COMPARATIVE STUDY OF FUNCTIONAL GROUPS IN NATURAL FIBERS: FOURIER TRANSFORM INFRARED ANALYSIS (FTIR)

Muhammad Khusairy Bin Bakri[1], Elammaran Jayamani[2]

[1,2]Faculty of Engineering, Computing and Science, Swinburne University of Technology Sarawak Campus, Jalan Simpang Tiga, 93350, Kuching, Sarawak, Malaysia.

Email: elammaranj@gmail.com[2]

Abstract

Throughout this research, the fundamental characterization of functional groups bond structure and the behavior of natural fiber before and after treatment were understood. Fibers were obtained by extracting it from the bamboo, betel nut and hemp plants. The Infrared spectrum of natural fiber were obtained and tested using Fourier transform infrared (FTIR) spectroscopy in the range of 400 cm-1 to 4000 cm-1 for untreated and alkali treated fibers. It was prepared quantitatively and qualitatively according to ASTM E168-06 and ASTM E1252-98 standards. Based on the Infrared spectral, the functional groups of the plant fiber were then collected and classified. The effect of chemical treatment was evaluated and discussed. Based on the result obtained, the bond structure of certain functional groups (i.e. hemicellulose, cellulose, and lignin) was removed and changed due to the alkaline treatment.

Index Terms—Natural Fiber, Alkaline Treatment, Infrared Spectral, Qualitative and Quantitative Anaysis.

I. INTRODUCTION

Fourier transform infrared (FTIR) spectroscopy is used to obtain infrared (IR) spectra of inorganic and organic materials. This technique measured the IR radiation absorption or transmittance by the sample material against its wavelength or wavenumbers. IR radiation causes the molecule structure to vibrate as the material being exposed to it. According to Mosiewicki et al. [1], the dipole moment of molecule in the material must be changed in order for a motion of vibration to be IR active. The total internal energy of a molecule can be resolved into the sum of vibrational, rotational and electronic energy levels. Furthermore, FTIR can also be used to determine the interactions between matter and electromagnetic fields in the IR region. The molecular components and structures can be identified through the IR absorption bands. The higher the changes in the magnitude of the molecule, the higher the intensity of the band created. The IR absorption bands are used to understand the characteristic of polymer interfaces and modified natural fibers in composite materials [1]. In general, a frequency will be strongly absorbed if its photon energy coincides with the vibrational energy levels of the molecule. IR spectroscopy is therefore a very powerful technique which provides fingerprint information on the chemical composition of the sample. FTIR spectrometer is found in most analytical laboratories.

A basic IR spectrum is essentially a plotted graph of infrared light absorbance or transmittance (vertical axis) vs. frequency or wavelength (horizontal axis). Typical frequency units used in IR spectra are reciprocal centimeters (it is also called as wavenumbers), with the symbol cm^{-1}. The IR wavelength units

are commonly given in micrometers (formerly called "microns"), symbol μm, which are related to wavenumbers in a reciprocal way. According to Stuart [2], the IR spectrum is divided into three sub-regions: the near-IR, approximately 14000 cm-1 – 4000 cm^{-1} (0.8 μm – 2.5 μm wavelength), the mid-IR approximately 4000 cm^{-1} – 400 cm^{-1} (2.5 μm – 25 μm wavelength) and the far-IR approximately less than 400 cm^{-1} – 10 cm^{-1} (25 μm –1000 μm wavelength). Besides that, based on Stuart [2] and Mosiewicki et al. [1], majority of analytical FTIR applications used are in mid-IR range, which is approximately around 4000 cm^{-1} – 400 cm^{-1}. The higher-energy near-IR can excite overtone or harmonic vibrations. The mid-IR may be used to study the fundamental vibrations and associated rotational-vibrational structure. The far-IR lying adjacent to the microwave region has low energy and may be used for rotational spectroscopy. The names and classifications of these sub-regions are pacts, and are only roughly estimated based on the relative molecular or electromagnetic properties.

Based on the research made by Ramadevi et al. [3], FTIR can be used to analyze the potential existing chemical bonding for the treated and untreated fibers. According to Rowell et al. [4], at 3400 cm^{-1} inside the large band, it is normally related due to the bonded O-H group stretching vibrations in the presence of carbohydrate (cellulose + hemicellulose) and hydroxyl groups. As for abaca fiber alkaline-treated, the removal of the hemicellulose component causes the 3347 cm^{-1} band assigned to the alcohol group to be reduced. Another peak due to the presence of alcohol group of cellulose OH deformation that appeared at 1310 cm-^{1} was reduced due to alkaline treatment. The FTIR can be used to investigate the bonding characteristics between natural fiber and matrices in the natural composite material. Based on the research made by Muniandy et al. [5], it showed that the formation of a bond between the silane coupling agents gives better interaction between the rubber matrix and rattan fiber. These peaks can be found in the region of 1586 cm^{-1} and 1172 cm^{-1} which corresponds to Si-O-C silanes and C-N stretching.

FTIR spectroscopy test on plantain fiber and composites are normally done to determine the strength of the composites and fiber before and after treatment. According to Ihueze et al. [6], there is a characteristic of O-H hydrogen bond stretching and vibration at a strong peak of 3406.40 cm^{-1}. Based on Ihueze et al. [6], this high peak of O-H stretching is due to the presence of intermolecular hydrogen bonding that tends to shift higher absorbance. The result obtained by Ihueze et al. [6] is coherent with the result observed by earlier works done by Clemsons et al. [7] and Rowell [8]. In this research, the fundamental behavior of natural fiber of bamboo, betel nut and hemp, before and after treatment were investigated. Respectively, all experiments were carried out using FTIR spectroscopy by quantitative and qualitative method and technique.

II. MATERIALS

Bamboo and betel nut were obtained from small local traditional agricultural and industrial factory in Kuching, Sarawak, Malaysia. Betel nut (*areca catechu*) was grown vastly in rural area of Sarawak typically and mainly Borneo Island. The fibers were extracted from the betel nut fruit rind (shells cover the nut of the fruits). Each betel nut fruit can produce around 2.5 - 2.8 g of fiber approximately depending on the size of the betel nut. Bamboo (*bambusa shrep*) is widely and wildly available in Sarawak forest. Bamboo is commonly used as a cooking utensil for preparing '*Pansuh Ayam*' (local Sarawak delicacy). The steam explosion technique was used in order to extract the fibers from raw bamboo trees. Hemp fibers were obtained from the skin or bark (or bast) of hemp (*cannabis sativa*) that imported from the local China market. Hemp is usually used to produce oil and create textile base products.

Sodium hydroxide (NaOH) with product code 'S/4920/AP1' was supplied by Fisher Scientific, UK. It is caustic soda pellet forms highly alkaline, odorless and completely soluble when react with water. Universal Indicator Solution was supplied by Fisher Scientific, UK.

III. METHOD

Fibers obtained were divided into two parts; untreated and treated fibers. For the treated fiber, the fiber underwent treatment using alkaline solution. The alkaline solution was prepared by

mixing 5wt % of sodium hydroxide with distilled water. The solution was stirred vigorously to ensure the caustic soda completely soluble in water. The alkaline solution was then poured into a beaker filled with fiber. The solution and fiber were stirred properly and were left for 30 min for reaction to occur. The fibers were filtered and wash with distilled water till the pH is neutralized. Universal Indicator Solution was used to test the pH level. Meanwhile, for the untreated fibers, the fibers were washed with distilled water to remove any dirt or impurity. Both untreated and treated fibers were then dried up in the oven at 60 °C for 48 hours to remove the moisture. The drying oven with model name 'ECOCELL EC55' with brand name 'MMM Group' equipped by Fisher Scientific, UK. The dried untreated and treated fibers were then blended into powder form. The samples were kept under 24±3 °C in temperature, 65 % in relative humidity and 101 kPa in pressure for 24 hours before testing. The composites were conditioned according to ASTM E41-92 [9] standards.

IV. FOURIER TRANFORM INFRARED (FTIR) SPECTROSCOPY TESTING

FTIR spectroscopy was used to understand the functional groups and its molecular bond structure in the range of 4000 cm^{-1} to 400 cm^{-1}. The FTIR spectroscopy with a model name of 'IRAffinity-1' was used and equipped by Shimadzu (Japan) Corporation. Approximately 0.5 mg of powder sample was mixed with approximately 100 mg of dry powder, potassium bromide (KBr) in a small agate pestle to create a sample pellet for FTIR spectroscopy. Then, the mixture sample pellet was then taken into the sample holder inside the spectroscopy. Vacuum pressure was applied onto the mixture sample pellet inside the spectroscopy. The vacuum pressure causes the removal of moisture inside spectroscopy. IR spectrum bands were obtained when laser of infrared projected onto the mixture pellets. All the information obtained was analyzed according to ASTM E168-06 [10] and ASTM E1252-98 [11] standards.

V. RESULTS AND DISCUSSION

A. BAMBOO FIBERS

Figure 1 shows the FTIR spectra of untreated and treated bamboo fibers. In Figure 1, the region of the broad absorption band at 3500 cm^{-1} to 3300 cm^{-1} for untreated and treated fibers are characterized with O-H stretching and H-bonded bond structure that mostly contains major functional groups of phenols, alcohols and waters. The O-H stretching and H- bonded broad absorption band in the region is decreased after alkaline treatment. These are due to decrease in functional group of phenolic or aliphatic hydroxyl in the fiber due to reaction with sodium hydroxide that promotes free hydroxyl that caused the addition of extra peak in free hydroxyl bond structure at 2873.06 cm^{-1} and 3597.24 cm^{-1}. A small peak at 2895.15 cm^{-1} for the untreated and 2912.51 cm^{-1} for the treated was attributed to the C-H stretching and O-H stretching bond structure that contains a functional group of alkanes (cellulose and lignin) and carboxylic acids. According to Khalil et al. [12], small peak in the region of the C-H stretching bond structure can also include a functional group of methyl (CH$_3$), methylene (CH$_2$), and aliphatic saturated (CH).
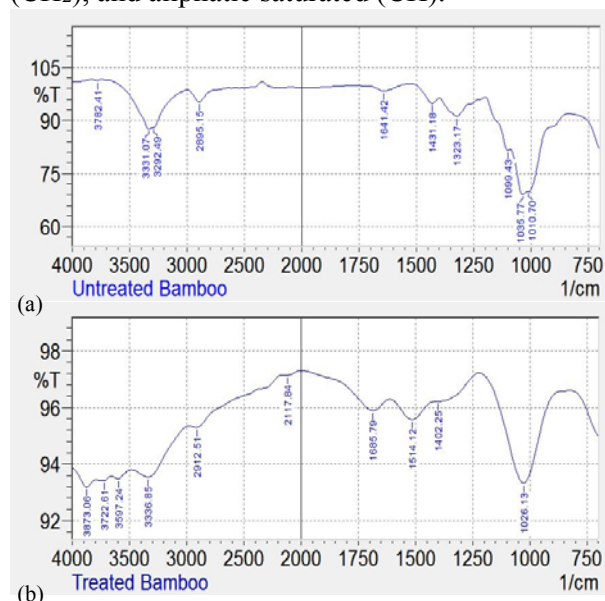

Figure 1 Fourier transform infrared spectroscopy analysis of (a) untreated bamboo fiber; and (b) treated bamboo fiber.

Table 1 shows the characterization of untreated and treated bamboo fibers as extracted from Figure 1. For untreated agarwood fiber, it shows the small peak band at 2117.84 cm^{-1}, are characterized as the C≡C stretching and C≡N stretching bond structure that contain functional group of nitriles and alkynes groups, the small peak at 1641.42 cm^{-1} is characterized as the C=C stretching bond structure from the functional

group of alkenes (lignin), the peak at 1431.42 cm^{-1} is characterized as the C-H bending bond from the functional group of alkanes (cellulose, hemicellulose and lignin), and the peak at 1099.43 cm^{-1}, 1035.77 cm^{-1} and 1010.70 cm^{-1} are characterized as the C-O stretching bond structure from the functional group of alcohol (cellulose, hemi-cellulose and lignin), carboxylic acids, esters and ethers.

Table 1 Characterization spectral analysis of bamboo fiber

Bond - Functional Group	Untreated bamboo fiber (wavenumber, cm^{-1})	Treated bamboo fiber (wavenumber, cm^{-1})
O-H stretching, Free hydroxyl - Alcohol, Water, Phenols	3782.41	3873.06, 3722.61, 3597.24
O-H stretching, H-bonded - Alcohol, Water, Phenols	3331.07, 3292.49	3336.85
C-H stretching, O-H stretching - Alkanes (CH; CH$_2$; CH$_3$), Carboxylic Acids	2895.15	2912.51
C≡C stretching, C≡N stretching – Nitriles, Alkynes	2117.84	-
C=C stretching - Alkenes (lignin)	1641.42	1685.79
C-H bending - Alkanes (cellulose; hemi-cellulose; lignin)	1431.42	1514.12, 1402.25
C-O stretching - Alcohol (cellulose; hemi-cellulose; lignin), Carboxylic Acids, Esters, Ethers	1099.43, 1035.77, 1010.70	1026.13

It is noticed that alkaline treatment caused the peak band at 2117.84 cm^{-1} to be disappeared. The disappearing of the peak may be due to the breaking of the triple bond structure of C≡C stretching from the functional group of nitriles and C≡N stretching bond structure of the functional group of alkynes into the double bond structure C=C and C=N. By compared with treated fibers, there was reduced in the intensity of the bands at 1099.43 cm^{-1} characterized as the C-O stretching band structure of the functional group of alcohol due to alkaline treatment. Furthermore, there are combinations of two different small peak band of 1035.77 cm^{-1} and 1010.70 cm^{-1} that form a single peak 1026.13 cm^{-1} after the alkaline treatment. According to Cao et al. [13] and Hinterstoisser et al. [14], the peaks band located in the region of 1100 cm-1 to 1000 cm^{-1} are also characterized as the C-O stretching bond structure of the functional group of glycosides linkage. The result obtained agreed with study reported by Xu et al. [15].

B. BETEL NUT FIBERS

Figure 2 shows the FTIR spectra of untreated and treated betel nut fibers. In Figure 2, the region of the broad absorption band at 3500 cm^{-1} to 3000 cm^{-1} for the untreated and treated fiber are characterized with O-H stretching and H-bonded bond structure that mostly contains major functional groups of phenols, alcohols and waters. The O-H stretching and H- bonded broad absorption band in the region is increased and the peak reduced after alkaline treatment. These are due to breaking of certain bond structure in functional group such as alkenes which forming O-H or H- bonded structure in the fiber after treatment. According to Ramadevi et al. [3], the O-H stretching, H- bonded and free hydroxyl were due to the presence of carbohydrates (hemicellulose and cellulose). The results obtained were also reported in Dwivedi and Mehta [16]. A small peak at 2906.73 cm^{-1} for the

untreated and 2939.52 cm⁻¹ for the treated was attributed to the C-H stretching and O-H stretching bond structure that contains groups of alkanes (cellulose and lignin) and carboxylic acids.

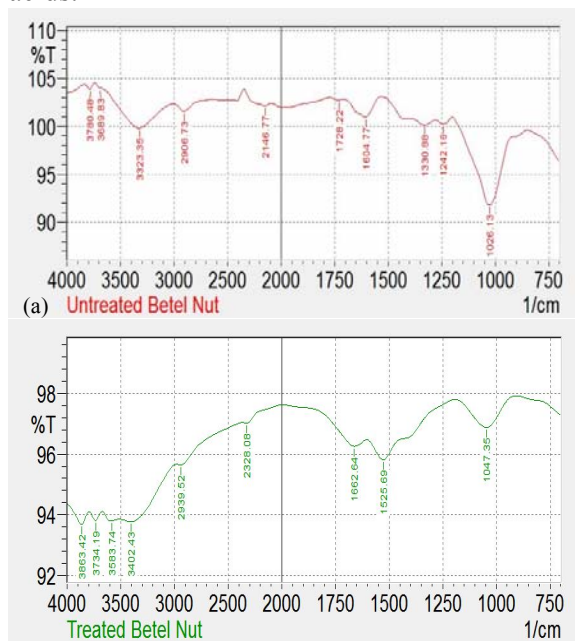(a) Untreated Betel Nut

Treated Betel Nut

Figure 2 Fourier transform infrared spectroscopy analysis of (a) untreated betel nut fiber; and (b) treated betel nut fiber.

Table 2 shows the characterization of untreated and treated betel nut fibers as extracted from Figure 2. For untreated betel nut fiber, it shows the small peak band at 2146.377 cm⁻¹ are characterized as the C≡C stretching and C≡N stretching bond structure from the functional group of nitriles and alkynes, the small peak at 1728.22 cm⁻¹ and 1604.77 cm⁻¹ is characterized as the C=C stretching bond structure from the functional group of alkenes (lignin), the small peak at 1330.88 cm⁻¹ and 1242.16 cm⁻¹ is characterized as the C-H bending bond structure of the functional group of alkanes (cellulose, hemicellulose and lignin), and the peak at 1026.13 cm⁻¹ are characterized as the C-O stretching bond structure of the functional group of alcohol (cellulose, hemi-cellulose and lignin), carboxylic acids, esters and ethers.

Table 2 Characterization spectral analysis of betel nut fiber

Bond - Functional Group	Untreated betel nut fiber (wavenumber, cm⁻¹)	Treated betel nut fiber (wavenumber, cm⁻¹)
O-H stretching, Free hydroxyl - Alcohol, Water, Phenols	3780.48, 3689.83	3863.42, 3734.19, 3583.74
O-H stretching , H-bonded - Alcohol, Water, Phenols	3323.35	3402.43
C-H stretching, O-H stretching - Alkanes (CH; CH₂; CH₃), Carboxylic Acids	2906.73	2939.52
C≡C stretching, C≡N stretching – Nitriles, Alkynes	2146.77	2328.08
C=C stretching - Alkenes (lignin)	1728.22, 1604.77	1662.64, 1525.69
C-H bending - Alkanes (cellulose; hemi-cellulose; lignin)	1330.88, 1242.16	-

Compared with the treated fibers, there was reduced in the intensity of the bands at 1330.88 cm⁻¹ and 1242.16 cm⁻¹ characterized as the C-H bending bond structure of the functional group of alkanes due to alkalization treatment. The alkaline treatment caused the breaking of the C-H bending bond structure of functional group alkanes that promote to H- bonding and free hydroxyl bond structure. Thus, this promotes extra band in that functional group of alcohol, water and phenols.

C. HEMP FIBERS

Figure 3 shows the FTIR spectra of untreated and treated hemp fibers. In Figure 3, the region of the broad absorption band at 3500 cm⁻¹ to 3000 cm⁻¹ for the untreated and treated fiber are characterized with O-H stretching and H-bonded bond structure that mostly contains

major functional groups of phenols, alcohols and waters. The O-H stretching and H- bonded bond structure of the broad absorption band in the region is decreased after alkaline treatment. These are due to decrease in functional group of phenolic or aliphatic hydroxyl in the fiber due to reaction with sodium hydroxide. A small peak at 2893.22 cm^{-1} for the untreated and 2893.22 cm^{-1} for the treated was attributed to the C-H stretching and O-H stretching bond structure that contains a functional group of alkanes (cellulose and lignin) and carboxylic acids.
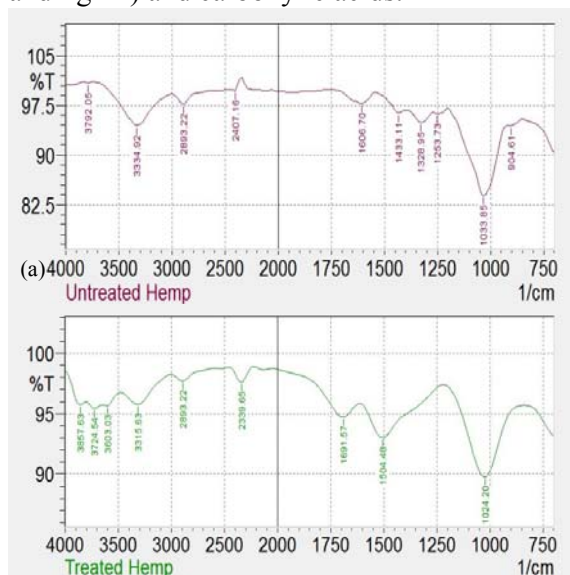

Figure 3 Fourier transform infrared spectroscopy analysis of (a) untreated hemp fiber; and (b) treated hemp fiber.

Table 3 shows the characterization of untreated and treated hemp fibers as extracted from Figure 4. For untreated hemp fiber, it shows the small peak band at 2407.16 cm^{-1} is characterized as the C≡C stretching and C≡N stretching from the functional group of nitriles and alkynes, a peak at 1606.70 cm^{-1} is characterized as the C=C stretching from the functional group of alkenes (lignin), small peak at 1433.11 cm^{-1}, 1328.95 cm^{-1} and 1253.73.11 cm^{-1} characterized as the C-H bending bond structure from the functional group of alkanes (cellulose, hemicellulose and lignin), the peak at 1033.85 cm^{-1} are characterized as the C-O stretching bond structure from the functional group of alcohol (cellulose, hemi-cellulose and lignin), and the small peak at 904.61 cm-1 characterized as C-H "oop" bond structure of the functional group of aromatic (lignin). The results obtained were reported almost similar to Garside and Wyeth [17].

Table 3 Characterization spectral analysis of hemp fiber

Bond - Functional Group	Untreated hemp fiber (wavenumber, cm^{-1})	Treated hemp fiber (wavenumber, cm^{-1})
O-H stretching, Free hydroxyl - Alcohol, Water, Phenols	3792.05	3857.63, 3724.54, 3603.03
O-H stretching, H-bonded, Free hydroxyl - Alcohol, Water, Phenols	3334.92	3315.63
C-H stretching, O-H stretching - Alkanes (CH; CH$_2$; CH$_3$), Carboxylic Acids	2893.22	2893.22
C≡C stretching, C≡N stretching – Nitriles, Alkynes	2407.16	2339.65
C=C stretching - Alkenes (lignin)	1606.70	1691.51, 1504.48
C-H bending - Alkanes (cellulose; hemi-cellulose; lignin)	1433.11, 1328.95, 1253.73	-
C-O stretching - Alcohol (cellulose; hemi-cellulose; lignin), Carboxylic Acids, Esters, Ethers	1033.85	1024.20
C-H "oop" – Aromatic (lignin)	904.61	-

By compared with treated fibers, there was reduced in the intensity of the bands at 1433.11 cm^{-1}, 1328.95 cm^{-1} and 1253.73.11 cm^{-1} characterized as the C-H bending from the functional group of alkanes due to alkalization treatment. Furthermore, there was a reduction in the intensity of the bands at 904.61 cm^{-1} characterized as C-H "oop" bond structure of functional group of aromatic. This caused disappearing of smell from the hemp fibers.

D. EFFECT OF ALKALINE TREATMENT

Sodium hydroxide (NaOH) was used widely in treatment of natural fibers. Its helps in modify the natural cellulose fiber either by bond structure or its functional groups. Alkaline treatment caused the increase in the amount of amorphous cellulose than crystalline cellulose. H-bonded bond structure in the fiber network structure was removed due to alkaline treatment. However, not all fibers tend to interact when reacted with sodium hydroxide. Alkaline treatment or mercerization is the most common method to produce quality fibers [18]. The mercerization is a process that allows natural fibers such as vegetable, wood and etc. to interact with low concentrated solution from strong base [19]. The process caused swelling structure due to changes in fine structure, morphology, mechanical and dimension properties [19].

Due to alkaline treatment, the alkaline sensitive hydroxyl groups (O-H bond structure) present in the natural fiber molecules were broken. It then react with water, phenols or alcohols molecules groups (H-O-H bond structure) and move in or out from the fiber structure depending on the characteristic of fiber toward the reaction of alkaline. Thus, the remaining of the reactive molecules indirectly forms the fiber cell of -O-Na bond structure between the cellulose molecular chains [20]. Thus, some hydrophilic hydroxyl groups are reduced and the fibers moisture resistance properties are increased. Some of the impurity, pectin, hemicelluloses, hemi-cellulose, lignin, wax and oil that covering were removed from the fibers as due to alkaline treatment [21]. Alkaline treatment also caused fibrillation that breaks the composites fiber bundle into smaller fibers and reduces the fiber diameter. Increase in the aspect ratio of the fiber lead to enhanced interface adhesion between fiber and matrix due to higher surface roughness. The sound absorption, mechanical, water absorption and thermal behaviors of the composites are improved significantly by this treatment. According to Kabir et al. [22], the treated fibers are known to have lower lignin content than the untreated fibers. Furthermore, the chemical treatment also partially removes the oil and wax that cover fibers and distension of crystalline cellulose order [22].

VI. CONCLUSIONS

Based on the result obtained, the bond structure in certain functional groups (this includes hemicellulose, cellulose, and lignin) was removed and changed due to the alkaline treatment. Furthermore, change in the bond structure of carbon and hydrogen gave an effect on the absorption properties of fiber. It is known that alkaline treatment removed the smell of the fiber and change the absorption properties of fiber. It becomes stronger and rougher than the untreated fibers. By understand the natural fiber characteristics; it can help improve the mechanical, acoustical, thermal and morphological properties of composites or related materials.

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