Hemicellulose and lignin removal on typha fiber by alkali treatment

Ikramullah, Samsul Rizal, Sulaiman Thalib, Syifaul Huzni
Department of Mechanical Engineering, Syiah Kuala University, Banda Aceh, Indonesia
E-mail: syifaul@gmail.com

Abstract. One of the methods commonly utilized to alter the surfaces of natural fibers for improving the interface compatibility among fiber and polymer matrix is by alkali treatment. Several natural fibers have been experimented with alkali treatments such as abaca, borassus and kenaf. There is a relatively few of literature that reports the FTIR investigation of Typha fibers. The purpose of this study is to determine the effect of alkali treatment on Typha fiber. Two of three bundle fibers are immersed in a 5% NaOH solution for one and two hours. The chemical structure of alkali-treated and untreated fibers are both being analyzed by Fourier Transform Infrared Spectroscopy (FTIR) instrument. The emergence of peak at 1155.36 cm⁻¹ in strong intensity denotes the C-O-C asymmetric stretching in cellulose compound. The lignin composition of the fiber is typified by the stretching band of C-O group at 1247 cm⁻¹. Meanwhile, the peak at 1735.03 cm⁻¹ wavenumber is allegedly C=O stretching evidencing the existence of hemicelluloses and pectin. The peaks which are suspected to be hemicellulose, lignin and pectin are no longer visible in alkali treated Typha fiber. Giving alkali treatment to Typha fiber has been successfully removed impurities (hemicelluloses and lignin), as approved by the FTIR analysis. This will lead to a better contact and bonding mechanism between fiber and polymer matrix.

1. Introduction
The advantages gained from using natural fibers as a composite polymer reinforcement are low cost, low density, low energy consumption, renewable and biodegradable [1]. However, natural fibers are less compatible with the polymer matrix due to moderate adhesion between natural fibers and matrix which make natural fibers do not stick well to hydrophobic polymers. On the other hand, natural fibers possessing disadvantages upon composite manufacturing process, i.e. poor moisture resistance, limited processing temperatures, the tendency of fibers to agglomerate into bundles, unequal matrix distribution, and poor wettability upon matrix which resulting weak interface adhesion [2].

Several chemical and physical modifications were tried to reduce the hydrophilicity of natural fiber, both modification aimed to increase fiber's wettability on polymers matrix and improving bonding mechanism between natural fiber and matrix. Physical and chemical modifications have their own different advantages and mechanisms to optimize the characteristics of the composite interface. Electric discharge, generally one of the physical modification methods that modify the surface of natural fibers and structural properties by inserting the bonds to the surface of the fiber, with the change of the fiber surface, bonding mechanism of natural fiber and matrix will increase [3].

1 syifaul@gmail.com
Meanwhile, chemical modification on natural fibers permanently transforms the properties of fiber by cross-linking cell walls [4].

One of the methods commonly utilized to alter the surfaces of natural fibers for improving the interface among fiber and polymer matrix by alkali treatment. The alkaline treatment is conducted by immersing the fiber in the water with NaOH solution. The advantage of alkali treatment in natural fibers is to make some of the hemicelluloses, lignin, pectin and wax disappear which cover the surface of the natural fiber [5]. The alkali treatment was capable of changing the smooth surface of the natural fibers to be rough, the roughness of the natural fiber surface resulting in better mechanical bonding. Furthermore, alkali treatment improves the bonding of natural fiber and matrix. The mechanical interlocking and bonding mechanism are the two factors responsible for the strength of the composite interface [6]. The alkali-treated natural fibers have been proved to optimize fiber and matrix interface bond which make composites being stronger compared to natural fibers that did not undergo alkali treatment processes. However, it should be noted the overuse alkali concentrations will damage the fibers [7].

Figure 1. (a) Typha plant; (b) untreated Typha fiber; (c) Alkali-treated Typha fiber 5 % 1 hour; (d) Alkali-treated Typha fiber 5 % 2 hour

The treatment with alkali has been experimented on various numbers of natural fibers such as abaca, borassus and kenaf [8]. There is a relatively few of literature that reports the FTIR investigation of Typha fibers. Typha plant is commonly found in swamps and is considered being parasitic and it is not widely used. The purpose of this study is to determine the effect of alkali treatment on Typha fiber.
2. Material and method

2.1. Materials
The Typha plants with 1.5-2 meters length were collected from swamps around Syiah Kuala districts, Banda Aceh. Typha trunks which are the base of the plant were manually extracted to obtain fibers, its trunk was hit by timber to ease the stripping of fibers. The fiber then stripped from its stalks by hand, afterward, the fibers were soaked in 5% NaOH solution for one and two hours, then fiber washed by water to quit the effect of alkali on fiber.

2.2. Fourier Transform Infrared Spectroscopy
The chemical structure of alkali-treated and untreated fibers were both analyzed by Shimadzu IRPrestige-21 Fourier Transform Infrared Spectroscopy (FTIR) device. All of the fiber samples were recorded in the 400 to 4000 cm⁻¹ wavelength to determine the chemical composition of fibers, such as cellulose, lignin, hemicellulose and other chemical compositions that may be present in Typha fibers.

3. Result and discussion
The effect of alkali treatment on Typha fiber is marked by the disappearance of hemicelluloses, lignin and pectin. It has been observed from the FTIR spectrum that the peaks that are assigned to be hemicelluloses, lignin and pectin have been dismissed from Typha fiber. Two hours of treatment appears to be ineffective because it can damage all contents of the fiber including cellulose, in which it is an important element that will withstand the load when the fiber becomes a reinforcement on the composite. Therefore, the proposed practical procedure is by retting Typha fibers in NaOH solution for an hour as it does not damage all components of the fiber.

![Figure 2. FTIR spectrum of untreated Typha fiber.](image)

Each peak of the absorption bands that resemble various chemical groups as summarized from [8] are presented in table 1. The characteristic of cellulose, lignin and hemicelluloses can still be found in untreated Typha fiber as shown in figure 2. The presence of hemicelluloses can be observed by the appearance of a peak at a wavenumber of 896.90 cm⁻¹ that indicates C-OH functional group
representing the relationship of β-glycoside, which is a bond between monosaccharide in cellulose and hemicelluloses. As can be seen from figure 2, the emergence of the peak (a) at 1155.36 cm⁻¹ in strong intensity denotes the C-O-C asymmetric stretching in cellulose compound. This peak usually appears at a spectrum range of 1070-1240 cm⁻¹. The stretching band of C-O group at 1247 cm⁻¹ (b), typifies the lignin composition in the fiber. Meanwhile, the peak (c) at 1735.03 cm⁻¹ wavenumber is allegedly C=O stretching evidencing the existence of hemicelluloses and pectin. The medium intensity of the C=O strain delineates the carboxyl and acetyl groups of high-concentrated hemicelluloses content.

**Table 1. FTIR Characteristic bands of Typha Fiber.**

| Wavenumbers (cm⁻¹) | Assignment and Remark | Possible Assignment |
|--------------------|-----------------------|---------------------|
| 896,90             | C-OH                  | The relationship of β-glycosidic is a bond between monosaccharide in cellulose and hemicelluloses |
| 1155,36            | C-O-C asymmetric stretch | Cellulose |
| 1247,94            | C-O stretching        | Lignin |
| 1735,03            | C=O stretching        | Hemicelluloses and pectin |

*Figure 3. FTIR spectrum of treated Typha fiber for 1 hour.*
The FTIR spectrum of alkali-treated Typha fibers for one hour and two hours are presented in figure 3 and 4. The peak (a) at 1155.36 cm\(^{-1}\) wavenumber which assumed to be cellulose is still clearly visible even after the treatment. Meanwhile, the peak (b) at a wavenumber of 1247 cm\(^{-1}\) which indicates the stretching of C-H in lignin is no longer detectable. Similarly, the peak (c) at 1735.03 cm\(^{-1}\) which allegedly hemicellulose and pectin have disappeared from Typha fiber. This indicates that hemicelluloses, lignin and pectin have been successfully removed from the fiber by alkali treatment.

From the results of FTIR spectroscopy analysis, it is confirmed that the alkaline treatment changes the three major chemical composition components of Typha fiber, namely; cellulose, hemicelluloses and lignin. The hemicelluloses content of Typha fiber is affected by the duration of alkaline treatment, because hemicelluloses are more sensitive to NaOH than cellulose or lignin. This can be assumed that increasing the duration of the alkaline treatment will eliminate the hemicelluloses content. However, longer duration of alkaline treatment, will decrease the level of cellulose, and increase the lignin content. The possibility of this occurrence is because the longer immersion time in alkaline will cause damage to cellular molecular structures [7].

4. Conclusion
The experimental results show that the untreated Typha fiber still has a lot of fiber contamination. The characteristic of cellulose, lignin and hemicelluloses can still be found in untreated Typha fiber. Giving alkali treatment to Typha fiber has been successfully removed impurities (hemicelluloses and lignin), as approved by the FTIR analysis. This will lead to a better contact and bonding mechanism between fiber and polymer matrix.

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