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ALKALI AND BLEACH TREATMENT OF THE EXTRACTED CELLULOSE FROM PINEAPPLE (*Ananas comosus*) LEAVES

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ABSTRACT

We successfully extracted cellulose from pineapple leaves (*Ananas comosus*) using alkali treatment and bleaching. Alkali treatment was done using aqueous sodium hydroxide while bleaching was done using acetate buffer and aqueous sodium chlorite. The extracted cellulose was characterized using Scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, Thermogravimetric analysis (TGA) and Differential thermal analysis (DTA). SEM micrographs revealed that alkali treatment removed the impurities in the pineapple leaf fibers and subsequent bleaching further purify the fibers leaving mostly cellulose only while hemicellulose and lignin are removed as revealed in the FTIR spectra. TGA spectra further revealed that alkali + bleached treated sample slowly degrade at temperature between 160 °C and 355 °C suggesting that small amount of hemicellulose and lignin are present as compared to the untreated sample.

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This finding was also confirmed in the DTA spectra suggesting that high quality of extracted cellulose is produced.

Keywords: Pineapple leaves; Cellulose; Alkali treatment; Bleaching.

1. INTRODUCTION

In recent years, intensive research has focused on the use of cellulose in a wide range of applications including composites, filter, film, transparent sheet, cosmetics, food, and coating mixtures [1]. Cellulose, the most abundant natural polymer on Earth, is extensively considered as a nearly inexhaustible raw material with interesting structures and properties [2]. The basic chemical structure of cellulose showing a long chain polymer with repeating units of D-glucose, called pyranoses which are joined by single oxygen atoms (acetal linkages) between the C-1 of one pyranose ring and the C-4 of the next ring, called β 1-4 linkages [3]. Each β 1-4 glucopyranose bears three hydroxyl groups and is able to form intra and intermolecular hydrogen bonds that play a major role in determining the physical properties of cellulose [4]. Currently, the interest in cellulose based-materials has been increasing due to the demand for renewable resources and growing on environmental awareness [5].

Cellulose is widely distributed over a variety of sources, including bacterial sources, such as algae (e.g., valonia), fungi, and even amoeba (protozoa), marine animals (e.g., tunicates), and plants (e.g., wood, cotton, or wheat straw) [6]. However, bacterial cellulose have some issues namely, high price, lack of large-scale production capacity, timely expansion and maintenance of the cell culture for production that have prevented large-scale commercialization so far [7]. Also, marine animal resources pose a great concern over the potential risks by these organisms; species are at risk of becoming extinct or endangered, may cause aquatic animals to starve and draining the life from the water [8]. Among these conventional sources of cellulose, plants have been greatly considered because of its low cost, environmental friendly, biodegradability, availability and sustainability [9]. These advantages of plants can be considered a good potential source for cellulose production. There are several plants such as cotton, wood, bamboo, flax, hemp, sisal, and jute that are rich in cellulose [10]. However, in the last decade research focusing on the use of cellulosic waste as filler has growing rapidly and generate many environmental problem such as deforestation and reduces the biodiversity of harvested forest. Agro-wastes materials such as coconut husk fibers, soy bean, wheat, straw and soy hulls [11] have been studied as a resource in the production of

cellulose fiber. Although a variety of natural plant fibers were investigated in detail, the use of pineapple leaves as a natural source for the production of cellulose has not been explored yet. In the Philippines, pineapple production is one of the main agricultural industries while pineapple leaves are considered agricultural wastes. In this work, extraction of cellulose from pineapple leaves is investigated. Alkali treatment and subsequent bleaching were done to produce high quality cellulose.

2. RESULTS AND DISCUSSION

Figure 1 shows the SEM micrographs of the alkali + bleached treated, alkali treated and untreated pineapple leaves. It is very apparent as shown Fig. 1(a) that alkali treatment + bleached treated, alkali treated and untreated pineapple leaves. It is very apparent as shown Fig. 1 (a) that alkali treatment + bleaching of pineapple leaves will produce a very defined individual fibers with an average diameter of 5-10 μm. It is observed that no impurities are present. On the other hand, alkali treated pineapple leaves [Fig. 1(b)] will produce a less

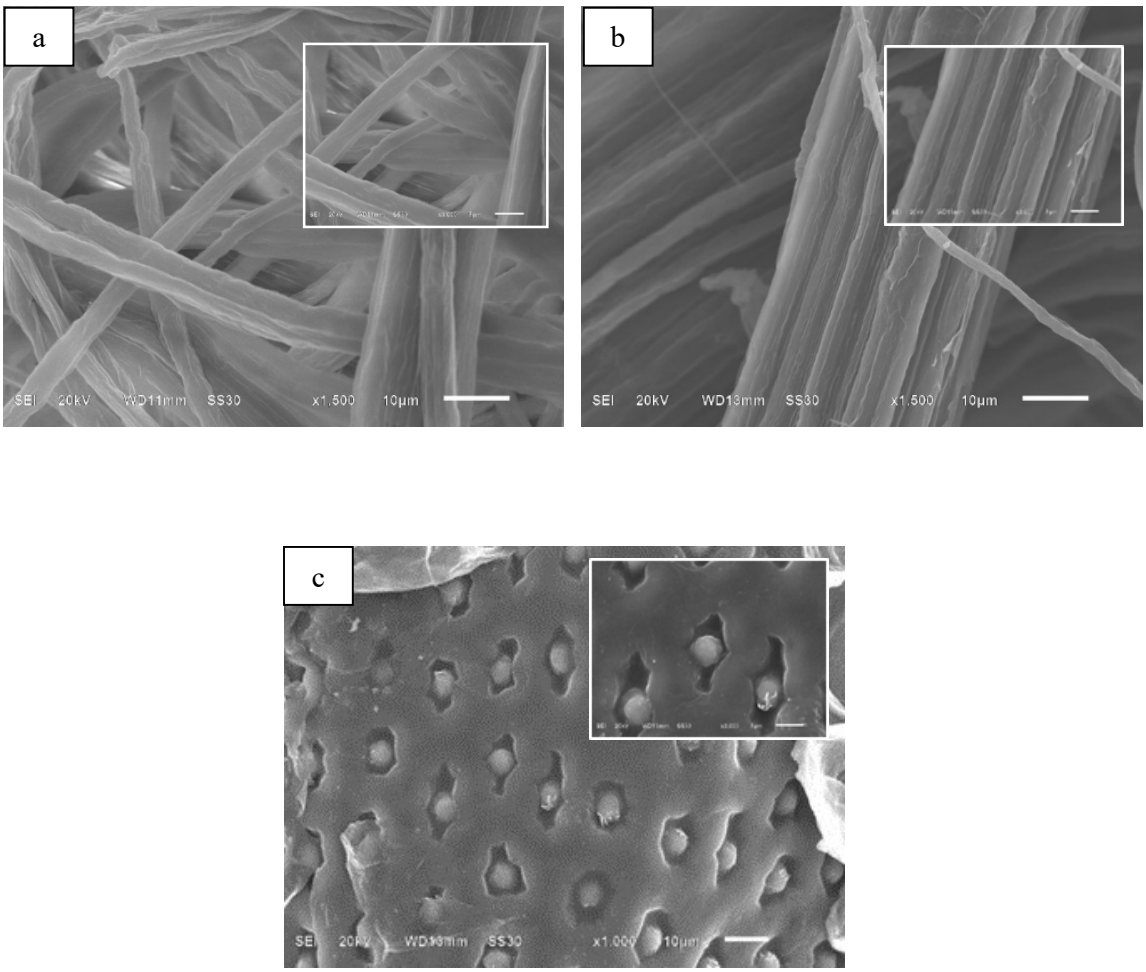

Fig.1. SEM images of the (a) Alkali + Bleach treated pineapple leaves, (b) Alkali treated pineapple leaves and (c) Untreated pineapple leaves. Inset photos are the lower magnifications.

defined fibers with bigger average diameter of about 100-300 μm. It can be easily observed the presence of impurities in the SEM images. Larger average diameter suggest that hemicellulose and lignin might be present in the sample. Moreover, the untreated pineapple leaves shown in Fig. 1(c) revealed that no definite formation of fibers were observed. This may be due to the presence of large amount of impurities that include hemicellulose, lignin and cement-like structures.

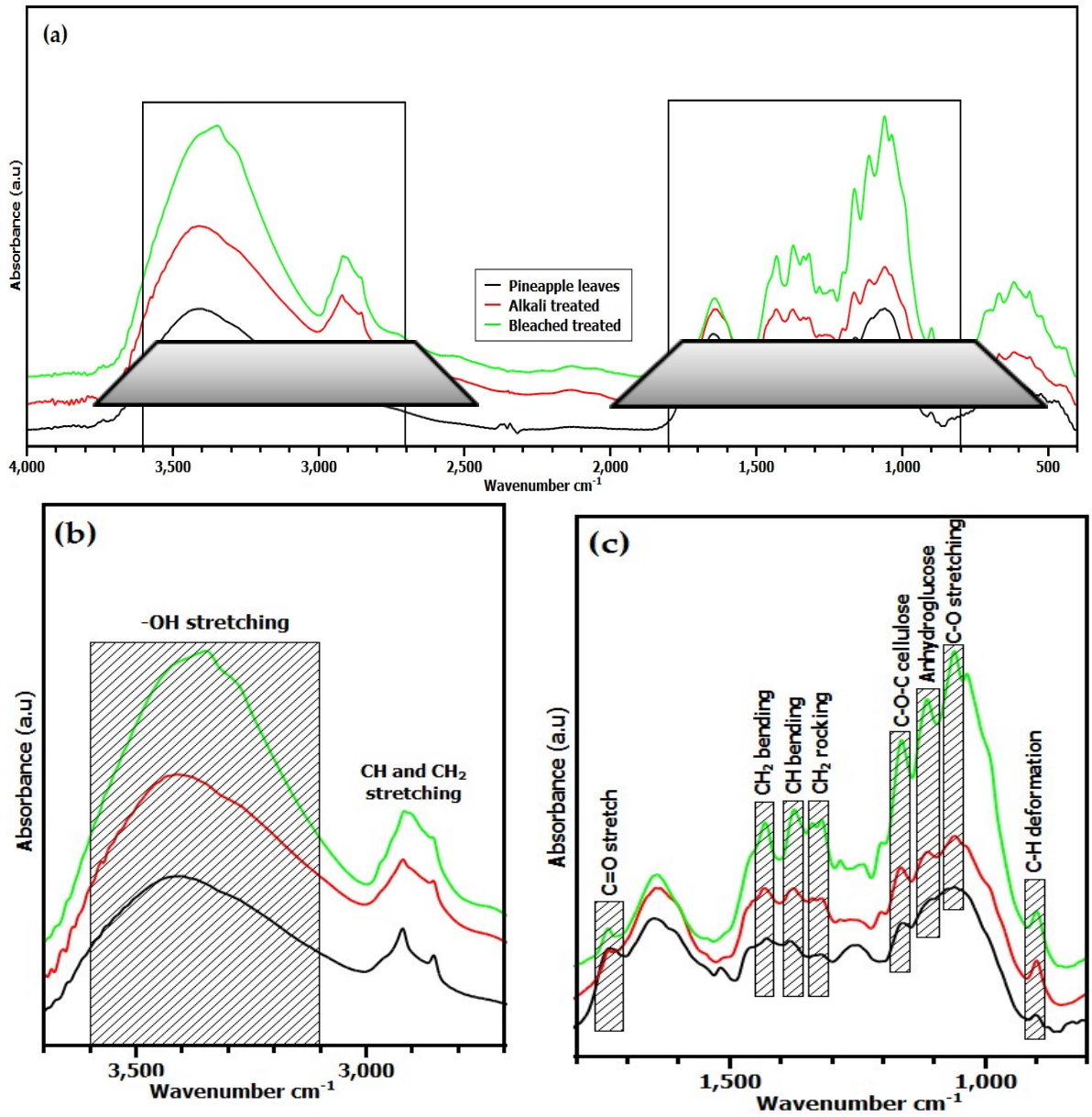

Fig.2. Over-all FTIR spectra of untreated, alkali treated and alkali + bleached treated pineapple leaves (a) in the region between 3700-2700 cm^{-1} (b) and between 1800-800 cm^{-1} (c).

Figure 2(a) shows the over-all FTIR spectra of untreated, alkali treated and alkali + bleached treated pineapple leaves. The FTIR spectra between 3700-2700 cm^{-1} are enlarged and shown in Figure 2(b) while the FTIR spectra between 1800-800 cm^{-1} are also enlarged and shown in Figure 2(c). It is observed that the O-H stretching ound between 3500-3100 cm^{-1} becomes more prominent for alkali + bleached treated sample as shown in Figure 2(b) suggesting that

larger number of hydroxyl groups triggered by the increase in number of hydrogen bonds associated to the production of high quality cellulose. The untreated pineapple leaves on the other hand has a lower absorbance of –OH stretching suggesting that more impurities are found in sample.

The vibrational mode of hemicellulose can be found at around 1733 cm^{-1} which attributed to C=O stretching vibration as shown in Figure 2(c). It is observed that the intensity of the vibrational mode corresponding to hemicellulose drastically reduces as alkali + bleached treatment was carried out. This clearly suggests that impurities such as hemicellulose was gradually removed as alkali + bleached treatment were done. Furthermore, the vibrational mode corresponding to cellulose are found 1426, 1372 and 1320 cm^{-1} which are associated to CH_2 symmetric bending, CH bending, and CH_2 rocking vibration. The bands at 1160, 1108, and 1054 and 898 cm^{-1} are associated to asymmetric C-O-C bridge stretching, anhydroglucose ring asymmetric stretching, C-O stretching and C-H deformation of amorphous cellulose, as shown in Figure 2(c). FTIR spectra revealed that absorbance of the vibrational mode corresponding to cellulose significantly increase as the alkali + bleached treatment was carried out. The significant increase as the alkali + bleached treatment was carried out. This significant increases is triggered by the removal of the impurities such as hemicellulose, lignin and cement-like structure after alkali + bleached treatment as confirmed in a well-defined fibers shown in SEM image Figure 1(a). On the other hand, the absorbance corresponding to cellulose in the untreated sample shown in Fig. 2(c) is very low. This mainly suggests that lots of impurities are still present in the untreated pineapple fibers.

Figure 3 shows the TGA spectra of untreated and alkali + bleached treated pineapple leaves.

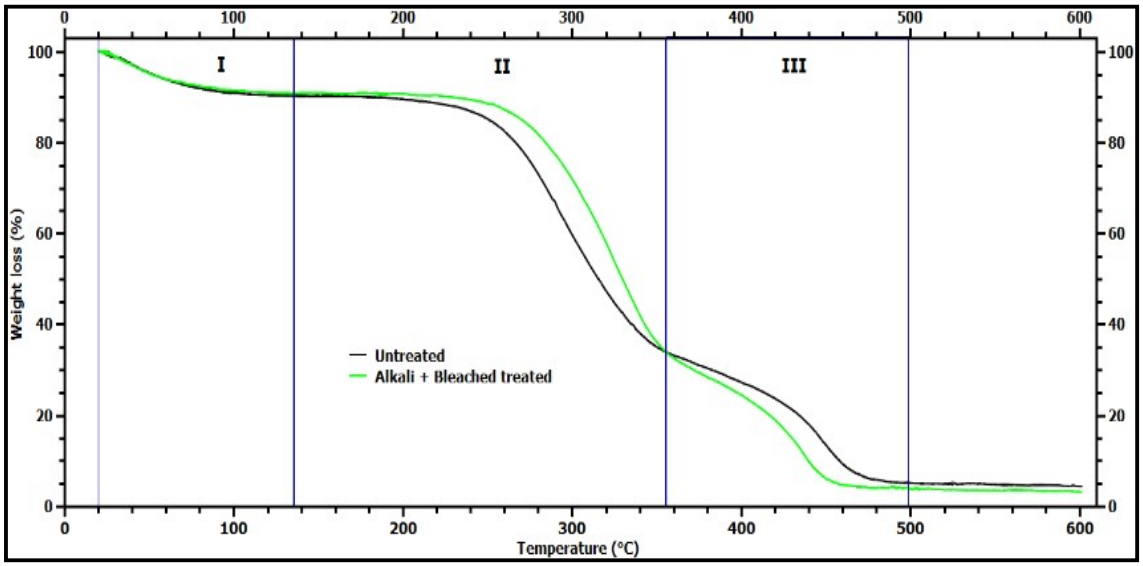

Fig. 3. TGA curves of untreated pineapple leaves and alkali + bleached treated pineapple leaves.

It is observed that the TGA spectra can be divided into three regions. Region I corresponds to the removal of moisture content. Region II in the TGA spectra correspond to the removal of hemicellulose and lignin in both untreated and treated pineapple leaves. It can be seen that slow degradation occurred in the alkali + bleached treated sample as compared to the untreated sample suggesting that hemicellulose and lignin were already removed during alkali + bleached treated sample. Similar to TGA spectra, it can be divided also into three regions.

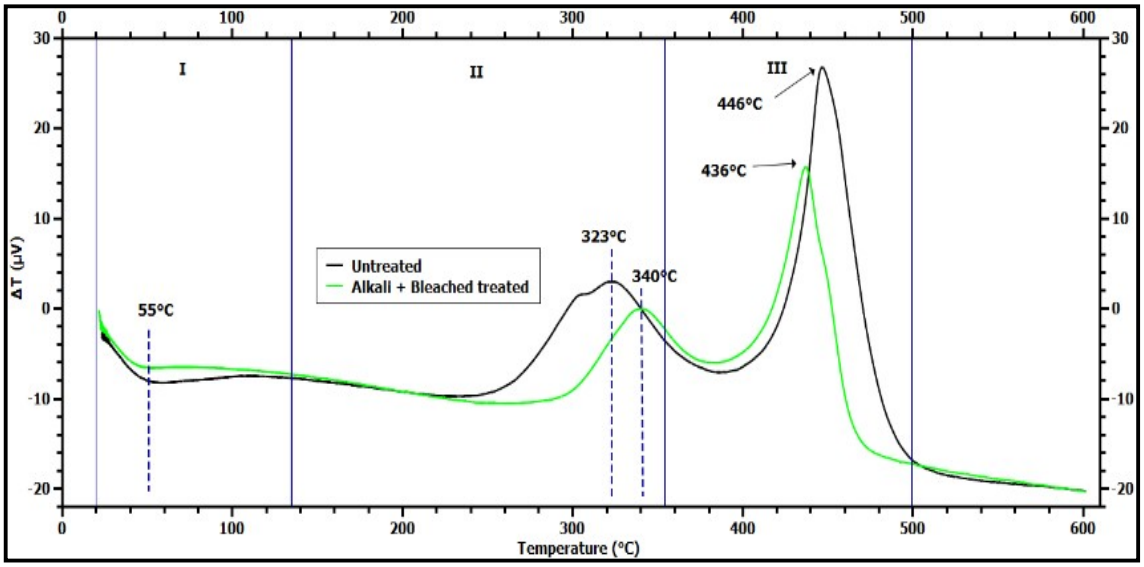

Fig.4. Differential thermal curves of untreated pineapple leaves and alkali + bleached treated

pineapple leaves.

The first region corresponds to the removal of moisture in the sample. The peak temperature that removes most of the moisture is around 55°C. Region II of the DTA spectra corresponds to the removal of hemicellulose and lignin in both untreated and alkali + bleached treated sample. It can be observed that untreated sample degrade at lower temperature (323°C) compared to treated sample (340°C). This simply suggests that untreated sample contains more amount of hemicellulose and lignin that could easily degrade at lower temperature. On the other hand, alkali + bleached treated sample degrade at higher temperature maybe because the hemicellulose and lignin were already removed during alkali + bleaching treatment of pineapple leaves. The third region of the DTA spectra of both untreated and treated pineapple leaves is shown in Figure 4. This region corresponds to the degradation of cellulose. It is observed that lower temperature for degradation of cellulose for treated pineapple leaves. This phenomenon supports our claim that treated pineapple leaves contain higher quality of cellulose as observed in the well-defined fibers show in the SEM image in Figure 1(a) and also supported in the TGA degradation in Figure 3 (Region III).

3. METHODOLOGY

3.1. Extraction of Cellulose

Pineapple leaves were dried and grind in small pieces. This was treated with sodium hydroxide aqueous solution of 2% (w/w) for 4 hrs at 100°C under mechanical stirring, washed several times with distilled water and dried in the oven. After this treatment, the material is bleached with a solution made up of acetate buffer and aqueous sodium chlorite. This bleaching treatment is performed at 100°C for 4 hrs. The bleached material was washed repeatedly in distilled water. Filter and rinse until the pH of the material became neutral and subsequently dried at 100°C for 5 hrs.

3.2. Characterization

The treated and untreated samples were characterized using scanning electron microscopy (JEOL JSM-6510LA) for its surface morphology. Fourier transform infrared spectroscopy (Perkin Elmer) was performed to determine the functional groups and vibrational modes present in the sample. Differential thermal analysis - thermogravimetric analysis (Shimadzu

60H) was used to measure the thermal stability of the samples.

4. SUMMARY

Extraction of cellulose from pineapple leaves using alkali + bleached treatment was successfully done. Hemicellulose, lignin and cement like structure were gradually removed during alkali + bleached treatment revealed in the presence of well-defined fibers shown in the SEM images The removal of impurities such as hemicellulose, lignin and cement like structure was also confirmed in the FTIR spectra. It was found out that high quality cellulose was produced after alkali + bleached treatment as indicated in the higher absorbance of FTIR spectra corresponding to cellulose vibrational modes. This was also supported by the TGA and DTA spectra that hemicellulose, lignin and cement like structure will degrade faster at temperature between 160-355°C. While cellulose will degrade faster between 355-500°C. This straight forward process is promising route towards the production of high quality cellulosic material.

5. ACKNOWLEDGEMENTS

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