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An Experimental and Theoretical Study on the Nano Cellulose with Different Morphology in Aqueous Solution

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Abstract- Cellulose has inspired great interest in the research over recent years due to its unique nanostructure and application to easy with properties such as elasticity, strong hydrogen bonding, and excellent improving durability for structure in compound with other materials. Among them, Cellulose nanocrystals (CNCs) and fibers (CNFs) are based on the hydrophilic characteristics by structure and morphology so that it can be promoted fluid interaction in aqueous media. In this paper, we were using that 120kV TEM, UV/Vis spectrophotometer, Lambda system, and Thermal analyzer for the sphericity CNCs and the long bundle CNFs were compared to reaction in a solvent according to the structure. Thus, we are focus on identifying the differences in the structure of two types of cellulose, and the propensity of cellulose with different shapes in aqueous media.

Keywords – Cellulose Nanocrystals, Cellulose Nanofibers, Nanofluid

I. INTRODUCTION

Cellulose has been studied due to their elasticity, strong hydrogen bonding, and excellent durability over recent years [1]. It's produced from nature resource that can be seen eco-friendly material such as wood [2], plant [3], and sea creatures [4]. Among them, the cellulose can be divided their main resource or manufacturing method. It can be divided into two main types, and it has been reduced nano-sized. Cellulose nanocrystals (CNCs) have been manufactured by chemical method as sulfuric acid. It composed only crystalline region that is taken from non-crystalline and crystalline of cellulose. Cellulose nanofibers (CNFs) have been manufactured by mechanical method as grinding. The cellulose nanofiber is composed non-crystalline and crystalline of cellulose. This represents the structural difference from cellulose nanocrystal [5]. Mainly, cellulose property has been organized hydrophilic structure by the abundant -OH bundles [6]. However, the non-crystalline region is different from crystalline region with relatively no gaps. Therefore, it is possible to know the hydrophilic tendency according to the difference of whether the non-crystalline region included or not. In this paper, it not only the inclusion of non-crystalline regions, but also the comparison of properties according to their shape accompanied. The sphericity CNCs and the long bundle CNFs were compared to reaction in a solvent according to the structure. Thus, we are focus on identifying the differences in the structure of two types of cellulose, and the propensity of cellulose with different shapes in aqueous media.

II. EXPERIMENT DETAILS

The fluid preparation with Commercial cellulose nano crystal (CNC, with a 99.0% purity) was obtained from SKB TECH, South Korea. Cellulose Nanofibers (CNFs, diameter ~22.5 nm and length ~5 μm) were obtained base

on the 3 times bleached red algae. The red algae is widely distributed marine plant in ocean. In this paper, the red algae was bleached three times for make the fibril soften and remove impurities such as tiny sea creature. It was simply prepared using a valley beater for dissociation and refinement. After pretreatment, the grinding was performed to reduce the length of the cellulose nanofiber. The grinding was performed using a micro grinder (Super Masscolloider, Masuko Sangyo Co., Ltd, Japan) at 1,500 rpm and the stone spacing of -150 ㎛. 1,2-Hexanediol ($C_6H_{14}O_2$, 99%) was used as agent for the antiseptic in CNF slurry.

The total 1 wt % of CNC and CNF fluids were prepared base on the distilled water (DW). All the fluids, the ultrasound treatment was performed for 60 minutes before the measurement.

III. Experiment and Result

The shape of particles was confirmed to 120kV Transmission Electron Microscope (Thermo Fisher (Talos L120C)). Figure 1 shows the difference according to the shape of CNCs and CNFs. First, CNCs does not contain the non-crystalline region, so it can be manufactured in a smaller and sphericity shape than CNFs. The CNFs can be seen the fibers, and it has been softened through bleaching and dissociation in the existing red algae. After, it is reduced to a nano size through grinding as mechanical method. Through the figure 1 confirmed the successful production of CNFs, and it showed that is formed long fiber bundles. Relatively, it can be seen that CNFs has a larger surface area than CNCs. Relatively, it can be seen that CNFs has a larger surface area than CNCs. It means that the area where CNFs can be interact become larger than CNCs area.

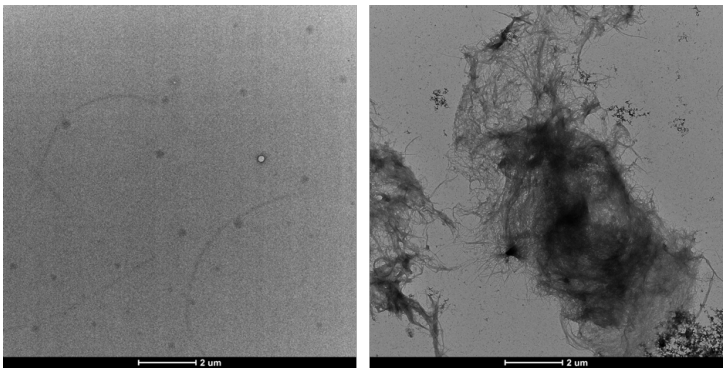

Figure 1. The TEM images of the CNCs(L) and CNFs(R).

The dispersion of particles was confirmed to UV/Vis spectrophotometer (X-ma3000 Series Spectrophotometer, Human Co., Ltd., South Korea). Figure 2 shows the difference according to the dispersion of CNCs and CNFs. The absorbed wavelength showed according to the electronic structure of an atom or molecule, and the absorbance showed the concentration of fluid [7]. Therefore, it is possible to extract of the qualitative dispersibility in the ultraviolet-visible light region with the absorbance and nanometer wavelength. As a visual photograph, both of CNCs and CNFs have excellent dispersibility. However, through the figure 2 shows that CNFs has high dispersibility due to the large surface area of abundant -OH groups compared to spherical CNCs. This result means that the interaction with solvent was smoother for CNFs than for CNCs, and it can be said that the Van der Waals force was relieved.

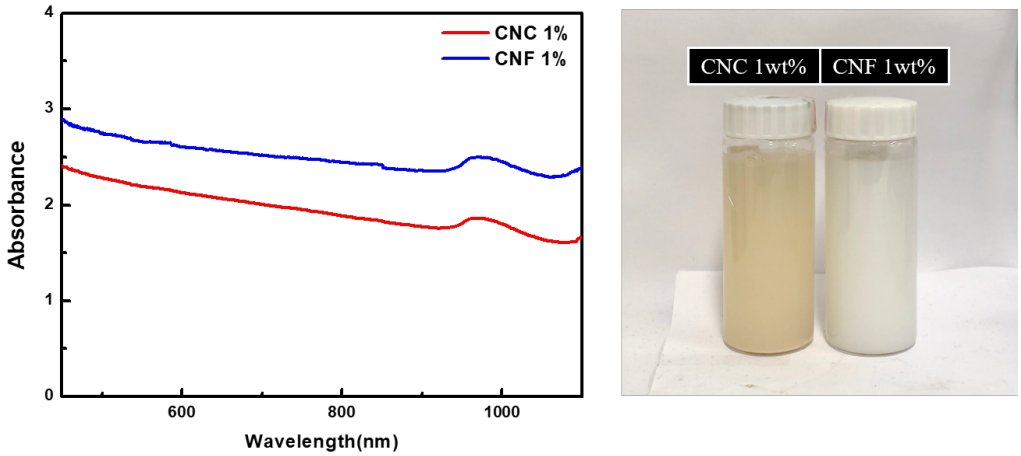

Figure 2. Comparsion of the CNCs and CNFs dispersion accroding to their molecular structure (The UV-vis spectrum graph(L) and visual image(R)).

Thermal conductivity of particles was confirmed to the Lambda system (Willingshausen, Germany), which is based on the transient hot-wire method. The thermal conductivity is one of the important indicators for nanofluids. This can explain the properties of particles with Brownian motion [8], and can also evaluate the motion of particles with respect to volume. Figure 3 shows the thermal conductivity of distilled water, CNCs fluid, and CNFs fluid. Also, the image on the right represents the thermal conductivity system. The thermal conductivity was compared CNCs fluid and CNFs fluid with distilled water as the standard point, and measured at temperatures ranging from 20 °C to 40 °C in total. First, CNCs fluid was noticed significantly lower thermal conductivity than distilled water. CNFs fluid was noticed higher thermal conductivity than CNCs fluid, but also lower than distilled water. This result means that the thermal conductivity of cellulose is low, unlike the tendency to increase as the volume of particles increases. However, CNFs was higher than CNCs that is a difference in the inclusion of the non-crystalline region. This is due to the non-crystalline region that structure is easy to penetrate because the gaps are not relatively tight, it can be easily accepted the molecules of the solvent.

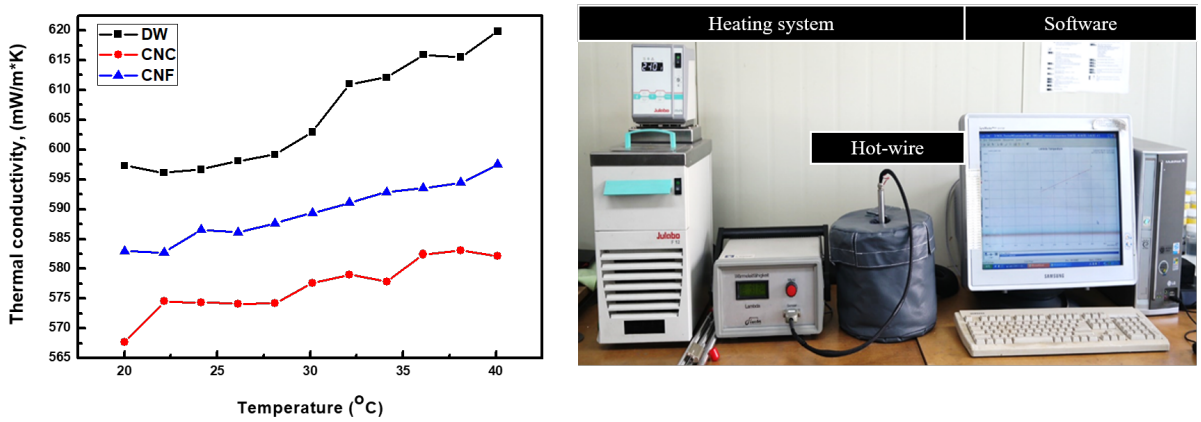

Figure 3. The thermal conductivity graph(L) and measrment component(R).

Thermal analysis of particles was confirmed to the Thermal analyzer (TA, Q600). Thermogravimetric analyzer (TGA) indicates the change in mass of the sample by applying temperature to the sample [9]. When the sample is heated, sequential reactions occur according to the characteristics of each component, which can be confirmed by

transition of the weight. It is mainly an experiment suitable for particles as a method to confirm the weight of the sample in a solid state. In this experiment, Thermogravimetric from 22°C to 890°C was analyzed. Figure 4 show the thermal analysis graph of CNCs and CNFs. In the figure 4, the first dehydration reaction of CNC occurs at about 100 °C, and the decomposition reaction occurs after about 200 °C. After that at approximately occurs 300 °C a residual. The residual occurs through a total of two reactions, the first loss of 7.91%, and the second loss of 5.65%. The first dehydration reaction of CNF starts at about 40 °C, the decomposition reaction ends at 400 °C, and formed the residual. Similarly, CNF shows a large decomposition reaction with the loss of 0.78% in the first, and the loss of 9.21% after. CNC and CNF have a fast dehydration reaction, and a difference in the origin of the decomposition reaction. CNC decomposes at a lower temperature than CNF that has a long dehydration reaction, and appears to be the result of hydrophilic fibrous strands.

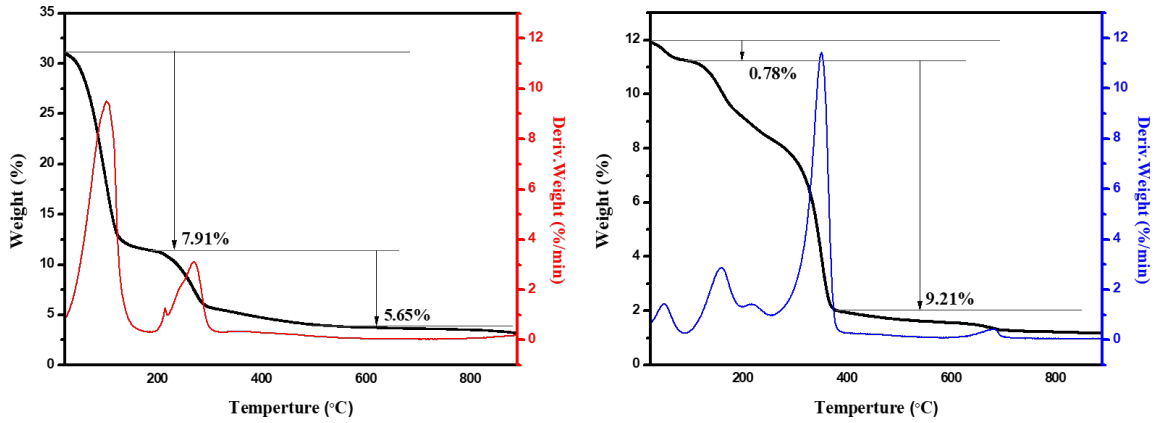

Figure 4. The CNCs(L) and CNFs(R) TGA curves.

IV.CONCLUSION

In this study, we were analyzed the sphericity CNCs and the long bundle CNFs the reaction in a solvent according to the structure and shape. First, the structural difference between CNCs and CNFs were explained in the TEM image, and it was confirmed that the fibrous form of CNFs had a larger surface area than spherical CNCs. The dispersion of CNFs was higher than CNCs, due to CNFs has the large surface to interact with solvent. Cellulose has low thermal conductivity, so both CNFs and CNCs were showed lower thermal conductivity than distilled water. The heat loss tended to be slower CNFs than CNCs. CNC decomposes at a lower temperature than CNF that has a long dehydration reaction, and appears to be the result of hydrophilic fibrous strands. Although the CNC particles have the advantage of being smaller due to spherical structure, it was observed that the CNFs with the large surface area of the fibrous showed higher performance. This results can be seen the difference by whether the inclusion of the crystalline region, and the interaction with the solvent.

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