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TESTING MICROCRYSTALLINE CELULLOSE USING SPECTROMETER AND POLARIZED LIGHT MICROSCOPE

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ABSTRACT

It has been proposed to test the microcrystalline of cellulose materials using polarized light microscope (PLM) and uv-visible spectrometer . The microcrystalline cellulose was prepared from kenaf bast fibers while the nano crystalline was prepared from alpha cellulose using thermal acid hydrolysis. We use at 20% (v/v) and 40% (v/v)H_2 SO_4 in the experiment mixed and stirred within an hour up to hours. The testing using PLM clearly indicates the existence of microcryatalline cellulose while the use of uv-visible spectrometer can reveal the effectiveness of the acid hydrolysis in extracting the nanofibril from the alpha cellulose.

Keywords: cellulose, micro, polarized light microscope, spectrometer.

I. INTRODUCTION

Natural fiber has drawn attention of researchers and industries because it may be produced to be various promising materials [1-3]. Basically natural fiber consists of cellulose, hemicelluloses, lignin, pectin, waxes, and water soluble substance. One of the three classes cellulose, the alpha cellulose is the most stable one. It is the major component of wood and paper pulp and is insoluble. It can be filtered from the solution and washed prior to use in the production of paper or cellulosic polymers. The cellulose is an interesting materials that may be utilized as based materials for energy resources [4,5]. It can be further made into nano fibril and nano whisker to be used as composite materials having novel properties thermally or mechanically [6]. Therefore, in the near future the used of the cellulose will cover wide materials application such as plastic utensils, binding materials, additive in food and drugs, paints and many other things [6,7]. However, in process of making it, some characterizations have to be made before it can be used properly. To characterize the microcrystalline of cellulose, many advance equipments are usually used. Scanning electron microscope and transmission electron microscope are common equipments to know the size of the cellulose. X-Ray diffraction equipment is used to test the crystalline of cellulose. Infra-red spectrometer is used the check its binding structure. When, a quick information about morphology and the crystalline of the cellulose are required, it needs a fast and simple way to get such an information. Since, nano crystalline cellulose usually showing a birefringence [9] phenomenon which might be check easily using polarized light, in this article, we will discuss the characterization of such a cellulose using

polarized microscope and Uv-visible spectrometer as an alternative method of characterizing micro to nano crystalline cellulose.

II. METHODS OF EXPERIMENT

In our ex*p*eriment the micro and nano cellulose are prepared from alpha-cellulose made by Merck. The preparation consists of preparing the solution of 30% to 40% v/v ratio of H_2SO_4 in aquabidest as a solution to be mixed with the alpha cellulose. Once both are made, it is then stirred using magnetic stirrer at 1500 rpm while heated at 50^oC at various time interval. Although in some reports this process could be done at 30 minutes to one hour, but in our experiment during such times no significant changes happening in the structure of cellulose. On the other hand, the use of H_2SO_4 will burn the cellulose indicating by the change of color of solution from yellow in to black. This is the reason we use a lower acid concentration. We make three samples having used stirring process done within 3 hours, 4 hours, and 6 hours respectively. The results are then centrifuged at 7000 rpm and rinsed using tap water several times until the pH of water reaching normal. Similar process we use to treat the kenaf cellulose. This cellulose was produced from a raw knead bast fibers from Malang, Indonesia, using our previous method [8]. The final result of the acid process of cellulose produces nano and microcrystalline cellulose as the colloid and the supernatant. The colloid and the supernatant is tested using PLM (MT 6130) to see the different morphology and the crystalline. The microcrystalline are seen using PLM by rotating the analyzer hitting by the polarized light pass through sample. Some parts of the colloid are tested using uv-visible spectrometer (Shimadzsu 1400) to check the morphology of the cellulose. In confirming the result, we use also SEM to test the size of the cellulose and XRD to test the crystalline.

III. RESULT AND DISCUSSION

After 6 hour and 12 hour heating, centrifuging, and rinsing we get the result shown at Fig.1. Both show that the cellulose to be in the state of microcrystalline with the diameter of microcrystalline is in the order of micrometer to ten micrometer. The treatment using HCl (20% v/v) shows better fiber dispersion than that of the use of H_2SO_4 (20%v/v). We suspect this is caused by the different electric charge distribution in the fibers resulted in the process. The treatment using HCl produces more uneven charge distribution in the fiber than that of using H_2SO_4. This will help to producing more repelling force between fibers.

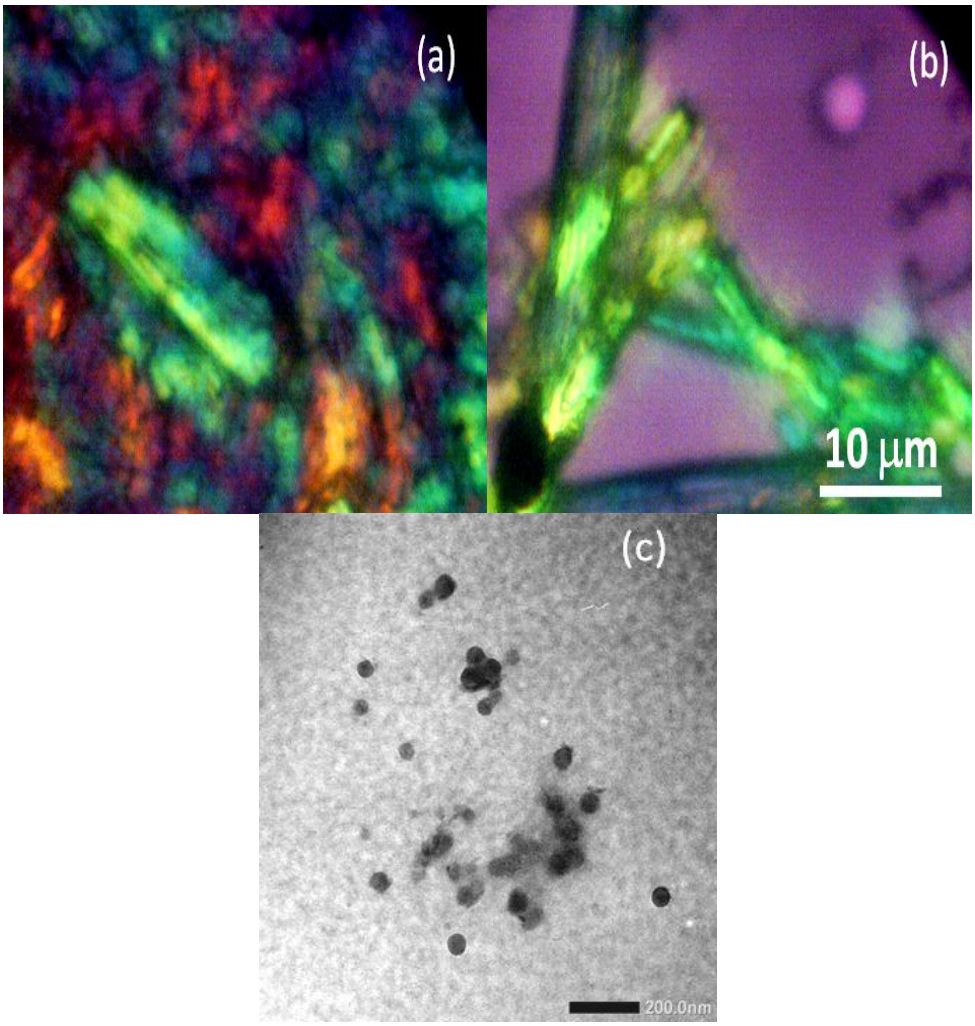

Figure 1. Microcellulose of kenaf processed using (a) H_2SO_4 (20%v/v) and (b) HCl (20% v/v) within 2 hours taken using polarized light microscope (PLM)showing different color indicating the microcrystaline properties of the cellulose. The further acid process using (c) H_2SO_4 (40%v/v) at 2 h producing nano particles cellulose taken using transmission elctron microscope (Jeol-1200).

We make a longer acid treatment together with heating at 50^{o}C to the kenaf cellulose, at 60 minutes, they become nano particles while if we quench after 30 minutes in heath bath with ice, we get whisker cellulose as shown in Fig. 1 (c) which can be clearly seen using TEM(Jeol-1200) set at 80 kV. From here we can see that PLM can be used to identify the crystalline of cellulose. Further we use also alpha cellulose precursor to be processed in to micro or nano crystalline cellulose using acid hydrolisis. Using PLM we can see after 2 hours acid

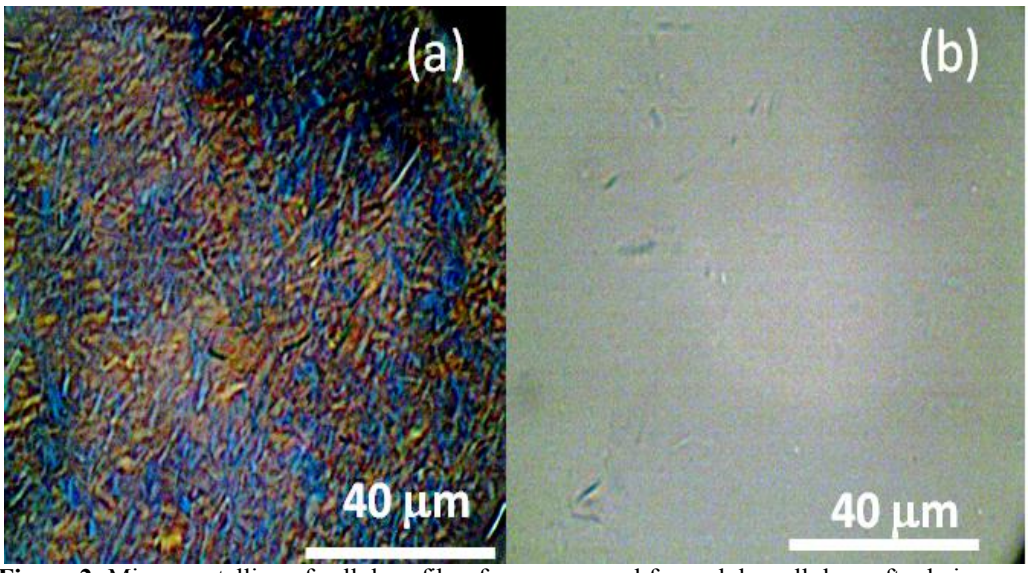

Figure 2. Microcrystalline of cellulose fiber from processed from alpha cellulose after being treated using H_2SO_4(40%v/v) at 1 hour (b) 4 hour.

treatment using H_2SO_4 (40%v/v) for an hour we can still see the crystalline cellulose indicating as shown in Fig. 2 (a). However, when 4 hours acid tretament is given, we can no longer see the collor using PLM because the size of cellulose become smaller and transparant as indicated by Fig. 2 (b). Further investigation using SEM indicates that the cellulose diameter in the order of tens nanometer. We use 3 hours and 6 hours acid hydrolysis process using H_2SO_4 (40% v/v) at 40^{o}C to the alpha cellulose and obtain

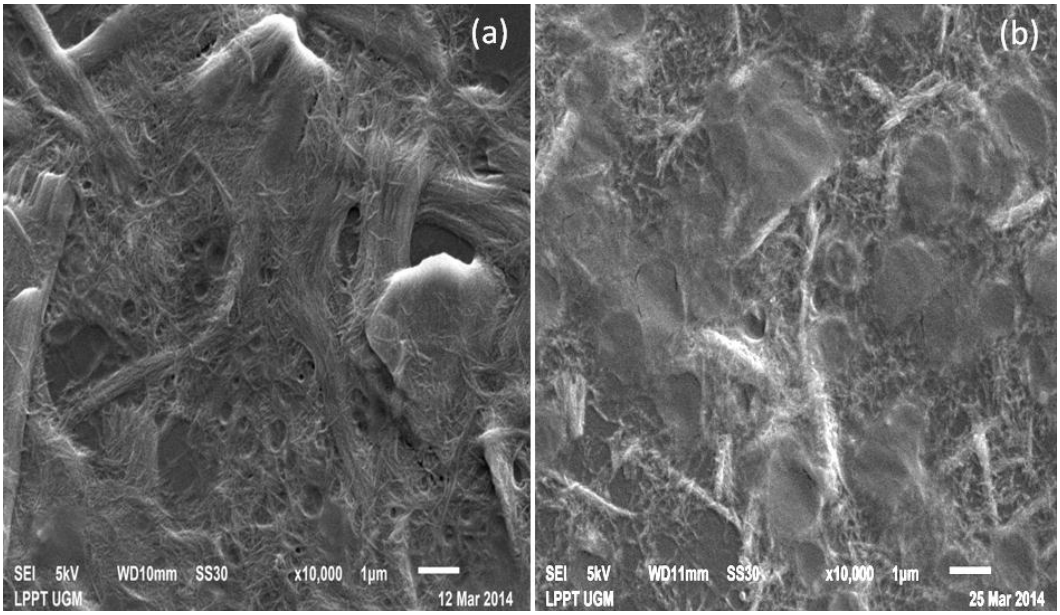

Figure 3. Scanning electron microscope of the alpha-cellulose after processed in 40%v/v H_2SO_4 at 40^oC (a) within 3 hour and (b) within 6 hour. The size of the cellulose particles can also be visually seen using laser showing Tyndall effect (c).

the result shown at Fig.3. This tells that if the result is hardly seen using PLM as in Fig 2., it means that the result is in the nano meters scale. As a whole, it can be seen clearly that the acid hydrolysis treatment brings the alpha cellulose in the state of nano fibril due to the lack of high pressure homogenization or high power ultrasonication. We find also that only a little diameter fiber change due time acid hydrolysis treatment from 3 hours to 6 hours. This indicates that strong fiber covalent bonding existing in alpha cellulose makes it insensitive to be broken down using the treatment. However, our previous investigation indicates that such treatment usually increases the crystalinity [8]. However, when we check also visually the colloid of nano crystalline using laser pointer (l=660 nm), both colloid producing similar Tyndall effect as shown at Fig. 3 (c). Further we test the colloid using uv-visible spectrometer (Shimadzu 1400), we find that there are absorbtion peaks at 285 nm 345 nm, 370 nm, 395 nm produced by the colloid. These peaks are atrributed to the peaks of alpha cellulose. It can be noticed that the concentration of cellulose become smaller as the hydrolysis time is increased indicating the forming of the nano fibril as confirmed in Fig. 3. The changing of the cellulose into nano fibril is saturated by time indicating that the longer acid hydrolysis at 40^oC has no longer effective to reduce the alpha cellulose into nanofibril. In addition, in our experiment we can not see the existence of birefringence effect of the crystalline showing that the nano fibril formed to be quite low in concentration.

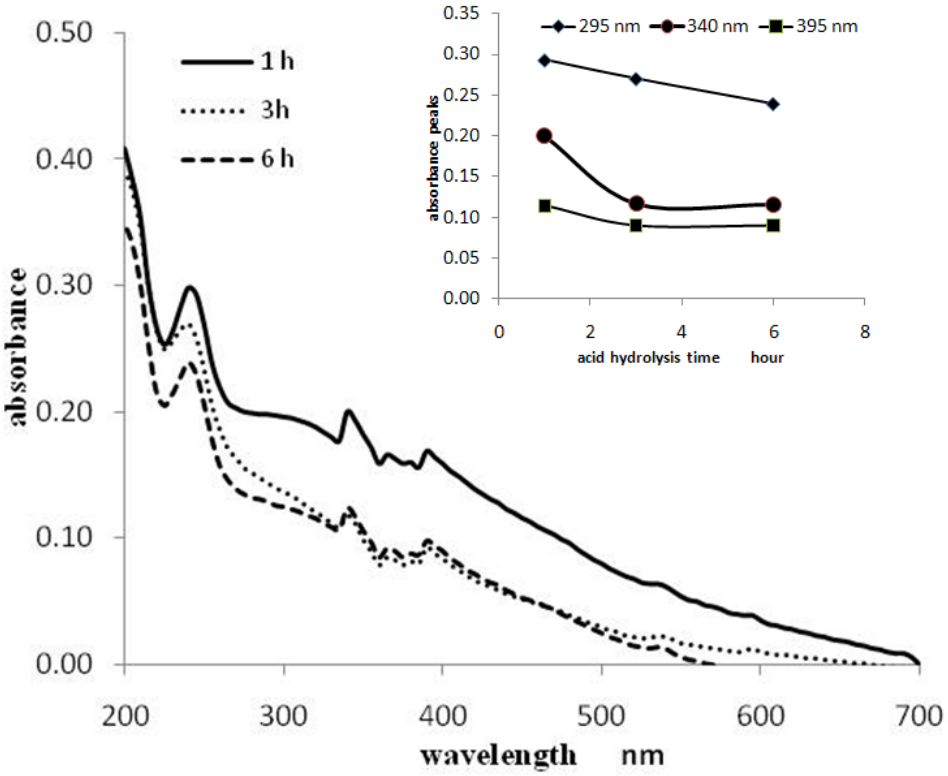

Figure 4. Uv-visible absorbance of the alpha-cellulose after processed in 40% v/v H_2SO_4 at 40^oC (a) within 4 hour, 6 hours and 12 hour and the absorbance of peaks at 295 nm, 340 nm, and 395 nm showing the decreasing of the concentration due to the increasing of acid hydrolysis (inserted picture).

IV. CONCLUSION AND SUGGESTION

We have characterized the micro and nanocrystaline cellulose using PLM and UV-Visible spectro and the result indicates that using PLM we can observe the microcrystalline formed from the cellulose while using UV-visible spectrometer we can observe the effectiveness hydrolysis process in reducing cellulose becoming nano fibrils. For a better result in forming nano crystalline, we are on the way to implement either high pressure homogenization or the high power ultrasonication treatment to the microfibril.

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