



Science

MODIFICATION AND CHARACTERIZATION OF CEIBA PENTANDRA (L.) GAERTN. (KAPOK) FIBER: PHYSICAL PROPERTIES

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Abstract

Kapok fiber is a natural material that has unique properties, can be renewed with a large lumen, it has hydrophobic properties, as an absorbent in oil, heavy metals, and sound. To increase the absorption capacity of heavy metals, physical properties, and chemical resistance, modifications were made to change the fiber properties from hydrophobic to hydrophilic. One way is through the process of soaking the kapok fibers in a solvent to remove non-cellulose compounds. In this study, to improve the properties of kapok fiber, soaking has been done with various solutions, there are HCl, NaOH, NaClO, NaClO-NaOH-NaClO, and NaClO₂-NaOH-NaClO₂ solution. Immersion of kapok fibers in NaClO₂-NaOH-NaClO₂ solution can increase cellulose content up to 93.69% with porosity content of 4.75%. NaClO₂-NaOH-NaClO₂-treated can absorb water up to 211.27%, the second highest after treatment with the NaClO-NaOH-NaClO-treated solution of 285.29%. Immersion in NaClO-NaOH-NaClO and NaClO₂-NaOH-NaClO₂ solvents also causes damage to the kapok fiber protective layer which indicates the loss of lignin, so that a significant change of the hydrophobic fiber becomes hydrophilic as seen in the SEM results.

Keywords: Kapok Fiber; Alkalization; Oxidation; Hydrophilic; Chemical Resistance.

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1. Introduction

The kapok tree (*Ceiba Pentandra L.*) is one type of tropical tree that grows in Indonesia, one of which is in the province of Gorontalo. So far, the utilization of kapok fiber for Gorontalo is still low. The use of kapok fiber is only based on conventional utilization, for example as raw material for bolster pillows. However, because there are many foam cushions available, this has caused a decline in the interest of kapok-based bolster pillow makers. Kapok fiber is a natural material that can be renewed with a large lumen and has hydrophobic properties and can be used as a sorbent in oil ^[1]. Kapok fiber is a natural hollow fiber with a thin layer and a large cavity that is used as an adsorbent for heavy metal ions ^[2]. Kapok fiber has unique properties and with these properties

allows many new uses that can be developed. Based on previous research, kapok fiber can be used as an adsorbent material for heavy metals, oil, and sound.

Wahi ^[3] said that the behavior of increasing waste oil pollution increased with the expansion of oil exploration and production activities. Wahi added that the use of natural fiber sorbents as adsorbents to remove oil from waste is the right step considering that besides being able to remove oil, it is also environmentally friendly, easy availability, feasibility, and effectiveness. Alkali treatment is able to change the hydrophobic nature of kapok fiber into new fibers which are hydrophilic and have the potential as natural sorbent oil because of their high adsorption rate, stable structure and high reusability ^[4]. Because it has hydrophobic-oleophilic characteristics, a homogeneous hollow tube shape, and low density, kapok fibers are often used as filler fibers in pillows and oil sorbents. Hori ^[5] in their research stated that kapok fiber can be used as an excellent oil adsorbent.

The use of kapok as an adsorbent material because this material is a biodegradable material. Wang ^[1] mentioned in his research that kapok fiber soaked in NaClO₂ can increase the absorbance properties of the oil. Soaking kapok fiber in NaClO₂ solution can change the nature of kapok fiber from hydrophobic to hydrophilic where almost all phenolic compounds are released from the fiber. The addition of NaClO₂ has the positive effect on the efficiency of oil content absorbance. Duan ^[2] in their research stated that the modification of fiber fibers using diethylenetriamine pentaacetic acid (DTPA) can be used as adsorbents in Pb²⁺ and Cd²⁺ ions. Based on the results of his research stated that kapok-DTPA fiber has better adsorbent capacity compared to the results of studies that have been reported specifically using natural fibers. Adsorption capacity decreases with decreasing pH. Chemical modification of kapok fiber application can be used as an alternative if we want to remove Pb²⁺ metal ion content in industrial waste ^[6]. Veerakumar ^[7] said that kapok/polypropylene fiber composites can be used as the sound absorbing material. Kapok fiber can also be mixed with thermoplastic cassava starch (TPCS) to reduce water uptake in TPCS / kapok composite and increase the stress on the maximum force and modulus young ^[8].

Based on the background above, the researcher has the idea of doing physical testing and chemical resistance of kapok fiber both before and after soaking in order to obtain data on kapok content specifically and obtain physical properties data, analyze changes in properties from hydrophobic to hydrophilic and increase hydrophilic properties, analyze types solvent to increase absorbency of kapok fiber to water absorption. This research is important to be carried out considering the number of kapok trees which are very abundant in Gorontalo Province and their utilization is not maximized.

This study uses solvent HCl, NaOH, NaClO, NaClO-NaOH-NaClO, and NaClO₂ - NaOH - NaClO₂ to remove lignin, pectin, hemicellulose, natural oil, and kapok wax so that changes occur from hydrophobic to hydrophilic. Researchers believe the use of a combination of solvents can change the hydrophobic nature to become hydrophilic for the better.

2. Materials and Methods

2.1. Materials

Kapok fiber derived from Gorontalo province, Indonesia. Hydrochloric acid (37%) solution, Sodium Hydroxide solids, Sodium hypochlorite (%) solution and Sodium chlorite solution (%) are obtained from PT. Sumber Utama Kimiamurni, Surabaya City, Indonesia.

2.2. Preparation of Kapok Fiber

Acid modification of Kapok Fiber with HCl

Kapok fiber (4 grams) was put into 400 mL HCl solution (1 M) for 2 hours at room temperature. Then washed with distilled water to pH = 7 and dried for 24 hours at 40°C. Preparation of natural materials be done in Metallurgy Laboratory.

Alkali modification of Kapok Fiber with NaOH

Kapok fiber (4 grams) was put into 400 mL of 2wt% NaOH solution for 1 hour at 70°C. After that, it was removed and washed with the glacial acetic acid solution (100 %) to pH 7. Then proceed with washing using distilled water and dried for 24 hours at 40°C.

2.3. Oxidation modification of Kapok fiber with NaClO

Oxidation uses NaClO solution

Kapok fiber (4 grams) was soaked in 200 mL of NaClO (0.5 N) and 2 mL glacial acetic acid solutions (100%) for 1 hour, 80°C. Then removed and washed with distilled water to pH 7 and dried for 24 hours at 40°C.

Oxidation uses NaClO – NaOH – NaClO solution

Kapok fiber (4 grams) was soaked in 200 mL of NaClO solution (0.5 N) and 2 mL glacial acetic acid (100%) for 1 hour, 70°C, then removed and suspended with 200 mL H₂O and 1.5 mL glacial acetic acid with the addition of 7.5 grams of NaOH. Then stirring 10 minutes at room temperature. Then wash with aquadest to pH 7, put in 100 mL of NaClO solution (0.5 N) for 10 minutes at 70°C, washed with acetone, dried for 24 hours, 40°C.

2.4. Oxidation modification of kapok fiber with NaClO₂

A total of 4 grams of kapok fiber was put into 200 mL of NaClO₂ solution (0.5 N) containing 2 mL of glacial acetic acid solution (100%) for 1 hour at 70°C. Then it was suspended with 200 mL of aquadest containing 1.5 mL of glacial acetic acid solution and 7.5 grams of NaOH. Then stirred for 10 minutes at room temperature, removed and washed with distilled water to pH 7. Then proceed with soaking in 100 mL of NaClO₂ solution (0.5 N) for 10 minutes at 70°C. After that it was washed with acetone, dried for 24 hours at 40°C.

2.5. Characterizations

Fourier transforms infrared spectroscopy

Fourier transforms infrared (FTIR) spectra were reported using scientific Thermo Nicolet iS10 at room temperature. The sample spectrum was measured at 500 - 4000 cm^{-1} wavelength. Material testing was carried out at the Material Characterization Laboratory, Department of Materials and Metallurgical Engineering, ITS, Indonesia.

Scanning electron microscopy

Scanning electron microscopy (SEM) is used to observe the microstructure and shape of the kapok fibers before and after treatment. SEM testing is done by coating the sample with Au / Pd and analyzed using INSPECTTM S50 SEM microscope apparatus. SEM testing was carried out at the Material Characterization Laboratory, ITS, Indonesia.

2.6. Determination of Porosity and Cellulose

Porosity and cellulose content in kapok fibers can be determined using bulk density, absolute density, and cellulose density^[9]. Bulk density (ρ_b) can be calculated using equation 1.

$$\rho_b = \frac{\rho_s W_{fa}}{W_{fa} - W_{fs}} \quad (1)$$

Where ρ_s is toluene solvent density, 867 kg/m^3 . W_{fa} is initial weight of fiber (kg) and W_{fs} is the weight of the immersed fiber. All measurements were determined at a temperature of 19°C and the weight was measured at the nearest 0,001 g.

2.7. Water Absorption

Water absorption in kapok fiber is determined by referring to the ASTM C209 and ASTM D5229 M testing standards based on equation 2. Water Absorption Tests were carried out at the Metallurgy Laboratory, Gorontalo State University, Indonesia. The specimens were immersed in water for 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, dan 24 h, respectively. After immersion, the surfaces of the specimens were dried with tissue paper and weighed to measure their wet weighth. Formula of the water absorption can be seen in equation 2.

$$\% \text{ Moisture sorption} = \left(\frac{W_i - W_b}{W_b} \right) \times 100 \quad (2)$$

Soaking is done for 24 hours in relative humidity at room temperature. Where W_b is the initial weight, W_i is heavy after being immersed in water. Tests are carried out on all kapok fibers both un-treatment and those that have been treated.

3. Results and Discussions

3.1. Modification of Kapok Fibers

Kapok fiber was modified through a process of soaking in various solvents, included HCl, NaOH, NaClO, NaClO-NaOH-NaClO, and NaClO₂-NaOH-NaClO₂ solution. Immersion in each solution through different stages. Immersion in HCl (1 M) solution did not show significant changes. This is proven by the results of HCl-treated kapok fiber FTIR which is similar to untreated kapok. Based

on Fig 1, the intensity of kapok fiber absorption in HCl solution at a peak of 1732.16 cm^{-1} in the form of C = O group did not change. Acidic solutions in HCl solution cannot break the π bond in the C = O group which indicates the presence of lignin, pectin, and kapok wax. As a result, after soaking the kapok still remains hydrophobic. This is evident in the data in Table 2. Water absorption in HCl-treated kapok is only 55.28%, while water absorption in untreated kapok is around 46%. Water absorption in HCl-treated is low compared to other solvents, both alkaline and oxidation solvents. The amount of water absorption on the untreated kapok and HCl-treated levels is caused by water molecules trapped in the hollow part of the kapok fiber. HCl-treated can only eliminate hemicellulose, especially xylose^[10,11].

Immersion of kapok fiber in alkaline solution in the form of NaOH solution (2%) causes changes in kapok from hydrophobic to hydrophilic properties. This can be seen from the loss of peak in the area of 1732.16 cm^{-1} which indicates that the π bond has been broken on the C = O function group. The change in nature from hydrophobic to hydrophilic is also evidenced by a large amount of sorption from the solution. The same thing also happens when kapok fibers are soaked in a solution of NaClO, NaClO-NaOH-NaClO, and NaClO₂-NaOH-NaClO₂.

3.2. FTIR Analysis of Kapok Fiber

The different types of solutions used as kapok fiber solvents cause intensity differences in peaks as shown in Fig. 1 At peak 3342.32 cm^{-1} shows the -OH group stretching vibration^[12] and indicates the presence of cellulose^[13]. There is an increase in the intensity of O-H stretching because immersion in acids, alkalis, and oxidation can increase the number of free hydroxyl groups in cellulose units. Peak 2916.26 cm^{-1} indicates asymmetric and symmetric aliphatic CH₂ and CH₃ stretching^[4,14]. This generally identifies the existence of surface wax^[1].

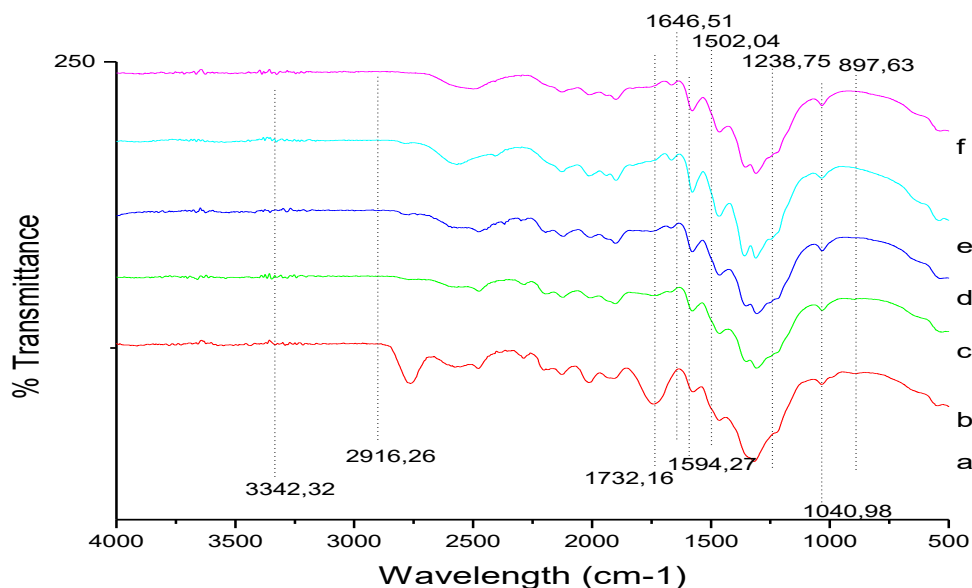


Figure 1: FTIR Spectra of (a) untreated kapok, (b) HCl-treated kapok, (c) NaOH-treated kapok, (d) NaClO-treated kapok, (e) NaClO-NaOH-NaClO-treated kapok, and (f) NaClO₂-NaOH NaClO₂-treated kapok

The decrease in intensity at peak 2916.26 cm^{-1} appears in NaOH-treated and NaClO_2 -NaOH- NaClO_2 -treated. This is due to the effect of alkali on NaOH solution and oxidation in NaClO_2 solution. Peak 1732.16 cm^{-1} is C = O stretching vibration group. This peak shows the presence of ketones, carboxylic groups, and esters in lignin and acetyl ester groups in xylan^[15,16]. The carbonyl group C = O in ester group also indicates that it is kapok wax^[4]. There was a decrease in intensity at peak 1732.16 cm^{-1} and indicating a decrease in the amount of lignin and hemicellulose due to the breakdown of the carbonyl C = O by the effect of the alkaline solution and oxidation solution. Acid (in this case HCl) is unable to break the carbonyl group. Peak 1646.51 cm^{-1} also shows group C = O stretching^[14]. This peak also indicates the presence of hemicellulose and in the presence of a peak at 897 cm^{-1} indicates the presence of β -linked hemicellulose^[2].

The peak of 1594.27 cm^{-1} shows the presence of the backbone ring benzene together with a peak of 1502.04 cm^{-1} ^[13]. This peak has decreased in NaClO-NaOH-NaClO-treated and NaClO_2 -NaOH- NaClO_2 due to phenolic compounds which are part of lignin damaged^[17] and turned into hydroxyl groups which increase the intensity at peak 3342.32 cm^{-1} . Peak 1238.75 cm^{-1} is a C-O bending vibration group. This peak shows the presence of lignin and hemicellulose. Peak 1040.98 cm^{-1} indicates carbohydrates and polysaccharides which signify cellulose clusters^[4]. This peak does not experience significant changes in intensity indicating no changes that occur specifically in cellulose.

3.3. Physical Properties of Kapok Fiber

Porosity and cellulose of kapok fiber

One of the physics tests used in this study is to test the porosity and cellulose levels of kapok fibers. The results obtained after testing can be seen in Table 1 about porosity and cellulose of kapok fiber. Based on Table 1, we can see the values of porosity and cellulose from kapok fibers.

Table 1: Porosity and cellulose content of kapok fiber

No	Fiber Type of Kapok	Diameter	Porosity	Non-cellulose	$P + X$	$[(P + X) - Y]$	Cellulose content
		(μm)	P (%)	+ air Y (%)	Y' (%)	Tair (%)	Z (%)
1	Untreated	17.62	40.79	41.66	42.27	0.60	57.13
3	HCl-treated	16.58	26.40	27.49	27.88	0.39	71.73
2	NaOH-treated	14.21	11.69	12.99	13.17	0.17	86.66
4	NaClO-Treated	15.76	11.99	13.30	13.48	0.18	86.34
5	NaClO-NaOH-NaClO-Treated	12.31	5.73	7.12	7.21	0.08	92.71
6	NaClO ₂ -NaOH-NaClO ₂ -Treated	11.12	4.75	6.16	6.23	0.07	93.69

Of the six types of kapok fiber, untreated kapok has the highest porosity value of 40.79% and the lowest cellulose content is 57.13%. The NaClO_2 -NaOH- NaClO_2 -Treated has the lowest porosity content (4.75%) and the highest cellulose content of 93.69%. The greater the value of porosity, the

lower the cellulose content. As previously explained, the effect of alkalization and oxidation on kapok fibers causes loss of lignin, pectin, hemicellulose, wax and natural oils which protect the fiber surface [3,10] so that only cellulose remain.

3.4. Water Absorption

In addition to testing the porosity and cellulose content, water absorption test was also carried out to measure the amount of water absorbed by kapok both untreated and treated kapok. Water absorption is also one of the determinants of changes in the nature of kapok from hydrophobic to be hydrophilic.

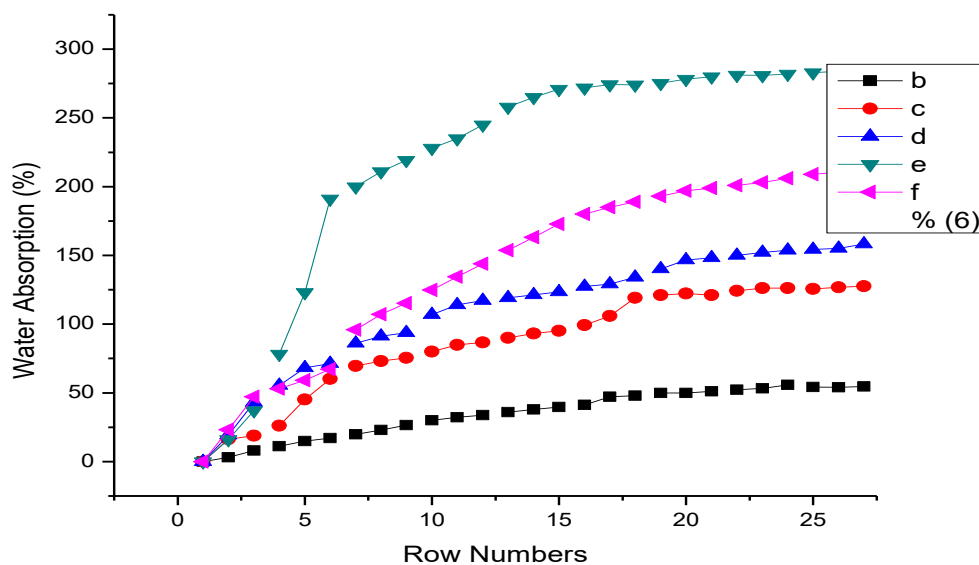


Figure 2: Water absorption of (a) untreated kapok, (b) HCl-treated, (c) NaOH-treated, (d)NaClO-treated, (e) NaClO-NaOH-NaClO-treated, and (f) NaClO₂-NaOH-NaClO₂-treated

Based on Figure 2 about water absorption of natural fiber can be seen the percentage of water absorption from fiber type of kapok. From the six types of kapok, untreated moisture sorption has the smallest moisture sorption value of about 45.29%, while NaClO-NaOH-NaClO-treated kapok has the highest moisture sorption percentage of around 285.29%. The difference in the percentage of moisture sorption from each type of kapok is due to untreated-kapok having hydrophobic properties because of the surface wax and natural oil in kapok which in theory kapok fiber cannot absorb water. The amount of percentage of water absorption on untreated kapok is due to the water being trapped between the fibers. Likewise, with HCl-treated acid, although it has the ability to remove kapok wax, HCl-treated cannot remove lignin and pectin from kapok and is only capable of removing xylose hemicellulose [10,11].

NaClO-NaOH-NaClO-treated has the largest water absorption percentage value. This is because there are more than one immersion stage, which begins with NaClO-treated oxidation, then proceed with the NaOH alkalization process, and after that it is treated again with oxidation of

NaClO, so that non-cellulose compounds (hemicellulose, lignin, and pectin) are eliminated significantly and cause changes in the nature of kapok from hydrophobic to be hydrophilic. When compared with NaClO₂-NaOH-NaClO₂-treated from the results of the study, NaClO solution is more effective in terms of the ability to absorb water even though it has a cellulose content lower than the NaClO₂ solution. Both NaClO and NaClO₂ are oxidation solutions.

3.5. Scanning Electron Microscopy of Kapok Fiber

Based on SEM analysis (Fig. 3), there is a significant difference between untreated kapok and treated kapok. Fig. 3. a and b are untreated. It can be seen the smooth and round hollow lumen shape of the kapok fiber. When treated well using a solution of NaClO-NaOH-NaClO (Fig 4. c and d) and a solution of NaClO₂-NaOH-NaClO₂ (Fig 4. e and f) there are a change in the shape of the kapok fiber. The fibers become wrinkled, rough, non-glossy, and there were particulates on the surface of the fiber after soaking. Particulate attached to the surface of the fiber is caused by stretching the pores of the fiber so that it can suck particles in the water (see red circle image 3 e and f). The loss of non-cellulose compounds causes kapok to change the nature of hydrophobicity to be hydrophilic. The loss of lignin, pectin, wax and natural oil that covers the surface of the fiber is also able to improve the surface and mechanical properties for polymer applications [3,18].

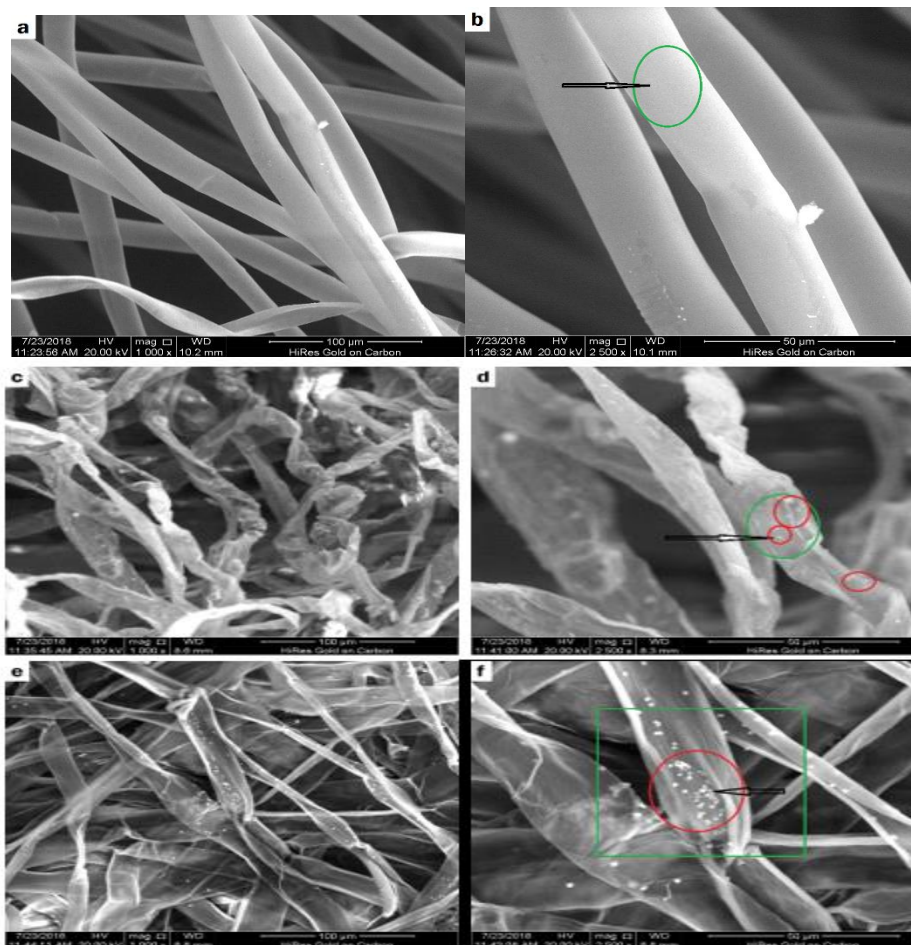


Figure 3: SEM Micrograph of (a and b) untreated kapok, (c and d) NaClO-NaOH-NaClO treated, and (e and f) NaClO₂-NaOH-NaClO₂-treated kapok

4. Conclusions and Recommendations

Physical properties and chemical stability of the kapok were investigated. The results of this study indicate that untreated kapok has hydrophobic properties. Treatment with various solvents causes changes in properties. HCl-treated does not significantly influence changes in kapok. This is because HCl is only able to reduce hemicellulose levels, especially xylose in kapok so that the kapok still remains hydrophobic. Treatment using an alkaline solution (NaOH) causes a change in the nature of kapok from hydrophobic to hydrophilic. This is because the NaOH base can damage the ester group in the kapok fibers so that both lignin, pectin, hemicellulose and kapok wax can be removed. NaOH-treated is better than HCl-treated. The use of oxidation solution has a better effect than acidic or basic solutions. The collaboration of alkaline and oxidation solutions such as NaClO-NaOH-NaClO-treated and NaClO₂-NaOH-NaClO₂-treated has a greater influence in removing non-cellulose compounds and is also able to increase amorphous cellulose which has the effect of increasing cellulose content, increased water absorption capacity and the nature of chemical stability is getting better.

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