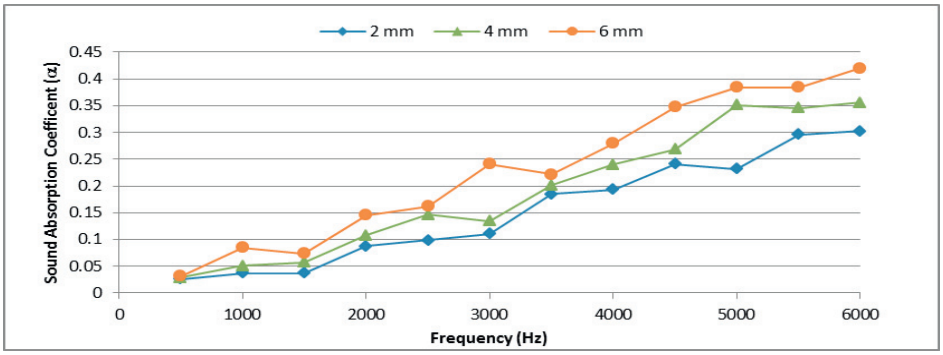

Fig.4. Effect of thickness of composites with 20% betelnut fiber on sound absorption coefficients.

This is due to, few sound energy converted into heat to achieve energy consumption due to thickness of the framework material and the smaller volume of air in the hollow structure units within the composites at low frequency range of incident wave [21]. Increasing the thickness of the composites, results in a increase in sound absorption coefficients, reaching to a maximum of 0.42 at 6000 Hz. According to Rayleigh mode of sound absorption mechanisms for porous materials, materials thickness enhanced acoustic impedance [22]. Numerous studies that dealt with sound absorption in porous materials have concluded that low frequency sound absorption has direct relationship with thickness. The effective sound absorption of a porous absorbers is achieved when the material thickness is about one tenth of the wavelength of the incident sound [23].

3.3. Mechanical Properties of composites

The variation of tensile strength as a function of fiber loading is represented in the figure 5. It was observed that the mechanical properties of the untreated betel nut/Unsaturated polyester composites increased linearly with the increase in fiber loading from 0% to 15%. This great increase in the mechanical strength is primarily attributed to reinforcing effect imparted by the fibers, which allow a uniform stress sharing from continuous polymer matrix to dispersed fiber phase [24]. However, noticeable decrease in mechanical strength of the composites was observed, as the fiber loading was increased from 15% to 20%. This decrease in the mechanical properties at high fiber loading implied poor fiber matrix adhesion which promoted micro-crack formation at the interface as well as non-uniform stress transfer due to fiber agglomeration within the matrix [25].

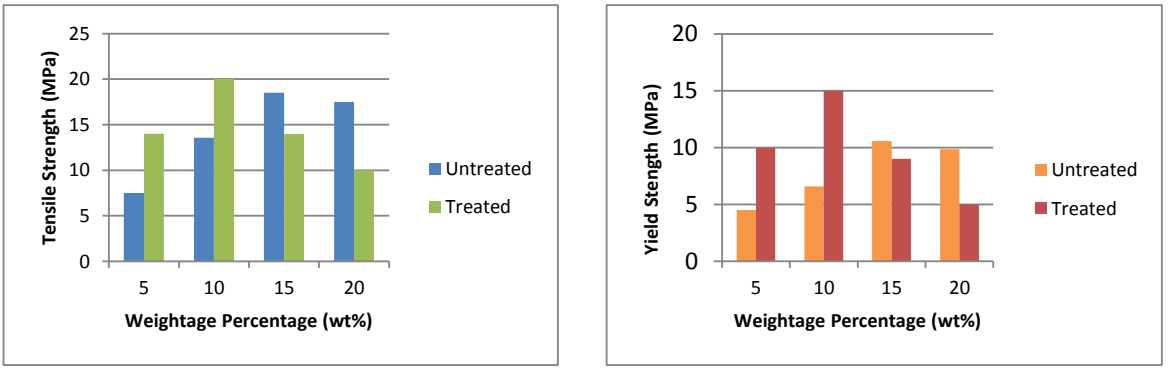

Fig.5. Variation of tensile strength of composites with fiber content and fiber treatment

Similar investigations have also been reported by Liu et al. [13], stated that the reason behind the increasing tensile strength is due to the attribution presence of fiber which creates a dispersed matrix that allowed uniform distribution stress on the materials. Apart from that, the decrease in the tensile strength is due to over presence of fiber in the material that contributed in initiating the crack which can cause non-uniform stress transfer due to the fiber agglomeration within the jute matrix [13]. As compared with the morphological analysis and FTIR, it showed that the changes in the lignocelluloses characteristic of the treated reinforced betel nut fiber composites also give an impact on the result of the tensile and yield strength. Balakrishna et al. [14] stated that the chemical treatment indirectly reacted to remove the moisture and impurity of the fiber and increase the strength of the fiber.

3.4. Thermal Analysis

The thermal degradation of natural fibers is a crucial aspect in the development of their composites and thus has a bearing on the curing temperature in the case of thermosets and extrusion temperature in thermoplastic matrix composites [26]. Thee composites reinforced with untreated fibers and treated by sodium hydroxide were evaluated by thermogravimetric analysis (TGA) for verification of the thermal characteristics of the materials. The thermal degradation of all the composites took place within the programmed temperature range of 30 to 900oC as shown in

fig. 6. Both treated and untreated fiber composites showed a two step decomposition process [27]. The thermal stability of treated fiber composites are higher than that of untreated fiber composites. This is also clear from the weight loss of these composites at different temperatures. The better thermal stability of treated fibers composites is related with the superior thermal stability of treated fibers. Another factor that contributes to the higher thermal stability of treated fiber composites is the improved fiber/matrix interactions, which generally generate additional intermolecular bonding between fiber and matrix [28].

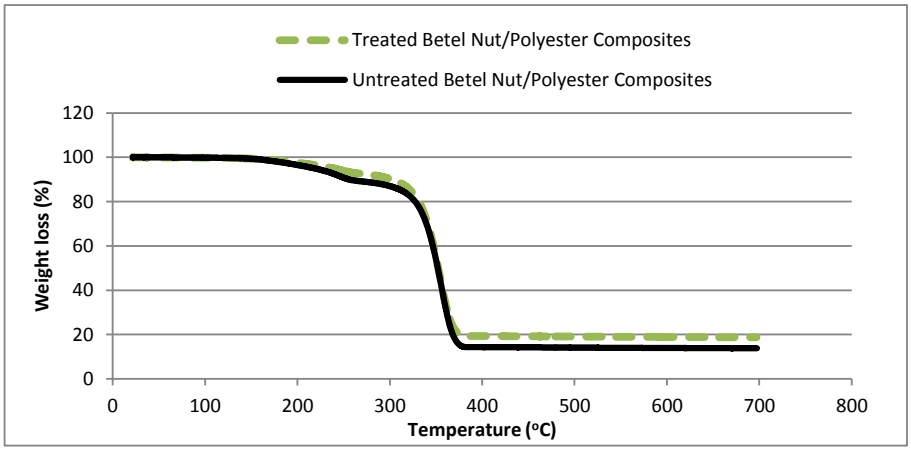

Fig.6. TGA curves of Polyester composites reinforced with treated and untreated betelnut fibers

An initial weight loss of less than 10 wt.% was observed in untreated betelnut fibers reinforced unsaturated polyester composites below 110°C and for treated fibers shifted between 120 – 140°C. This was usually associated with evaporation of water from the fiber surface. In practice, even if a natural fiber undergoes oven-drying before incorporation into a polymer composite, the total elimination of water is not possible because of the hydrophilic characteristic of the fiber. Sodium hydroxide treatments partially extract the highly hydrophilic hemicellulose, considered as greatest responsible for water absorption [29]. The decomposition of hemicellulose occurred between 210°C and 250°C. The final degradation which corresponds to the decomposition of the lignin, occurred at 230 – 380°C. The thermal degradation in a natural fiber composite is a complex process, which depends not only on the comparative thermal stability of fiber and matrix but also on the experiment atmosphere. In petroleum based polymers such as polyester possess a high degree of biodegradability resistance under nitrogen atmosphere compared with air [13].

3.5. Characterization of surface modified betelnut fibers

Both the untreated and treated betelnut fibers were characterized by FTIR spectroscopy to confirm the chemical reactions with the lignocelluloses elements of the fibers. FTIR spectra of untreated and treated betelnut 15wt.% fiber loading in the region 4000 to 700 cm-1 wavenumbers were presented in fig. 7. Plants comprise up to 80% of their dry weight of carbohydrates, with the most important including cellulose, starches, pectin and sugars, such as glucose and sucrose. The mid-infrared assignments of common plant carbohydrates are cellulose 1170-1150, 1050, 1030 cm-1, lignin 1590, 1510 cm-1, hemicellulose 1732, 1240 cm-1, pectin 1680-1600, 1260, 955 cm-1, β-D-cellulose 916, 908 cm-1. The infrared analysis of plant materials has traditionally relied upon the use of harch chemicals and modification of the intractable cell walls [30].

The sharp peak observed in untreated betel nut fiber at 1722.43 cm-1 is due to C=O stretching vibration of carboxylic acid and ester components of hemi cellulose. In sodium hydroxide treated fibers the peak 1722.43 cm-1 corresponding to C=O stretching vibration of hemicellulose disappeared owing to structural change [31]. This may be attributed to dissolution of hemicelluloses in chemical solution. In treated fibers, the intensity of the bands 1265

cm-1 from O-H in plane bending was reduced because of the formation of glycoside bonding. In chemical treatment, the hydrogen of droxyl group was substituted by acetyl groups, resulting in lower hydroxyl groups able to carry through hydrogen bonds. After chemical treatment there was a decrease in OH band and its displacement from 3400-3700 cm-1[27]. The treatment had removed most of the lignin and hemicellulose component and the treatment had changed the hydrophilic of the fibers to hydrophobic nature.

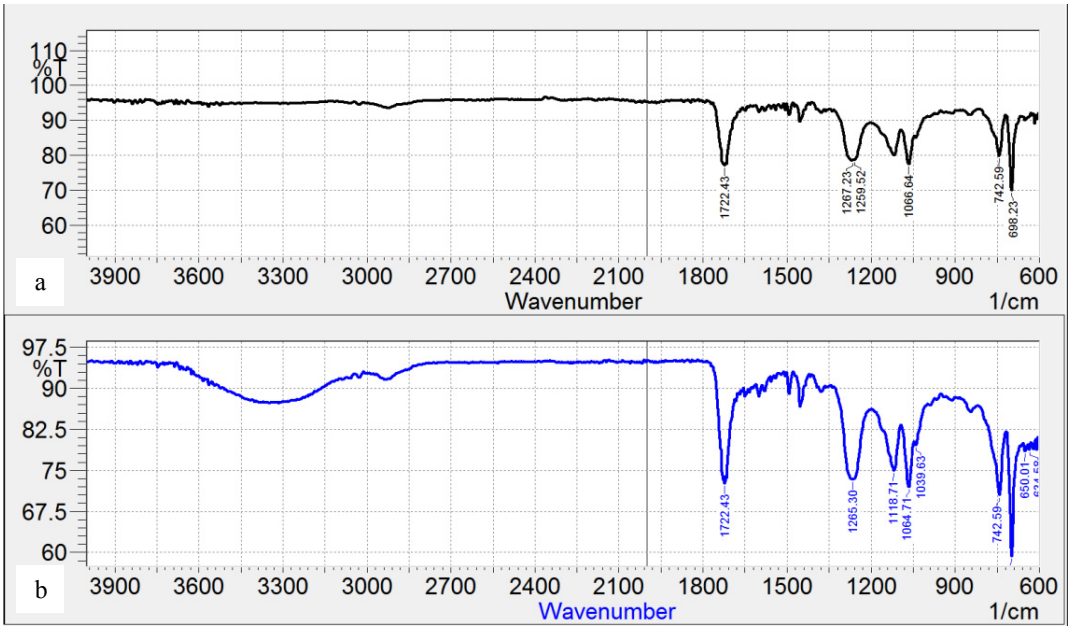

Fig.7. The FTIR spectra of Betelnut fibers a) Treated, b) untreated

3.6. Morphology Analysis

The SEM micrograph of untreated and sodium hydroxide (NaOH) treated betel nut fibers were shown in Fig. 8a and 8b. Figure 8a showed some gaps between the fiber and matrix in the untreated composites, indicating that there was no bonding between the fiber and matrix. The sodium hydroxide treated fiber composites showed improved fiber matrix adhesion as shown in figure 8b. The SEM micrographs confirmed the chemical modification and its influence in the morphological aspects of fibers. Because of chemical modification, the mechanical and thermal properties could be strongly influenced. Due to alkali treatment the impurities present in the fiber surface were removed and the fiber stands are getting separated which lead to the rough surface which affect both the mechanical and sound absorption properties of the composites.

The fracture surface morphology of the composites was shown in figure 8d, It can be observed that the fibers were broken off, which indicated worthy interfacial adhesion between the fiber and matrix. It was reported that the interfacial interactions affects the mechanical properties of the composites [32], which has been confirmed by the tensile test results discussed in the previous section. Mechanical interlocking is one of the bonding mechanisms that occur between fiber and polymer matrix in fiber reinforced composites. The mechanical interlocking involves diffusion of polymer matrix into porous fiber surface. The polymer matrix will flow into the porous fiber surface and the embedded polymer will solidify in the pores and become inextractable [33]. As shown in fig. 8c, a lot of short betel nut fibers were arrayed with random distribution in the unsaturated polyester matrix composites. This random distribution can let most of the sound waves hit the micro-pores of fiber bundle and strengthen the sound absorption effect. These special structures and the distributions are the main reason for the sound absorption coefficients of composites.

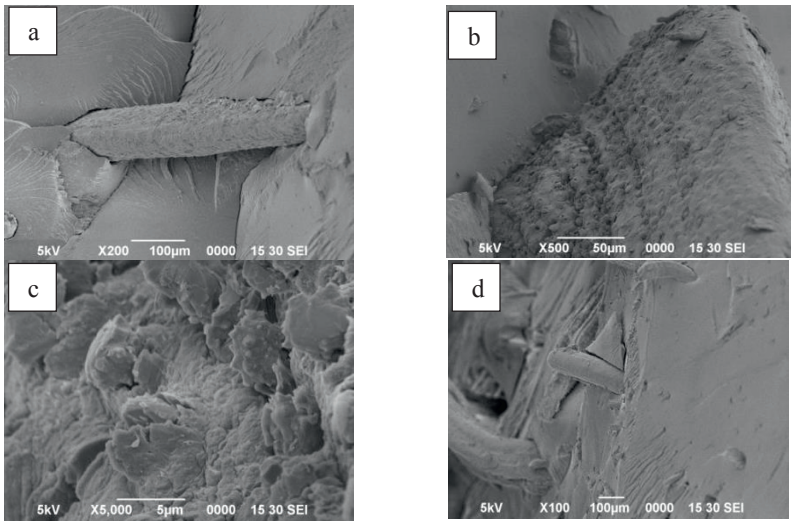

Fig.8. SEM Micrographs of Betelnut fiber a) Untreated, b) Treated, c) Distribution of fibers, d) Broken off fibers.

4. Conclusions

The acoustical, mechanical and thermal properties of betel nut fiber reinforced composites were studied as a function of fiber surface treatments. Composites prepared at 10% fiber loading and 5% NaOH treatment showed optimum mechanical strength. In general it is possible to enhance the properties of fiber reinforced composites through fiber surface modification. This is primarily a result of improved adhesion and enhanced polar interactions at the fiber matrix interfaces. FTIR spectroscopic studies revealed that surface modification of the fibers occurs after every fiber treatment. Addition of lignocelluloses materials into the pure unsaturated polyester increases the sound absorption coefficient of composites. Morphological studied by SEM reveal the micro-phase separation in the betel nut fibers/polyester composites, the porosity of the betel nut fibers and the distribution of short betel nut fibers in the unsaturated polyester composites. These special structures and the distribution are the main reason for better sound absorption. It was observed that when the thickness of the composites increases the sound absorption coefficient also increases. Untreated fiber composites exhibited lower thermal stability compared to sodium hydroxide treated composite

Acknowledgements

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