Cellulose Nanofibril Grades' Effect on the Strength and Drainability of Security Paper

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The aim of this study was to evaluate the effect of the grades of cellulose nanofibril (CNF) on the strength and drainage of security paper made from cotton lint mixed pulp (CLMP). Refined CNF (RE-CNF), enzymatic CNF (EN-CNF), and carboxymethylated CNF (CM-CNF) were prepared, and their characteristics were analyzed. Handsheets were made *via* the addition of three CNFs into CLMP furnish, and their physical properties were measured. The drainability of the CLMP in the presence of CNFs was also determined depending on the grades and the dosage of the CNFs. The CM-CNF was the most effective at enhancing tensile strength by 50%, folding endurance by 464%, and sheet density by 10% when 5% of CM-CNF was added in the CLMP furnish. Moreover, 5% of EN-CNF improved tensile strength by 30% and folding endurance by 156%, but it was less effective as a paper strength promotor than CM-CNF and RE-CNF. A dramatic drainage reduction by 34% was observed when 5% of CM-CNF was added into the CLMP furnish, and EN-CNF presented the highest drainage rate. A high dosage of CNFs deteriorated the furnish drainage and promoted the strength. Therefore, papermakers should select the proper grade and the dosage of CNF for the manufacture of high-strength security paper.

Keywords: Cellulose nanofibril (CNF); Security paper; Refining; Enzymatic; Carboxymethylation; Paper strength; Drainage

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INTRODUCTION

The demand for security papers has rapidly increased because of their use as banknotes, checks, share certificates, lottery tickets, and airline tickets (Bobalek and Hamza 2016; Park *et al*. 2018). Security papers require high strength and high durability because they are used in harsh service environments. Many types of raw materials have been utilized so that security paper meets these requirements, but they have not been steadily and widely applied because most of them are not eco-friendly and are artificially synthesized products. Therefore, a new eco-friendly strength enhancer, along with its application technology, should be developed to manufacture strong and durable security paper (Park *et al*. 2018).

The term "nanocellulose" refers to cellulosic extracts or processed materials that have defined as nanoscale structural dimensions (Abitbol *et al*. 2016; Nechyporchuk *et al*. 2016). Nanocellulose has a low density, high aspect ratio, high biodegradability, and high tensile strength and stiffness (Salas *et al*. 2014; Park *et al*. 2018; Xu *et al*. 2018). Due to these excellent physical and chemical properties, nanocellulose has attracted growing scientific and technological interest from various research and industrial fields (Rajinipriya *et al*. 2018; Xie *et al*. 2018). Nanocellulose can be divided into three types: (i) cellulose nanocrystals (CNC), (ii) cellulose nanofibrils (CNF), and (iii) bacterial cellulose (BC) (Ling *et al*. 2018; Sheikhi *et al*. 2019); however, CNC and CNF are much more commonly reported (Nechyporchuk *et al.* 2016).

Both CNC and the CNF are utilized in different ways because they have significantly different dimensions and crystallinity (Xie *et al.* 2018). The CNFs have been thought to be useful in applications such as packaging, painting, printed electronics, paper, composites, and medicines (Rol *et al*. 2019). Particularly, the unique properties of CNF have created interest in a new family of paper components (Boufi *et al*. 2016). Many researchers have reported that CNFs can be effective in making strong paper as well as barrier-coated packaging paper products (Boufi *et al*. 2016; Ankerfors *et al*. 2017; Hollertz *et al*. 2017; Tyagi *et al*. 2019). Moreover, CNFs improve the physical properties of paper, such as tensile strength and density, much more than conventional strength enhancers. However, the most serious problem of adding CNF to the wet-end part is the deterioration of drainability (Brodin *et al*. 2013; Boufi *et al*. 2016). The authors' previous work (Park *et al*. 2018) investigated the effect of cellulose micro/nanofibrils made from hardwood bleached kraft pulp on the strength and drainage of security paper. It was found that micro/nanofibrils made by microgrinding enhanced the strength of security paper significantly, but the fibrillation degree and the dosage of CNF should be controlled while considering both the strength and drainage. However, the effect of CNF grades made by the chemical and enzymatic pretreatments on the strength and drainage of security paper was not investigated.

The aim of this study was to evaluate the effect of CNF grades made *via* different pretreatments on the strength and drainage of specialty paper made from cotton lint mixed pulp (CLMP). The fiber width, low-shear viscosity, and zeta potential of CNFs were analyzed, and the sheets were made *via* the addition of CNFs into CLMP furnish in a laboratory. The tensile strength, folding endurance, and the bulk of the sheets were measured, and the drainage rate of CLMP furnish in the presence of CNFs was measured.

EXPERIMENTAL

Materials

Hardwood bleached kraft pulp (HwBKP), supplied by Moorim Paper (Jinju, Republic of Korea), was used to make RE-CNF. The EN-CNF and CM-CNF were supplied by Moorim P&P (Ulsan, Republic of Korea). Table 1 shows the grades of CNF used in this study.

Table 1. Manufacturing Conditions of CNFs Used in this Study

CNF Grade	Pretreatment	Mechanical Treatment	Production
RE-CNF	Refining	Microgrinding (pass number = 9)	Laboratory
EN-CNF	Enzymatic hydrolysis	Homogenization	Pilot machine
CM-CNF	Carboxymethylation (Carboxylate group = 400±50 μmol/g)	Homogenization	Pilot machine

The CLMP was obtained from KOMSCO Co., Ltd. (Daejeon, Republic of Korea) and was used to prepare the handsheets and to measure the drainage rate in the laboratory. Ethyl alcohol (C_2H_5OH, 95.0%; Daejung Chemicals & Metals Co., Ltd., Siheung, Republic of Korea), acetone (CH_3COCH_3, 99.9%; Thermo Fisher Scientific, Waltham, MA, USA), and n-hexane (C_6H_{14}, 95.0%; Daejung Chemicals & Metals Co., Ltd., Siheung, Republic of Korea) were used for the solvent exchange of CNF pads to measure the fiber width.

Methods

Manufacture of RE-CNF made from bleached hardwood kraft pulps

The RE-CNF was manufactured by refining and microgrinding in the laboratory. The HwBKP with 1.57% solid content was soaked in tap water and then refined to 450 ± 5 mL CSF using a laboratory valley beater (PTI Laborausrüstung Laboratory Equipment, Vorchdorf, Austria). The refined pulp slurry was diluted to 1.0% consistency for fibrillation. The pulp slurry at 1.0% solid content was then fibrillated using a Super Mass Colloider (MKZA6-2; Masuko Sangyo Co., Ltd., Kawaguchi, Japan) at 1,500 rpm. The pulp slurry was fed continuously to the grinder, which consisted of two stone grinding disks positioned on top of each other. The gap between the two disks was adjusted to -150 μm. The RE-CNF slurry was corrected at a pass number of nine.

Characterization of CNFs

The fiber width of CNFs was analyzed using a field emission-scanning electron microscope (FE-SEM) (JSM-7610F; JEOL Ltd., Tokyo, Japan) to ensure the material was manufactured to the nanoscale. Wet CNF pads were prepared as test specimens for the measurement of the fiber width using the vacuum filtration system. The wet CNF pads were dried using the solvent exchange method and with ethyl alcohol, acetone, and n-hexane to complete the specimens. After the FE-SEM images of the pads were captured, the fiber widths of 100 individual nanofibrils for each CNF sample were measured using three-dimensional (3D) image software (MP-45030TDI; JEOL Ltd., Osaka, Japan); the average value was calculated, as was the standard deviation of the fiber width of the CNF samples.

The low-shear viscosity and zeta potential of the CNF samples were measured to evaluate their main characteristics. The low-shear viscosity was determined using a low-shear viscometer (DV-IP; Brookfield Engineering Laboratories, Inc., Middleborough, MA, USA) with a spindle number of 64 and a speed of 60 rpm. The zeta potential was measured using a zeta potential analyzer (Nano ZS; Malvern Panalytical, Malvern, UK).

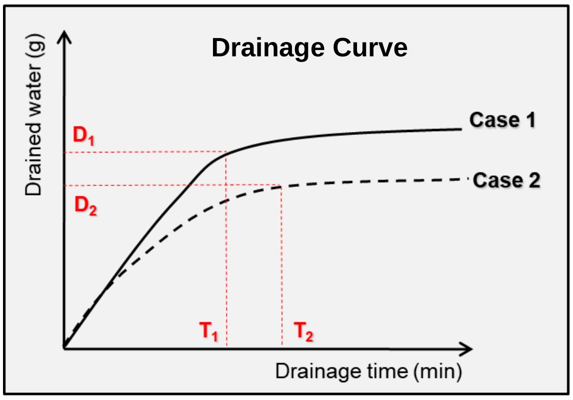
Handsheet manufacture and physical property measurement

The CLMP at 1.57% solid content was soaked in tap water and then refined to 170 ± 5 mL CSF using a laboratory Hollander beater. The refined pulp slurry was then diluted to 0.7% consistency for handsheet preparation. Handsheets with a grammage of 100 ± 5 g/m^2 were produced according to TAPPI T205 sp-06 (2006) after the CNF suspension was added to the pulp slurry and mixed for 2 min at 600 rpm. The dosages of CNF suspension were 1%, 3%, and 5% of oven-dried fibers. The handsheets were wet-pressed at 345 kPa for 5 min and then dried at 120 °C using a laboratory wet-press (model 326; Wintree Corporation, Osaka, Japan) and a cylinder dryer (Daeil Machinery Co., Ltd., Daejeon, South Korea), respectively.

The sheets were conditioned at 23 ℃ and 50% relative humidity (RH) to maintain their moisture content at 8%. The tensile strength, according to TAPPI T494 (2006), folding endurance, according to TAPPI T511 (2008), and bulk, according to TAPPI T411 (2010) were measured to identify the effect of the CNFs on the physical properties of the sheets.

Drainage measurement

The CLMP at 1.57% solid content was soaked in tap water and then refined to 170 ± 5 mL CSF using a laboratory valley beater. The drainage tests were performed in the same manner as described in the authors' previous study (Park *et al*. 2018). Before the drainage tests, the refined CLMP furnish was diluted to 0.7% consistency. The drainage tests were completed using a dynamic filtration system (DFS, Bonnier Technology Group, Eclépens, Switzerland). The stirring chamber was filled with 860 g of 0.7% CLMP furnish, and then the furnish was stirred for 30 s at 600 rpm before the addition of a CNF suspension. After the CNFs were added into the CLMP furnish, the mixture was stirred for 5 min at 600 rpm. The dosages of the CNF suspension were 1%, 3%, and 5% of oven-dried fibers. Filtration through a 60-mesh screen began immediately after the final stirring for 5 min, and the filtrate was continuously measured for 10 min. The drainage curve was obtained with the amount of drained water as a function of drainage time, and the drainage rate was calculated, as shown in Fig. 1.



$$\text{Drainage rate (g/min)} = \frac{\textit{Maximum drainage weight (D)}}{\textit{Maximum drainage time (T)}}$$

Fig. 1. Determination of the drainage rate of CLMP furnish in the presence of CNFs (Park *et al*. 2018)

RESULTS AND DISCUSSION

Characteristics of the Three CNFs

The FE-SEM was used to measure the fiber size and the dispersion properties of the CNFs used in this study. The FE-SEM images of CNF pads were captured, and the fiber width of CNFs was measured using 3D imaging software (Nie *et al*. 2018). Figure 2 shows the FE-SEM images of the CNFs. All of the CNFs showed a lower fiber width than 100 nm, which is a standard between micro- and nanofibrils (Chinga-Carrasco 2011; Isogai *et al*. 2011). Figure 2 shows that the RE-CNF had a fibrous shape with a nanometer-sized fiber width; various nanofibril bundles were also observed. The EN-CNF presented regular long nanofibril bundles, and CM-CNF exhibited regular short nanofibrils. Figures 3 and 4 show the average fiber width and the fiber width distribution of the CNFs. The average fiber width of RE-CNF was the highest and that of CM-CNF was the lowest. The fiber width range for RE-CNF was wider than that of EN-CNF and CM-CNF, which indicated that the CNFs made using only mechanical treatments had relatively lower regularity of nanofibrils than the ones made by chemical or enzymatic pretreatments (Henriksson *et al*. 2007; Qing *et al*. 2013).

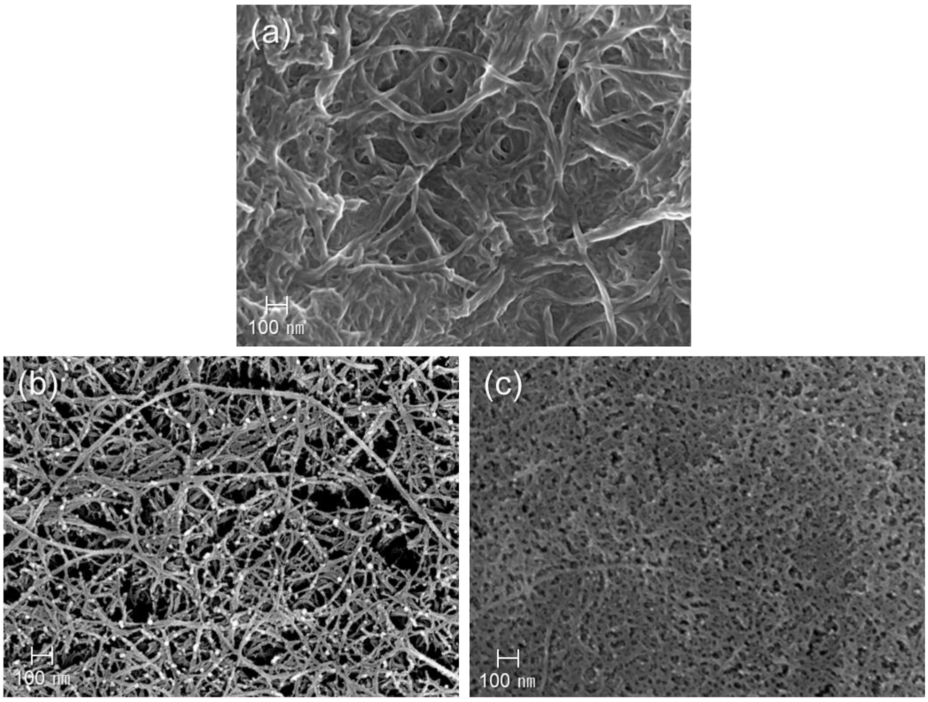


Fig. 2. FE-SEM images of (a) RE-CNF, (b) EN-CNF, and (c) CM-CNF

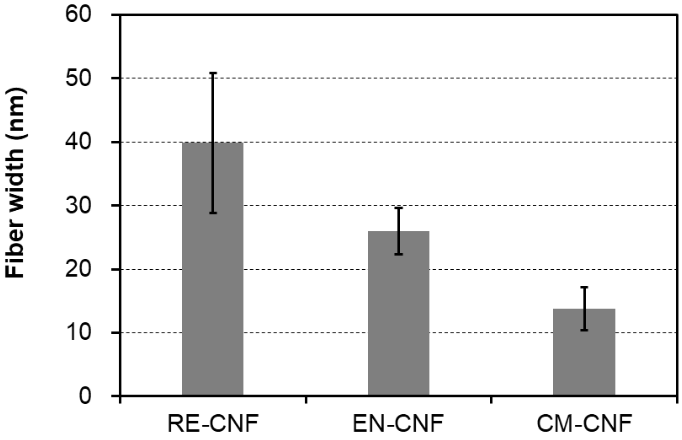


Fig. 3. Average fiber width of CNFs

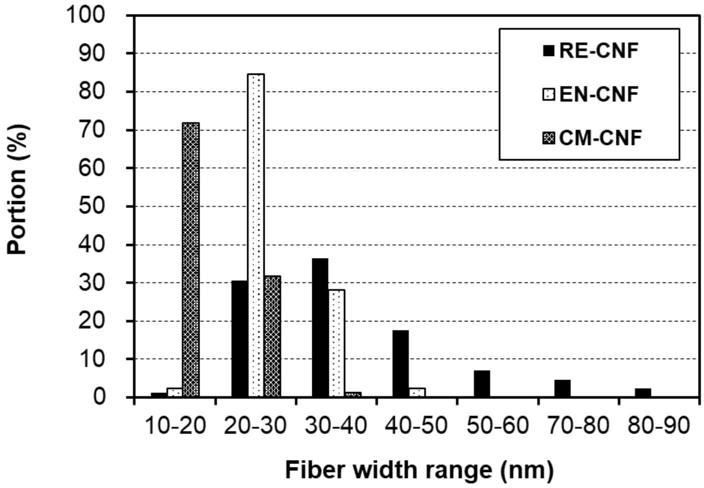


Fig. 4. Fiber width distribution of CNFs

Figure 5 shows the low-shear viscosity of the CNF suspensions with 1% concentration. The EN-CNF and CM-CNF showed the lowest and the highest viscosity, respectively. The viscosity of CNF has been reported to be proportional to the nanofibril content in the suspension (Lasseuguette *et al*. 2008). Because EN-CNF consisted of regular and long narrow fibril bundles and had low nanofibril content, its viscosity was lower than that of other CNFs. Meanwhile, the viscosity of CM-CNF was the highest because of the high content of short and narrow nanofibrils.

Figure 6 shows the zeta potential of the CNFs. All of the CNFs were made from HwBKP, which carried anionic charges mainly due to carboxyl groups (Hubbe and Rojas 2008), so they showed negative zeta potential. The CM-CNF presented the lowest zeta potential and the highest electrical stability, as CM-CNF was pretreated by a carboxymethylation reaction before homogenization; a relatively higher anionic zeta potential was formed by carboxymethyl groups (Wang *et al*. 2015; Shui *et al*. 2017), as compared to other CNFs used in this study. Martelli-Tosi *et al*. (2016) reported that the cellulose nanofiber produced by commercial enzymes had zeta potentials ranging from -20.8 mV to -24.5 mV, and the zeta potential of EN-CNF used in this study was approximately -26.0 mV. Thus, the EN-CNF showed a similar zeta potential as that reported by Marelli-Tosi. The RE-CNF showed the highest zeta potential because it was not treated with any chemicals or enzymes.

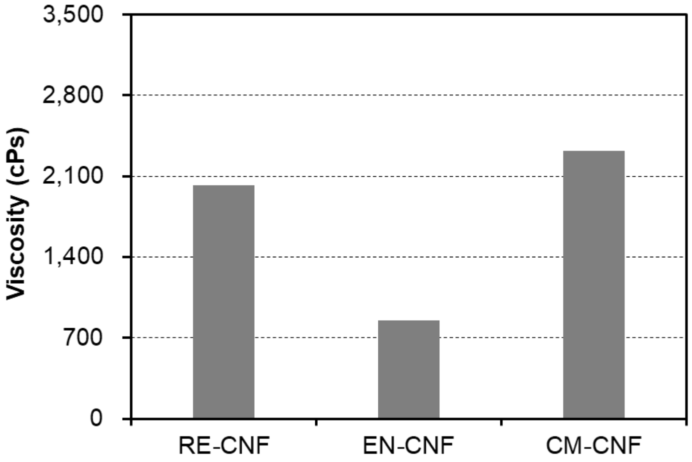


Fig. 5. Average low-shear viscosity of CNFs at a consistency of 1.0% (23 °C)

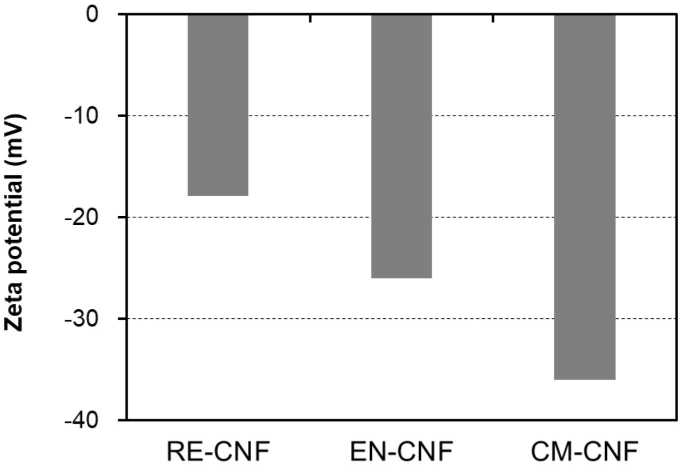


Fig. 6. Average zeta potential of CNFs

Evaluation of the Physical Properties of Sheets in the Presence of CNFs

The sheets were made in the laboratory to investigate the effect of CNF grades on the physical properties of specialty papers made from CLMP. Figures 7 and 8 show the effect of CNFs on the tensile index and the folding endurance of handsheets. All of the CNFs rapidly improved the tensile index and folding endurance. As the dosage of CNFs increased, the strengths of the sheet linearly increased. The enhancement of sheet strength is a direct consequence of the increase in specific surface area (Brodin *et al*. 2015; Park *et al*. 2018). Such an increase promotes the formation of fiber-fiber bonds, which consolidate paper structure and increases the sheet density (Brodin *et al*. 2015; Boufi *et al*. 2016; Park *et al*. 2018). The strengths of CM-CNF and RE-CNF improved more than the EN-CNF. Particularly, CM-CNF had the highest improvements in tensile index and folding endurance, and EN-CNF showed the lowest improvement in sheet strengths. The CM-CNF showed the highest tensile strength and folding endurance, and the lowest bulk, as shown in Fig. 9. This was attributed to the fact that the CM-CNF suspension had a higher nanofibril content than the other CNFs. The promotion in folding endurance by CNFs was much higher than in the tensile index; the highest promotion in paper strength was observed in the presence of CM-CNF, which had a higher viscosity than both RE-CNF and EN-CNF.

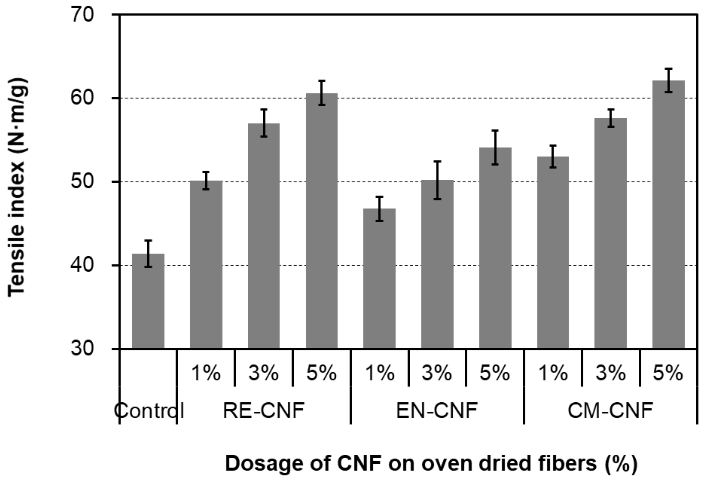


Fig. 7. The effect of CNFs on the tensile strength of sheets

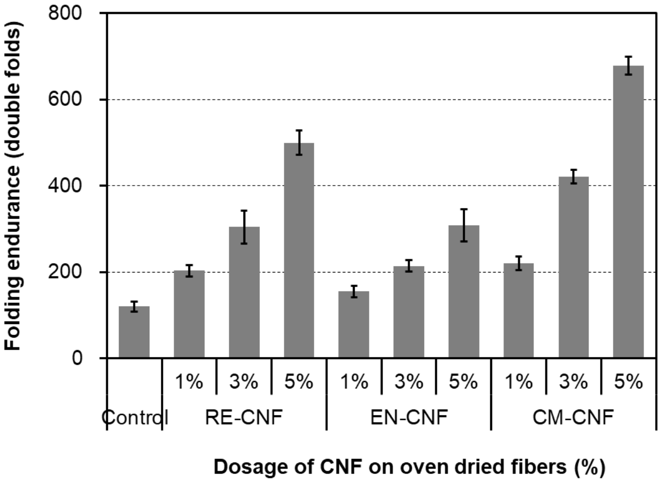


Fig. 8. The effect of CNFs on the folding endurance of sheets

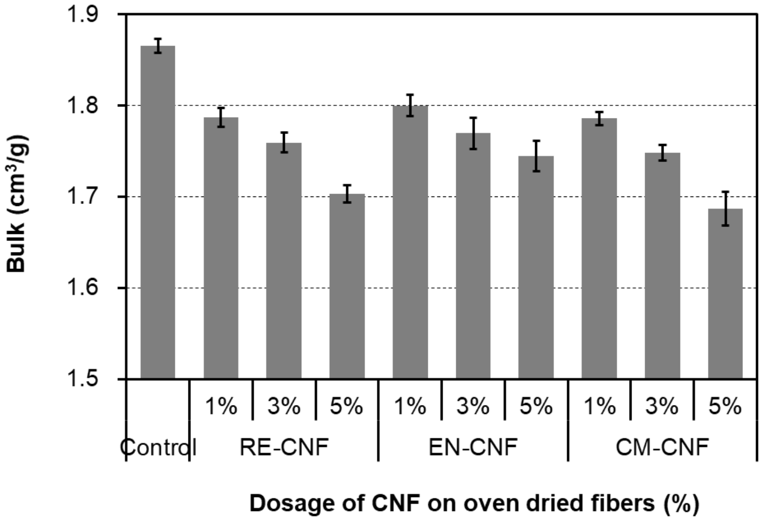


Fig. 9. The effect of CNFs on the bulk of sheets

Therefore, it was concluded that CM-CNF improved the tensile strength and the folding endurance most effectively, and the viscosity of CNF was the most important parameter to consider when selecting the grade of CNF to enhance the paper strength.

Evaluation of the Drainage Rate of CLMP Furnish in the Presence of CNFs

The deterioration of drainage by adding CNF to pulp suspension is one of the major limitations for utilizing CNF in the papermaking process. Drainage is a critical parameter because it limits the production efficiency of the paper machine (Boufi *et al*. 2016). Therefore, the effect of CNF on the drainage of the furnish should be analyzed before utilizing it as a paper strength additive (Park *et al*. 2018).

The drainage rate was calculated from the drainage curve to evaluate the effect of CNFs on the drainage of the CLMP furnish. Figures 10, 11, and 12 show the drainage rate of the CLMP furnish depending on the grades and the dosage of CNFs.

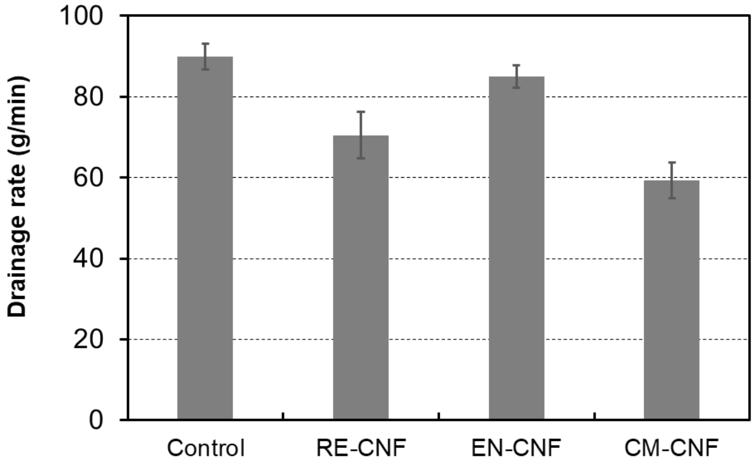


Fig. 10. The effect of CNFs on the drainage rate of CLMP furnish at CNF dosage of 1%

The addition of CNFs resulted in the deterioration of the drainage rate, as the dosage of CNFs increased irrespective of the CNF grades. A dramatic drainage reduction was observed when CM-CNF was added into the CLMP furnish. The EN-

CNF presented the highest drainage rate compared to the other CNFs. The nanofibrils of CNF, present in the wet web, decrease the available pore area and lengthen the capillaries required for water flow (Taipale *et al*. 2010). Moreover, they also increase the total surface area of the furnish, which enhance water retention through hydrogen bonding (Taipale *et al*. 2010) and cause drainage resistance (Hubbe and Heitmann 2007; Park *et al*. 2018). Therefore, the CM-CNF with the highest viscosity showed the lowest drainage rate among three CNFs.

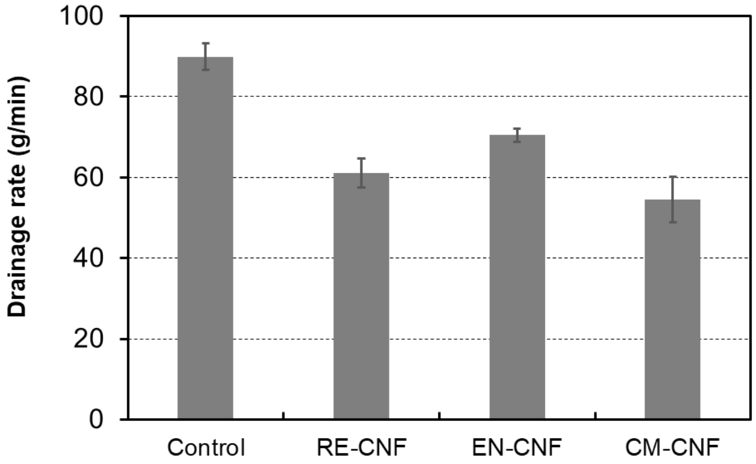


Fig. 11. The effect of CNFs on the drainage rate of CLMP furnish at CNF dosage of 3%

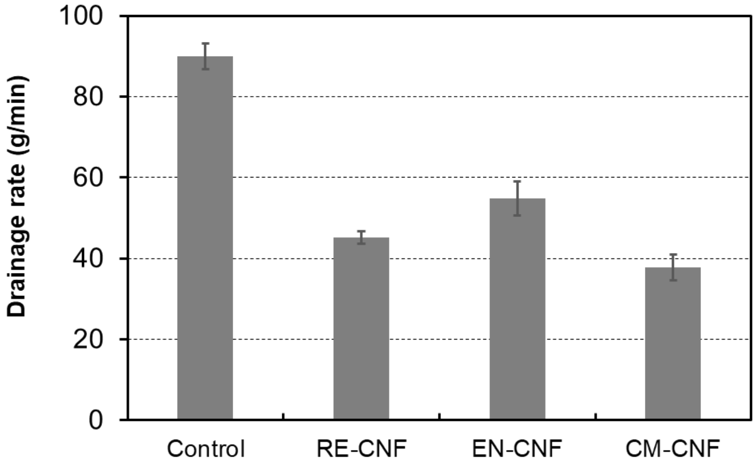


Fig. 12. The effect of CNFs on the drainage rate of CLMP furnish at CNF dosage of 5%

Finally, the CNF with high viscosity and high nanofibril content effectively promoted strength, but it also deteriorated the furnish's drainability. The dosage of CNF also directly affected the strength and the drainage rate. Therefore, papermakers should select the proper grade and dosage of CNF for the manufacture of high-strength specialty paper.

CONCLUSIONS

1. Refined CNF, enzymatic CNF, and carboxymethylated CNF were prepared, and their characteristics were analyzed. The average fiber width of RE-CNF was the

highest and that of CM-CNF was the lowest; the range of fiber widths for RE-CNF was higher than that of EN-CNF and CM-CNF. The CM-CNF resulted in the highest viscosity, the lowest fiber width, and the most negative zeta potential among the three CNFs. The EN-CNF showed the opposite characteristics to CM-CNF, and RE-CNF presented intermediate characteristics between the CM-CNF and EN-CNF.

2. The CM-CNF was the most effective for enhancing tensile strength, folding endurance, and sheet density. The EN-CNF also increased the paper strength, but it was less effective at promoting paper strength than both CM-CNF and RE-CNF.
3. The addition of CNFs resulted in the deterioration of the drainage rate as the dosage of CNFs increased. A dramatic drainage reduction was observed when the CM-CNF was added into the CLMP furnish. The EN-CNF presented the highest drainage rate among the CNFs.
4. Papermakers should select the proper grade and the dosage of CNF for the production of high-strength specialty paper. EN-CNF may be advantageous in the papermaking process where drainage is more critical than strength improvement. It is preferable to select CM-CNF in the papermaking process where the drainage can be afforded and the strength improvement is a priority. Finally, CM-CNF requires a lower dosage than EN-CNF to achieve the same level of strength.

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