

Morphology and Thermal Properties of Alkali Treated Pineapple Leaf Natural Fiber and Betel Nut Husk Natural Fiber

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Abstract: In present research, natural fiber obtained from betel nut husk and pineapple leaf was chemically treated with 5% NaOH. Both raw and chemically treated fibers were subsequently characterized using structural (FTIR) and (SEM) and thermal (TGA) analysis. Fourier analysis showed the presence of (NaOH) group both in the raw and treated pineapple leaf fiber. It also shows the removal of hemicellulose in the treated betel nut fiber. Scanning electron micrographs revealed rougher surface and deeper pores in case of alkali treated betel nut fiber. However, the surface of the treated pineapple leaf fiber was smoother as compared the raw pineapple leaf fiber. Thermogravimetric analysis indicated that thermal stability of both treated fibers was slightly higher than those of the corresponding raw fibers.

Keywords: *Pineapple leaf fiber; Betel nut husk fiber; Morphology; Thermal properties*

Introduction: Natural fibers are gaining more interest in research than synthetic fibers due to environmental concerns. Synthetic fibers were commonly used in composites fabrication, owing to their excellent mechanical properties. However, synthetic fibers are non-biodegradable; hence many attempts are made to find alternatives for synthetic fiber. These natural fibers include flax, hemp, jute, sisal, kenaf, coir and many others. The various advantages of natural fibers are low density, low cost, low energy inputs and comparable mechanical properties and also better elasticity of polymer composites reinforced with natural fibers, especially when modified with crushed fibers, embroidered and 3-D weaved fibers [1]. These fibers are less hazardous to humans during fabrication and handling as compared to synthetic fiber; hence it is a good option to employ natural fiber as reinforcement in polymer composite. Betel nut is the fruit of Areca palm tree (*Areca catechu*), a species of palm. Betel nut husk fiber is a type of agro-waste from commercial crops that appears to be a good alternative to synthetic fiber. BNH fibers are obtained from the husk of the betel nut fruit. The husk of the betel nut is a hard fibrous portion covering the endosperm. It constitutes 30-45% of the total volume of the fruit. Betel nut husk fibers are predominantly composed of hemicelluloses and not of cellulose. BNH fibers contain 13 to 24.6% of lignin, 35 to 64.8% of hemicelluloses, 4.4% of ash content and remaining 8 to 25% of water content [2]. The fibers adjoining the inner layer are irregularly lignified group of cells called hard fibers and the portions of the middle layer contain softfibers. BHN fiber is highly hemicellulosic and is much greater than that of any other fibers. Betel nut fiber (Areca nut hush fiber) is characterized as extremely strong and light weight. The fibers are predominantly

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composed of cellulose and varying proportions of hemi cellulose, lignin, pectin and protopectin [2]. Pineapple leaf fiber (PALF) is obtained from the leaves of the plant, *Anannuscomosus*, belonging to the Bromeliaceae family. PALF has high specific strength and stiffness; it is hydrophilic in nature due to high cellulose content. Fresh leaves yield about 2 to 3% of fiber. PAL fiber is composed of many chemical constituents. It is multicellular lignocellulosic fiber containing polysaccharides, lignin in major amount, and some minor chemicals like fat, wax, pectin, uronic acid, anhydride, pentosan, colour pigment, inorganic substance, and so forth [3]. Fiber is collection of thin and small multicellular appears like a thread. These cells are tightly joined with the help of pectin. PALF constitute cellulose (70–82%) and arrangement of fibers is the same as in cotton (82.7%) [3]. The cellulosic molecules model of PALF is a three-dimensional structure and parallel to crystalline region of the fiber. Remaining parts of molecular structure are supposed to associate within amorphous regions. Pineapple leaf fiber (PALF) is vital natural fiber, which have high specific strength, rigidity, and flexural and torsional rigidity as much as jute fibers. Considering these exclusive properties of PALF, industries can use it as an outstanding alternative raw material in the prospect of reinforcing composite matrixes [3]. The objective of present research is to characterize raw and chemically treated pineapple leaf fiber and betel nut husk fiber by finding out their morphology and thermal properties. The properties of raw fiber are also compared with those of chemically treated ones.

Materials & Method:

Fiber Extraction: Pineapple fiber collects from the pineapple leaf. The leaves were smashed and then the fibers were collected. Betel nut fibers are collected from the upper portion of the betel. Then they were cleaned and dried.

Chemical Treatment: Both betel nut and pineapple leaf fibers are solution treated in 5% NaOH solution. 5% NaOH solution was prepared by adding 5 gm NaOH to 100 ml water in a beaker. Both were then treated in 5% NaOH solution and gave it to the furnace at 70°C for 2.5 hrs. The fiber was washed and dried in an oven at 80°C for 20 minutes.

Scanning Electron Microscopy (SEM): Surface morphology of the raw and solution treated fiber was observed by using a scanning electron microscope. The surface of the fiber was made conductive by giving platinum coating using a sputtering machine. The fiber was then observed in vacuum condition into the SEM machine.

Fourier Transform Infrared Spectroscopy (FTIR): Powder sample was made to perform FTIR, Potassium bromide (KBr) was taken as a reagent which was mixed with the sample (KBr: sample=100:1). The mixture was taken into a dice. Then it was pressed by a hand pressing machine. The infrared spectra of fiber were recorded on a Nicolet 380 spectrophotometer with co-addition of 32 scans.

Thermo gravimetric Analysis (TGA): TGA measures the amount of weight change of a material, either as a function of increasing temperature, or isothermally as a function of time, in an atmosphere of nitrogen, helium, air, other gas, or in vacuum. Inorganic materials, metals, polymers and plastics, ceramics, glasses and composite materials can be analyzed. Temperature ranges from 25°C to 900°C routinely. The maximum temperature was set at 500°C during present research.

Results and Discussion:

SEM Analysis of Pineapple Leaf Fiber and Betel Nut Fiber: SEM observations were conducted on the surface of the raw and treated BNH fiber. SEM micrographs, presented in Figures 1 (a) and 1 (b) show the morphologies of raw and treated BNH fiber surfaces. The removal of trichomes in alkali treated fiber was observed on the surface of the fiber, leaving pit like pores in BNH fiber. This is due to the removal of waxy layer on BNH fiber surface, creating deeper pores and rougher surface. SEM observations were conducted on the surface of the raw and treated PAL fiber. SEM micrographs, presented in Figures 1 (c) and 1 (d) show the morphologies of raw and treated PAL fiber surfaces. Figure 1 (c) shows cellular structure and these cells together form fibrils with tissues connected with each other at several locations along the length to form fibers. After alkali treatment the surface of the pineapple leaf fiber becomes cleaner and smoother than the raw fiber because of the removal of all impurities from the surface.

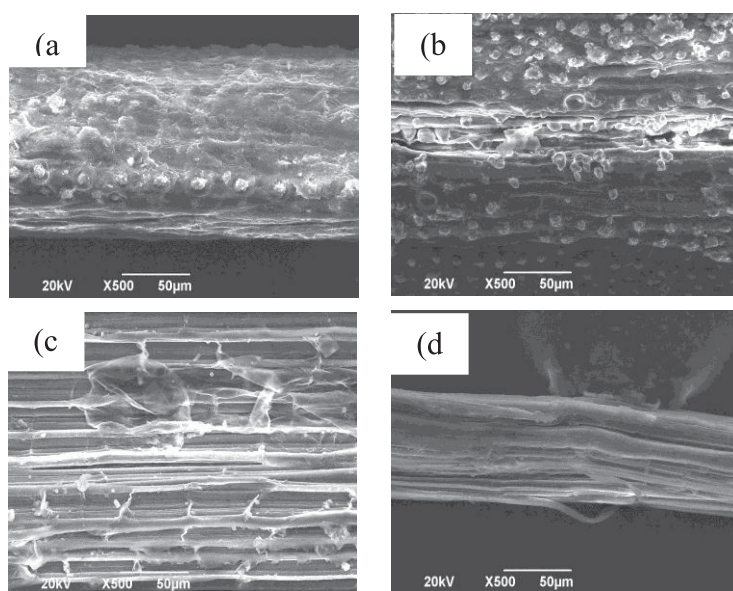


Fig. 1: SEM micrographs of (a) raw BNH, (b) alkali treated BNH, (c) raw pineapple leaf and (d) alkali pineapple leaf fiber.

FTIR Analysis of Pineapple Leaf Fiber and Betel Nut Fiber: Both the untreated and treated BNH fibers were characterized by FTIR. FTIR spectra of untreated and treated BNH fiber loading in the region 4000 to 400 cm^{-1} wave numbers are presented in Figures 2 (a) and 2 (b). 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 harsh chemicals and modification of the intractable cell walls [5].

FTIR figures clearly show the strong and broad characteristics band of (–OH) at the regions of 3600–3200 cm^{-1} , lignin and hemicelluloses at about 1735.6 cm^{-1} and (C–H) aromatic rings and alkane at 2920.6 cm^{-1} [6]. The alkali treated fiber (Figure 2 (b)) shows the characteristics band of (–OH) of high concentration at around 3404.7 cm^{-1} . Peaks for lignin and hemicelluloses are not very significant.

The alkali treated fiber (Figure 2(d)) shows the characteristics band of (–OH) of almost same concentration at around 3355.5 cm^{-1} and aromatic rings and alkanes at around 2919.7 cm^{-1} . In sodium hydroxide treated fibers the peak 1736.4 cm^{-1} corresponding to C=O stretching vibration of hemicellulose disappeared owing to structural change [7].

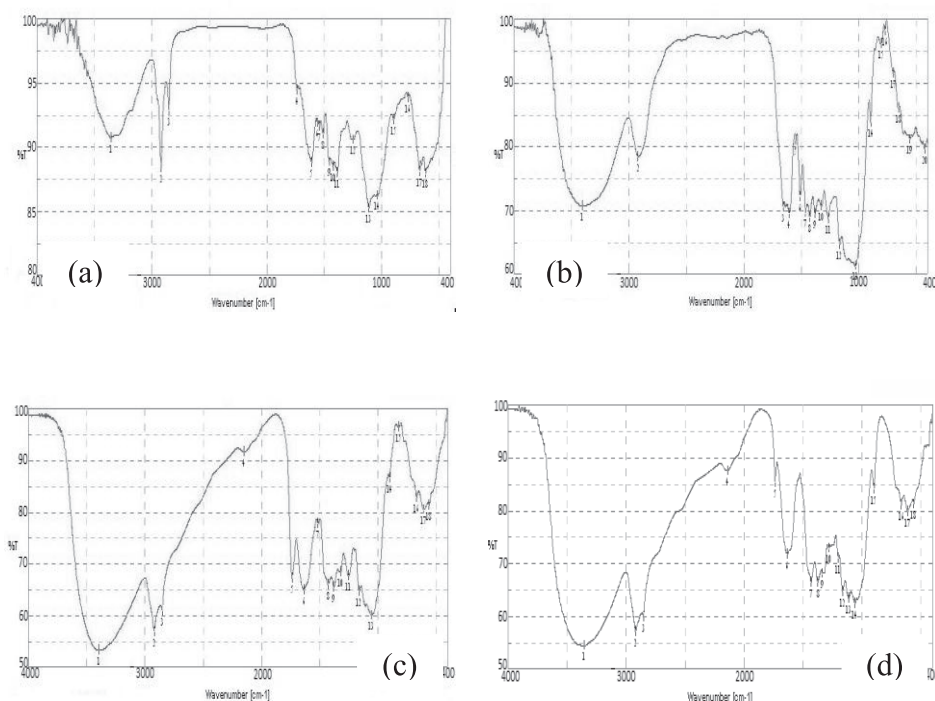


Fig. 2: FTIR spectra of (a) raw BNH, (b) alkali treated BHN, (c) raw pineapple leaf and (d) alkali pineapple leaf fiber.

FTIR spectra of raw and treated PAL fibers are shown in Figures 2 (c) and 2 (d). The IR spectrum for raw PAL fiber (Figure 2 (c)) clearly shows the strong and broad characteristics band of (–OH) at the regions of 3600–3200 cm^{-1} , lignin and hemicelluloses at about 1736.5 cm^{-1} and (C–H) aromatic rings and alkane at 2919.5 cm^{-1} [6].

Thermo Gravimetric Analysis of Pineapple Leaf Fiber and Betel Nut Fiber: Figure 3 shows TGA curves of raw and treated BNH and pineapple leaf fiber. In DTG curves of raw PAL and BNH fiber, the first very small peak <100 $^{\circ}\text{C}$ corresponds to the heat of vaporization of water absorbed in the fiber. Though this moisture loss peak has not shown any considerable change but the weight loss is slightly higher in the 5% NaOH treated fiber. Figure 3 (a) shows that in raw BNH fiber moisture loss occurs at

around 70 °C. Thermal degradation of raw BNH fiber occurs at around 225 °C to 275 °C. Figure 3 (b) shows that in treated BNH fiber moisture loss occurs at around 65 °C. Thermal degradation of treated BNH Fiber occurs at around 245 °C to 315 °C. Thermal degradation temperature increased in the case of treated fiber [8, 9]. In raw PAL fiber moisture loss occurs at around 70 °C. Thermal degradation of raw PAL fiber occurs at around 230 °C to 275 °C. In treated PAL fiber moisture loss occurs at around 50 °C. Thermal degradation of treated PAL fiber occurs at around 250 °C to 320 °C. Thermal degradation temperature is increased in the treated fiber.

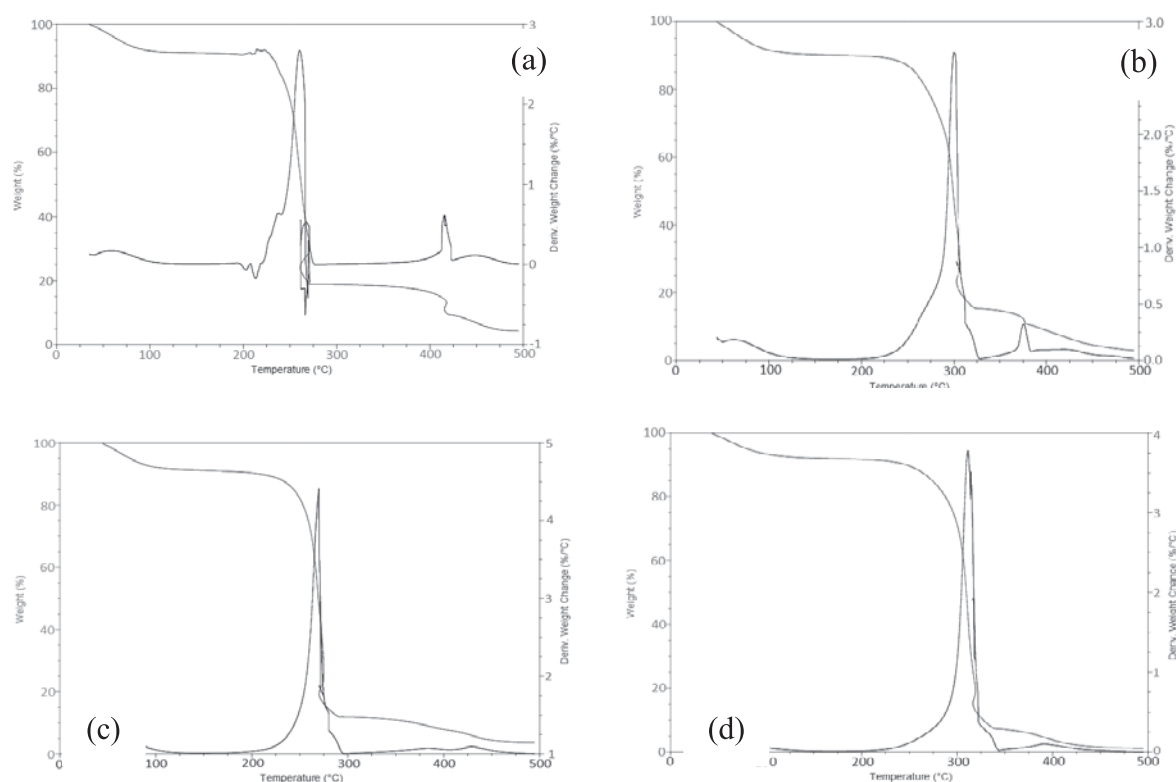


Fig. 3: TGA curves of (a) raw BNH, (b) alkali treated BNH, (c) raw pineapple leaf and (d) alkali pineapple leaf fiber

Conclusions: In present research chemical treatment of natural fibers affected the thermal, morphological and structural properties of the pineapple and betel nut husk fiber. From the SEM observation it was seen that the removal of trichomes in alkali treated betel nut husk fiber was observed which leave pit like pores in BNH fiber. This is due to the removal of waxy layer on BNH fiber surface, creating deeper pores and rougher surface than raw BNH fiber. But for another case after alkali treatment the surface of the pineapple leaf fiber become cleaner and smoother than the raw fiber because removal of all impurities from the surface. Fourier analysis showed the presence of (–OH) group both in the raw and treated pineapple leaf fiber. The IR spectrum for raw PAL fiber clearly shows the strong and broad characteristics band of (–OH), lignin and hemicelluloses, and (C–H) aromatic rings and alkane. IR spectrum also shows the removal of hemicellulose in the treated betel nut fiber. DTG curve of raw PAL-BNH fiber, the first very small peak corresponds to the heat of vaporization of water absorbed in the fiber. From the DTG curve it is clearly seen that the thermal stability of both fibers increased after alkali treatment.

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