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ORIGINAL RESEARCH

Characterization of spray dried cellulose nanofibrils produced by a disk refining process at different fineness levels

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Abstract Three types of wood pulp feedstocks including bleached softwood kraft, unbleached softwood kraft and old corrugated containers were disk refined to produce cellulose nanofibrils at different fineness levels ranging from 50 to 100%, and the resulting aqueous suspensions of cellulose nanofibrils were spray dried. The spray drying experiments were carried out to examine different processing conditions for the different CNF feedstock types and fines level at various suspension concentrations to produce dry samples with free-flowing powder morphologies. The fineness levels and solids contents of CNF suspensions were set to 80% or more and 1.8% or less, respectively. If the solids content of the CNF solutions was high and the fibrillation level was low, plugging was experienced in the spray head because of the high viscosity of the suspensions, resulting in production of poor-quality powders. In terms of reduction in processing energy, even if the CNF suspension solids content was increased to 1.5 wt.%, the powder quality and the production yields were excellent. It was confirmed that high-quality powder under 20 μm were produced at a 90% fibrillation level of all CNF feedstocks. The resulting dry CNF powders were characterized to determine particle size distributions and morphological properties via a scanning electron microscope and a laser diffraction particle size analyzer. The particle sizes were smaller at higher fibrillation levels and lower solids content of the CNF suspensions. The CNF suspension derived from bleached kraft pulp, the average particle size decreased by 43% and 33% with the lowered solids contents from 1.8 to 1%, and the increased fineness levels from 80 to 100%, respectively.

Keywords Cellulose nanofibrils · Spray drying · Bleached kraft pulp · Unbleached kraft pulp · Old corrugated cardboard pulp

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Introduction

Conventionally, hardwoods and softwoods are the feedstocks for bleached kraft pulp (BKP) or unbleached kraft pulp (UKP) production through pulping and refining processes, and those pulps are widely used in the paper industry (Bajpai et al. 1992; Sixta et al. 2006). BKP is produced by removing lignin and extractives from UKP using a bleaching agent (Hans 2006; Jin et al. 2022). UKP is the virgin pulp that has not undergone the bleaching process, is cheaper than BKP, and is widely used in filter papers, hygiene papers, cardboard boxes, etc. (Hubbe et al. 2003; Pei et al. 2013). After a cardboard box is used and becomes waste, it can be classified as an old corrugated container (OCC) which can be recycled for other applications (Ryu et al. 2001; Jahan et al. 2016; Chibani et al. 2016). OCC is approximately 75% cheaper than BKP. As the use of delivery services with packaging increases, recyclable OCC is increasingly valued as a feedstock for eco-friendly materials (Ruamsook and Thomchick 2015; Copenhaver et al. 2021). OCC is commonly recycled as a raw material for cardboard boxes. OCC fibers tend to become stiff and rigid through repeated recycling processes known as "hornification", leading to the application of OCC in the middle layer of various paper or box board products (Diniz et al. 2004; Lin et al. 2020; Sangtarashani et al 2020).

BKP, UKP, and OCC are good feedstock sources for manufacturing cellulose nanofibrils (CNFs) via mechanical treatment (Yousefhashemi et al. 2019; Kargupta et al. 2021). The delivered physical shear forces for breaking down the cellulose fibers lead to an increase in the number of hydroxyl groups with an increased specific surface area. (Habibi et al. 2010; Charreau et al. 2013; Abdul et al. 2014). Thus, a porous network with strong fibril-fibril interaction is formed, which are utilized in pulp and paper, and also in lithium-ion separation media, aerogels, coatings, and polymer nanocomposites (Dufresne and Belgacem 2013; Oksman et al. 2016; Desmaisons et al. 2017; Gou et al. 2021). In particular, the thermal-disk refiner enables mass production of CNFs using only water as a production medium, and the utilization of thermal processes involving steam reduces the energy consumption required to produce CNFs (Chen et al. 2019). Furthermore, alkaline, oxidation, and enzyme pretreatments that are used prior to mechanical treatments to reduce the energy consumption for disintegrating pulp fibers to cellulose nanofibrils have been reported (Dufresne et al. 1997; Paakko et al. 2007; Osong et al. 2016).

Commonly, CNFs produced through mechanical treatment are in an aqueous suspension that can be subject to bacterial contamination and high shipping costs (Eyholzer et al. 2010). In addition, to properly utilizing CNFs in various applications, proper drying methods should be developed for the targeted industries (Pakowski et al. 2007, Gardner et al. 2008). For decades, researchers have explored the use of bio-fillers derived from both woody and non-woody materials as reinforcing fillers in thermoplastic matrices. The excellent distribution and dispersion of fillers within thermoplastic matrices are crucial for enhancing plastic properties. Wood flour has often been preferred filler for polymer matrix composites (PMCs) in the production of wood-plastic composites (WPCs) (Ashori et al. 2013; Mirmehdi et al. 2014). CNFs can be a viable alternative to wood flour attributable to its shorter fibril lengths and narrower fiber widths with high specific surface area (SSA), provided the CNFs undergo appropriate drying techniques. According to Peng et al. (2016), the tensile strength, tensile MOE, flexural strength, and flexural MOE increased by up to 11%, 36%, 7%, and 21%, respectively, compared to the neat polypropylene, after adding the 6 wt.% of spray-dried CNF powders and 2 wt.% of MAPP into the PP matrix. Peng used a lab scale-two fluid nozzle referred as to mini-spray dryer (Buchi B290 laboratory spray dryer New Castle, DE, USA) to dry CNF suspensions. Common methods to dry CNF suspensions include air-drying, oven-drying, freeze-drying, and supercritical drying (Eyholzer et al. 2010; Peng et al. 2012a, b; Peng et al. 2013; Gardner et al. 2013). The advantages of air- and oven-drying techniques are their scalability and low cost. However, these two drying techniques are not suitable for application CNFs into PMCs as a reinforcing filler due to the bulky materials formed, which have the largest agglomerates among all drying methods. Additionally, the freeze- and supercritical- drying can produce CNFs with nano-dimensions. However, agglomeration of CNFs during freeze-drying presents a challenge if freeze dried CNFs are applied in PMCs. Moreover, freeze-drying has a relatively higher cost compared to oven- and spray- drying due to its extended dehydration times (Fan et al. 2019). A disadvantage of drying CNFs via supercritical drying is the most expensive among all drying techniques and is not practical for scaling up (Peng et al. 2012b; Posada et al. 2020).

Spray-drying (SD) including different atomizing techniques such as a rotary disk atomizer, two-fluid nozzle, and ultrasonic atomizer is an excellent drying technology for producing dry powders from liquid suspensions (Huang et al. 2006; Kemp et al. 2016; Arpagaus et al. 2017). Spray drying is fast, simple, and scalable, so it is used in various industries including the pharmaceutical, food, and chemical manufacturing (Zhou et al. 2006; Gallardo et al. 2013; Sosnik and Seremeta 2015). Furthermore, spray-dried CNF (SDCNF) has been reported to have higher thermal stability and superior crystallinity index than fibers dried via other drying methods including air-drying, oven-drying, freeze-drying, and supercritical-drying (Eyholzer et al. 2010; Peng et al. 2012a, b; Peng et al. 2013; Gardner et al. 2013). Among the different techniques of spray-drying, the pilot scale-rotary disk atomizer has many advantages such as large capacity and drying efficiency at lower pressures and less feed blockage (Huang and Mujumdar 2008). In brief, the feedstock in liquid suspension and hot air are transported into the atomizer by a feed pump and hot air blower, respectively, then the centrifugal energy from rotation of the spinning disk atomizer and hot air are delivered to the feedstock. After the disintegration of liquid film into the formation of droplets, the droplets are evaporated creating dry particles. The resulting particles collide with the surface of the cyclone, leading to a loss of kinetic energy and causing the particles to fall into the collector (Teunou and Poncelet 2005; Woo et al. 2007; Chegini et al. 2012).

In this study, mass production of CNF powders was accomplished using a pilot-scale rotary disk atomizer. Different feedstock sources including BKP, UKP, and OCC were explored to manufacture the spray dried powders. Furthermore, different fibrillation levels denoted here by "fines percent" and variable solids contents of CNF suspensions were adjusted to reduce the energy consumption of the thermo-mechanical refiner and the pilot scale-rotary atomizer, respectively. The important factors, such as fines levels and solids contents of the CNF suspensions were assessed for their impacts on the resulting powder morphological properties, size distributions, and production yield.

Materials and methods

Cellulose sources

Three cellulose sources were utilized including: bleached softwood Kraft pulp (BSKP), unbleached softwood Kraft pulp (USKP) and old corrugated cardboard pulp (OCC). The cellulose nanofibril samples were obtained from the Product Development Center (PDC) at the University of Maine (Orono, Maine, USA) as suspensions at 3 wt.% solids content (US Patent, US 20170073893A1), which were then diluted to various solids contents (1.0 to 1.8 wt.%) for spray drying experiments. Each cellulose feedstock was supplied at three different fines levels (80%, 90%, and 100%) which represent the degree of fibrillation in the process of thermo-mechanical defibrillation of the sample pulp. The fines content (level) of CNF suspensions was reported based on the percentage of under 200 µm length fibers in the total amount of fibers. The fines content was measured via the MorFi Fiber Analyzer (TechPap, France), and the measurement was made using two cameras that measure the fibers in a 50-µm-wide chamber and then deliver the data to the software. The CNF suspensions produced at less than 80% fines level could not be spray dried into a powder form attributed to larger particle size distributions in the suspensions and difficulties in the drying process such as clogging of atomizer holes. Figure 1 shows the UMaine's thermo-mechanical refiner.

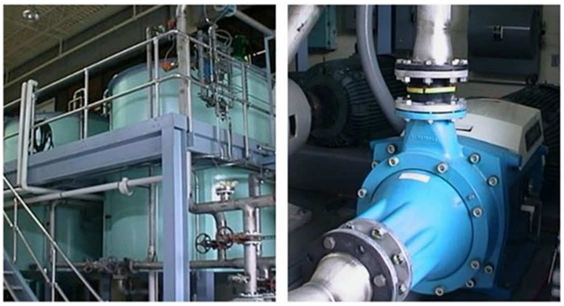

Fig. 1 UMaine thermo-mechanical refiner at process development center (PDC)

Spray drying

The drying conditions for all samples were inlet temperature of 240 ℃, outlet temperature of 125 ℃, bag house temperature of 118 ℃, air fan speed of 85%, heater capacity of 54%, pump capacity of 50%, and the disk spinning speed of 30,000 rpm. Figure 2 shows the UMaine's pilot-scale spray dryer, while Table 1 lists the spray drying conditions used in this study.

Sizes and morphological properties of the powder samples

The morphological properties of the CNF powders were measured via a Hitachi Tabletop Microscope SEM TM 2000 (Hitachi High-Technologies Corporation, Japan). SDCNF powders were placed on the SEM stub covered with carbon tape using a lab scoop. Air flow was then used to secure minor particles to the carbon tape. The set accelerating voltage was changed automatically between 5 and 15 kV. The particle size distribution was measured via a Mastersizer 2000 (Malvern, United Kingdom). 1 g of SDCNF powders were placed on the tray in the Scirocco 2000 attachment (Malvern, United Kingdom), and the powders analyzed with a particle refractive index of 1.53 (Liu and Smalyukh 2017).

Results and discussion

Production feasibility and yields

Table 2 shows the production feasibility of all CNF samples at different processing conditions. The effect of solids content is distinct on the production yields and morphological properties as determined by scanning electron microscopy (SEM). To calculate the production yield of the spray-dried CNF, the measured final weight of the spray-dried CNF was divided by the weight of the dried pulp input into the CNF suspensions, followed by multiplication by 100. The equation is as follows.

$$\text{Production yield } (\%): \frac{\text{Weight of SDCNFs}}{\text{Weight of dried pulp input}} \times 100$$

*Yield (%): Production yield of the spray-dried CNF powders as a percentage. *Weight of SDCNFs: Final measured weight of powders after spray-drying. *Weight of the dried pulp input: Weight of the dried pulp that was used to prepare the CNF suspensions.

The higher solids contents caused a decrease in the production yields of the CNF samples (Figs. 3, 4). The solids contents of 1.3 wt.% and 1.5 wt.% showed similar production yields, which implies that the production efficiency is not degraded with

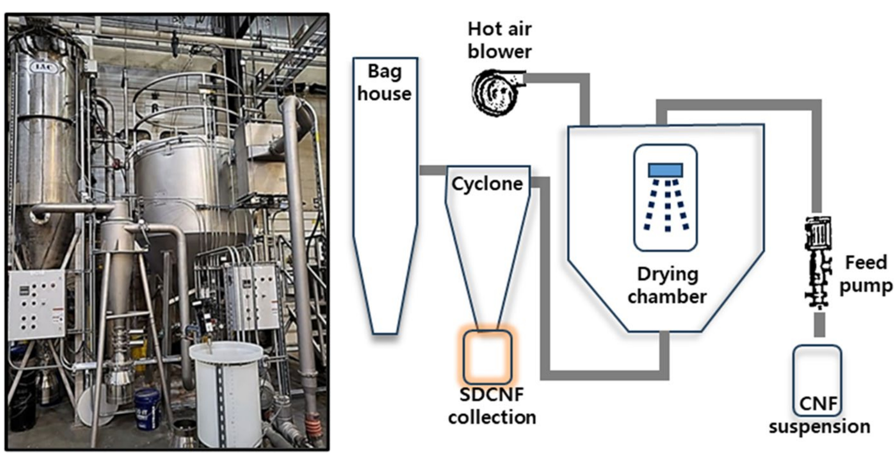

Fig. 2 UMaine pilot-scale spray dryer

Table 1 Conditions of spray drying for cellulose nanofibrils

	Inlet Temp, ℃	Outlet Temp, ℃	Bag house Temp, ℃	Spinning disk, RPM	Feeding rate, Kg/hr	Air speed, %
Conditions	240	125	118	30,000	17	85

Table 2 Production yields and processability of all samples of cellulose nanofibrils in spray drying

Resources	Fine level, %	Solids contents, %	Yield, %	Production quality
Bleached CNF	80	1.0	54	Good
Bleached CNF	80	1.3	53	Poor (high viscosity/plugging)
Bleached CNF	90	1.0	76	Good
Bleached CNF	90	1.3	70	Good
Bleached CNF	90	1.5	70	Good
Bleached CNF	90	1.8	55	Poor (high viscosity/plugging)
Bleached CNF	100	0.5	73	Good
Bleached CNF	100	1.0	85	Good
Bleached CNF	100	1.3	68	Good
Bleached CNF	100	1.5	69	Good
Bleached CNF	100	1.8	61	Fair & poor
Bleached CNF	100	2.0	61	Poor (plugging)
Unbleached CNF	90	1.0	73	Good
Unbleached CNF	100	1.0	83	Good
OCC	90	1.0	80	Good
OCC	90	1.5	-	-
OCC	100	1.0	82	Good

Fig. 3 Pictures of quality example of CNF powders **a** poor, **b** good

increasing the solids contents up to 1.5 wt.%. It is very valuable to report the maximum solids contents without a loss in production yield in terms of economical CNF powder production. Figure 5 shows the production yields of bleached CNFs, unbleached CNFs, and OCCs dried by spray drying at different fines levels (90% and 100%). The higher fines levels, which represents exposure of CNFs to a longer disk refining process, resulted in shorter fiber lengths of the dried samples, and tended to increase the production yield for all cellulose feedstocks. The fiber length in CNF suspensions were measured by a MorFi Compact (Techpap, Grenoble, France) at 0.005 wt.% (Kelly et al. 2021). The average fiber length of 80% fines level of bleached CNFs was about 405 µm, and the length was reduced by 25% and 31% after refining up to fines level of 90% and fines 100%, respectively. Although the changes in the production yields were minor for all cellulose feedstocks, the increasing yield trend was clearly observed. The shorter fibrils under average 350 µm of fiber lengths in suspension appear to be beneficial for improved production yields during the CNF spray drying process (Kelly et al. 2021). The longer fibril length in lower fines level of CNF suspensions and high solid contents, increases fiber entanglement. This leads to an increase in the solution's resistance to flow, resulting in the increased viscosity of CNF suspensions. The high viscosity can be a critical factor to hinder correct droplets formation. Figure 3a shows an example of the clumps of CNF powder produced after spray drying. Formation of these large clumps can be attributed to a lowered drying efficiency driven from less fibrillation and very high solids contents of the suspension. The high fibril length and solids content in suspension leads to clogging of the atomizer nozzle and agglomeration of particles (Peng et al. 2013). In contrast, individual fine particles can be formed with high production yields at appropriate fines levels and solids contents (Fig. 3b). The droplets begin

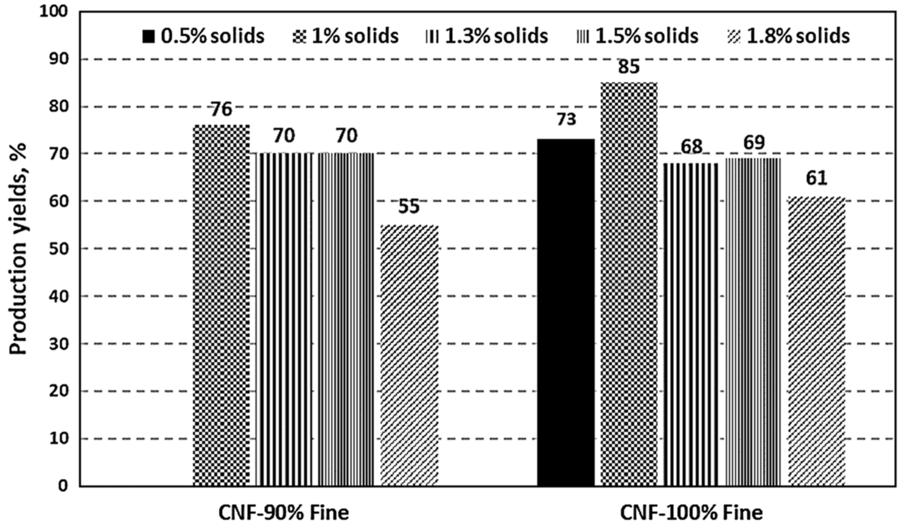

Fig. 4 Production yields of spray-dried cellulose nanofibrils with different solids contents

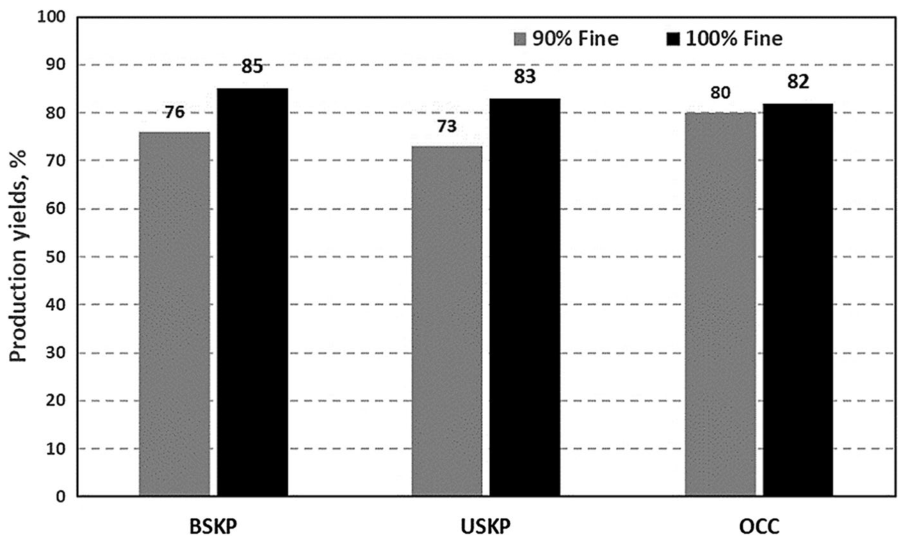

Fig. 5 Production yields of spray-dried cellulose nanofibrils from different feedstock sources

to evaporate rapidly in the drying chamber, achieving its maximum evaporation rate. This phase is referred to as the constant-rate drying period. During this phase, water from the interior of the droplet migrates to its surface, ensuring the surface remains saturated and allowing evaporation to continue at a consistent rate. When the rate of evaporation from the surface becomes faster than the rate at which water can migrate from inside the droplet to the surface, the constant-rate drying period is finished. The length of this period might depend on the water content of the droplet, the viscosity of the solution, and the temperature and humidity of the drying air. These factors affect the powder production feasibility and yields. At the critical moisture content following the constant-rate drying period, the droplet cannot maintain a saturated surface attributable to reduced moisture migration. The falling-rate drying period then begins. As the evaporation rate slows, the temperature of the droplet increases. The surface becomes unsaturated, and the evaporation rate decreases. A solid crust surrounds the droplet, and subsequent moisture removal is governed by this crust's permeability (Lukasiewicz et al. 1989; Parikh et al. 2005).

Suggestions on the best cellulose feedstock sources and optimal drying conditions.

Figure 6 shows the change of the production yield with solids content and fines level of the cellulose suspensions. It is noted that there are optimal combinations of these two variables that lead to the best production yields. A balancing between solids content and fines level is required for the optimum production of spray-dried CNF powder. The production of final powders increases with the higher solid content of CNFs, and the manufacturing cost for powder production decreases when the energy consumption of refining is reduced to produce CNF suspensions. It was determined that the solids content of 1.5 wt.% and 90% fines level of CNF suspensions would be the most preferred conditions in terms of drying quality and production efficiency in this research. All drying conditions remained unchanged, while solids contents and fines levels of CNF suspensions were changed. It is worth noting that the high quality of the produced powder was determined by the particles being individually dried, evidenced by the absence of fiber clumps in the collected powders. The interactions between the solids content and fines level must be considered for better estimation of the production yields. Furthermore, the energy consumption that is required to reach the targeted CNF fibrillation level through the disk refiner can be potentially reduced using OCC pulp that has already undergone a refining process at least once. Figure 7 shows a comparison of the energy consumptions of converting BSKP, USKP, and OCC pulps into CNFs at up to 90% fines level through a disk refiner. The energy consumption for refining USKP and OCC was measured to be less than 20% and 50%, respectively, compared to that of BSKP. According to Kelly et al. (2021), the refinement energy consumption targeting 80% fines level of BSKP and OCC were 1630 and 1140 kWh hr/MT, respectively, which shows that the energy consumption for refining OCC was significantly lower than that of BSKP. Moreover, the gap of energy consumption targeting 90% fines level between BSKP and OCC becomes greater (3378 vs. 1945 kWh hr/MT, respectively), and the difference increased even more at the 100% fines level of the two pulps.

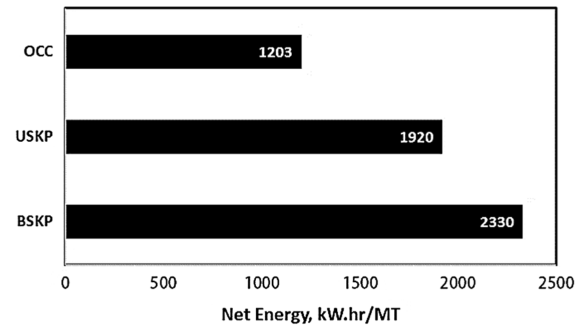

Fig. 7 Refinement energy consumption targeting 90% fines level for BSKP, USKP, and OCC

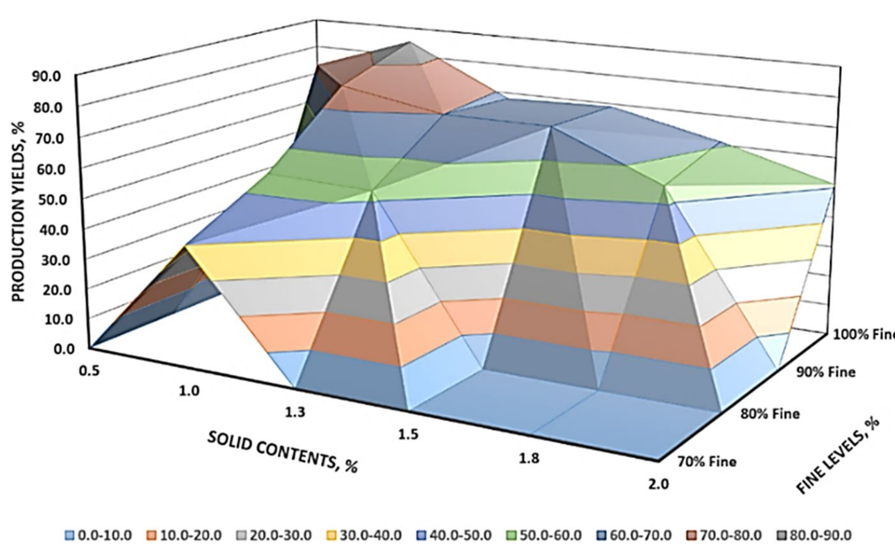

Fig. 6 Production yields of cellulose nanofibrils with different fines levels and solids contents of the suspensions dried by UMaine's pilot spray dryer

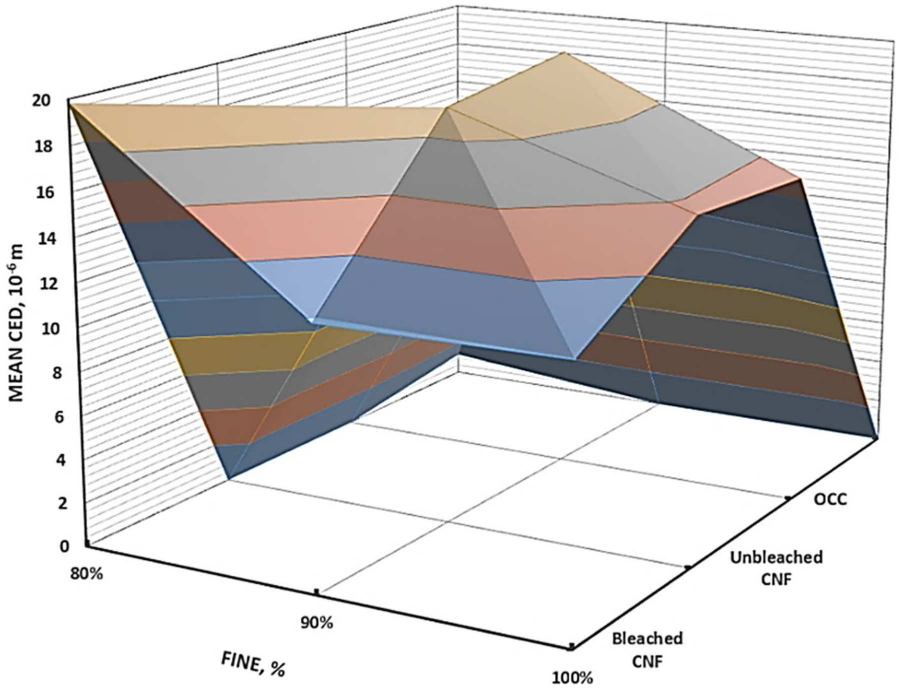

Fig. 8 CED means of CNF made of bleached, unbleached, and OCC pulps

CED (circular equivalent diameter) of spray dried cellulose nanofibril powders.

Figure 8 shows the mean CED of CNF particles produced using three different feedstocks (BSKP, USKP, and OCC). The CEDs given in the chart are the arithmetical mean values of the particle samples, based on surface area [D3,2] of the samples. This is also referred to as Sauter's mean diameter, defined by (Linsinger et al. 2019; Bellino et al. 2001):

$$d_{3.2} = \frac{1}{\sum_i \frac{p_i}{d_i}} = \frac{6}{\text{specificarea}}$$

*d_i: Mean diameter of class *i*. *P_i: Relative volume probability of class *i*.

At the highest fines level, the CED of the bleached SDCNF is smaller than that of the unbleached and OCC SDCNFs. It appears that the further fibrillation (high fine levels) reduces total length and width of pulp fibers, leading to the smaller particle sizes during the spray drying process. The unbleached and OCC pulps contain much thicker fibers about 40% compared to that of bleached pulps even at the highest fines levels attributable to less fibrillation efficiency (Kelly et al. 2021). The fiber bundles should be separated into individual fibers appropriately after refinement but the lignin and hemicellulose in pulps can act as binding agents "cement" between fibers resulting in the reduction of refining efficiency. Furthermore, lignin can lead to rigid and brittle fibers if lignin content is too high in pulps. USKP and OCC pulps had not undergone a bleaching process, thus they contain a relatively higher amount of the lignin than that of BSKP resulting in reduced disk refining efficiency (Yook et al. 2020; Copenhaver et al. 2021). Figures 9 and 10 show the different size distributions of samples from different feedstocks and fines levels. The difference in size distribution is very minor between the fines levels of 100% and 90% in the bleached SDCNFs, while unbleached and OCC SDCNFs showed different size distributions (Fig. 9). It can be noted from Fig. 11, the degree of fibrillation between the fines levels of 100% and 90% does not affect the final sizes of CNF fine powder (SDCNFs) samples via the spray drying process. Even for the bleached SDCNFs, the fines level of 80% showed significant increase in the sizes of the particle distribution curves (Fig. 11). The size distributions of all samples are well matched with the images of scanning electron microscopy (SEM) (Figs. 12 and 13). In the unbleached and OCC SDCNFs, considerable amounts of fibrous material and fiber fragments were

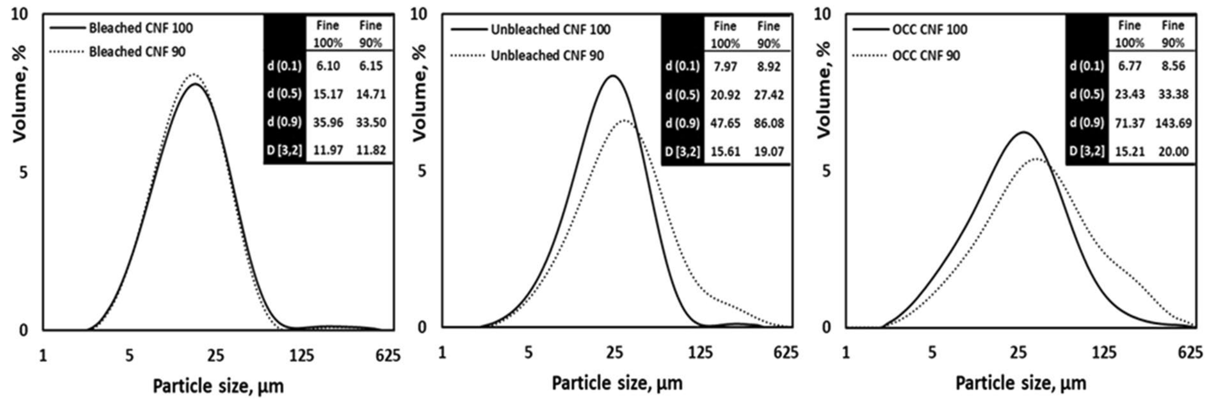

Fig. 9 Particle size distributions of bleached, unbleached, and OCC cellulose nanofibrils with different fine levels (90% and 100%)

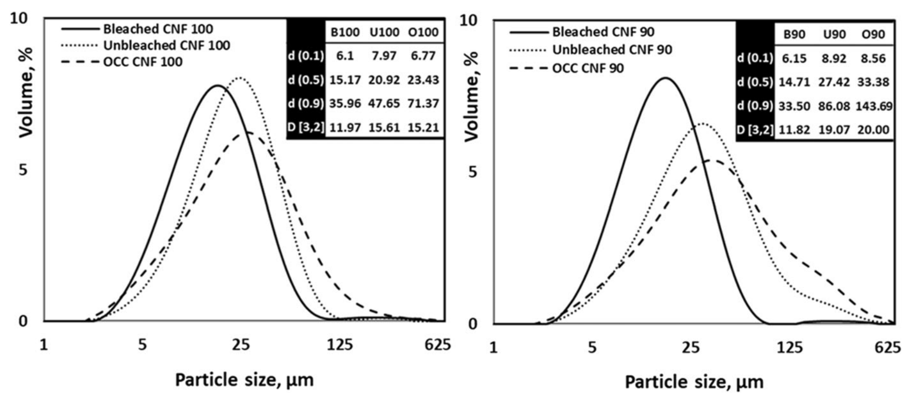

Fig. 10 Comparisons of particle size distributions of different feedstock of cellulose samples

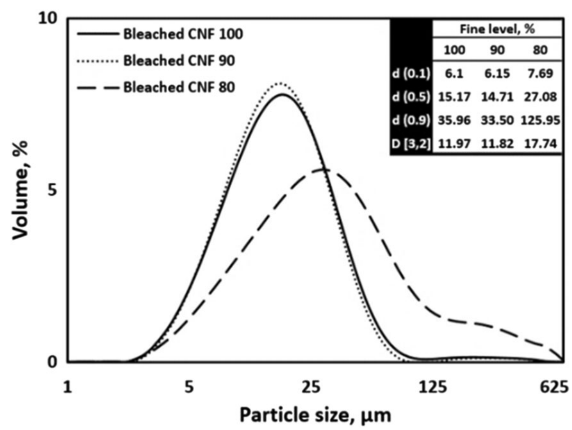

Fig. 11 Comparisons of particle size distributions of bleached cellulose nanofibrils with different fines levels

observed (Fig. 12). While lower fines level SDCNFs showed greater portions of fibers and fragments in general, the bleached SDCNFs looks very uniform in terms of particle sizes and shapes (Fig. 13). In particular, OCC SDCNF appeared to have a wider size distribution than bleached and unbleached SDCNFs, and this is likely attributable to lower density of individual OCC particles, and the small zeta potential of OCC (Copenhaver et al. 2021; Kelly et al. 2021). OCC CNF containing low amounts of hydroxyl groups on larger fibers that induces a weaker hydrogen bonding may become larger individual particles after spray drying, and a low zeta potential value in OCC may not prevent particle flocculation. In contrast, BSKP and USKP fibers, which contain a higher amount of hydroxyl groups, can be tightly aggregated with each other to form high-density particles, and a larger negative zeta potential of the BSKP and USKP particles tend to repel each other (Cellard et al. 2007).

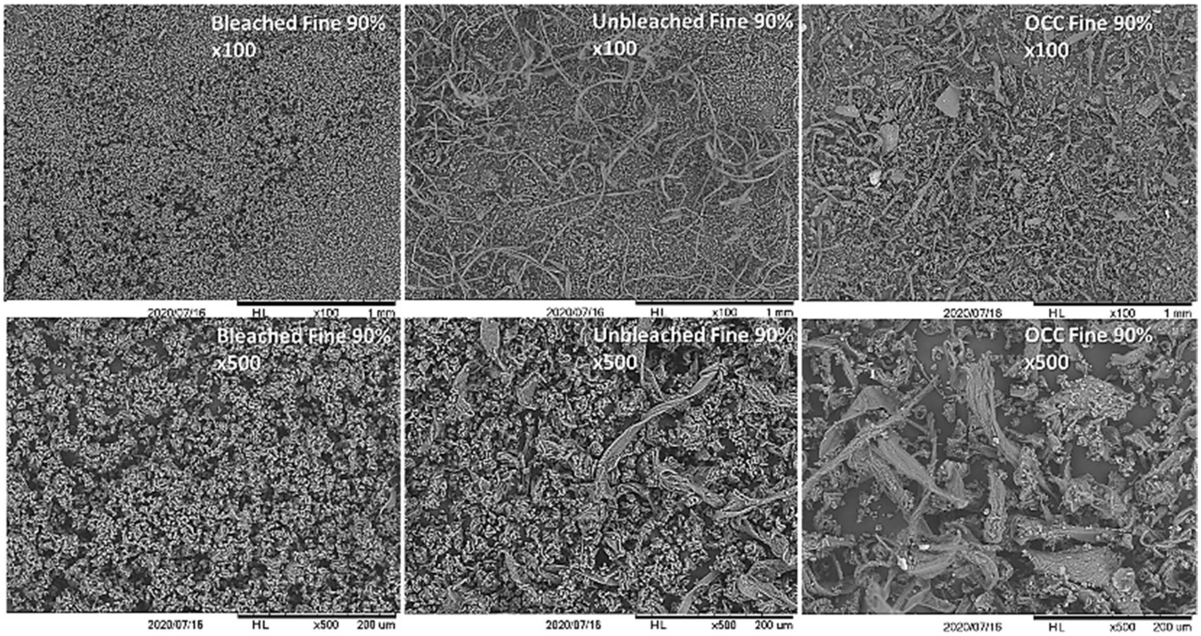

Fig. 12 SEM micrographs of CNF samples from different feedstocks

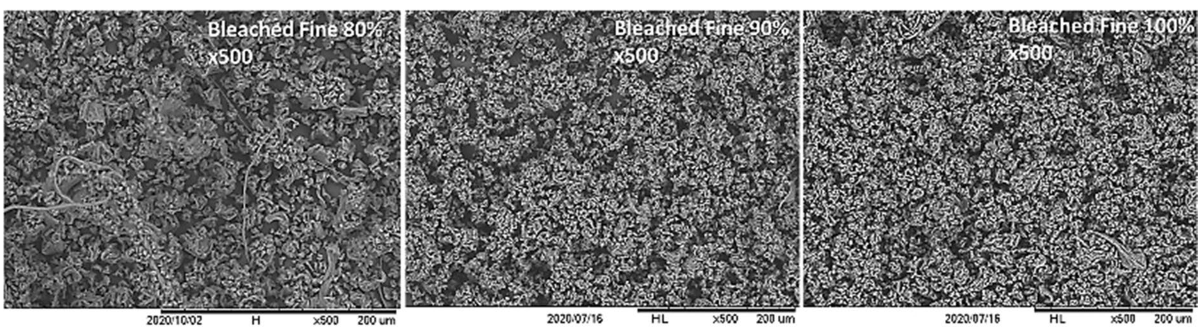

Fig. 13 SEM micrographs of bleached CNF sample from different fine levels

Effect of solids content on size distribution of CNFs

Figure 14 shows the size variation among the bleached CNF samples at different solids contents (1.0—1.8 wt.%) that is the net weight percentage of cellulose fibers in their liquid suspensions. The original solids content of the cellulose suspension directly from the refiner is 3.3 wt.%. The higher the solids content, the larger the mean CED of the CNF fine powder samples. The higher solids content of the CNF suspensions provides more CNF fibers per unit volume of the water droplets created during the spray drying process, resulting in larger agglomerates of CNF fibers (Fig. 15). The CNF solids contents of 1.3, 1.5, and 1.8 wt.% didn't show a significant variation in their powder size distributions. At the same time, the production yield was 70% at 1.3 and 1.5 wt.% solids contents but the production yield dropped to 55% after an increase in solids content to 1.8 wt.%. Therefore, a solids content around 1.5 wt.% is preferable for successful production with better economic benefits, such as lower manufacturing costs. Figure 14 also shows the effect of fines levels on the size distributions of bleached CNF samples.

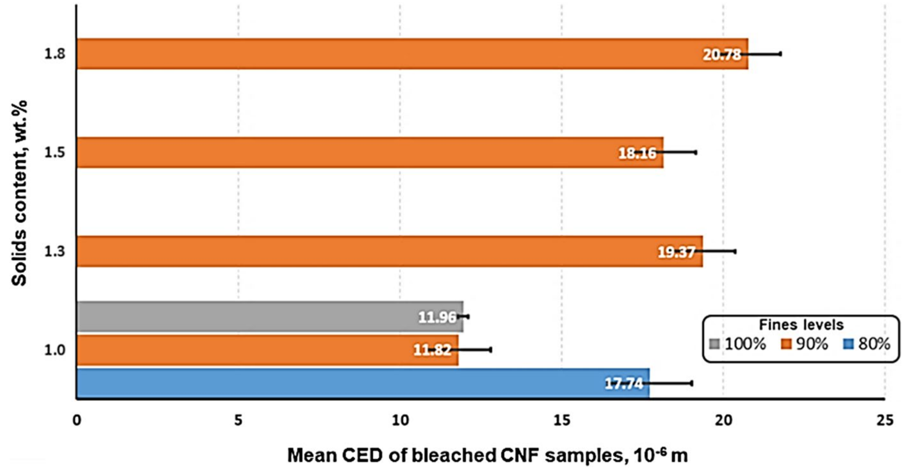

Fig. 14 Mean CED of bleached cellulose nanofibrils with different solids content and fines levels

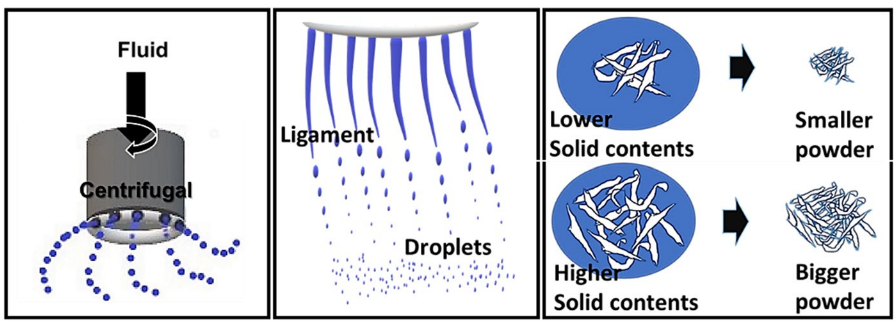

Fig. 15 Schematic of different size particles formed depending on the amount of nanofibrils in a droplet

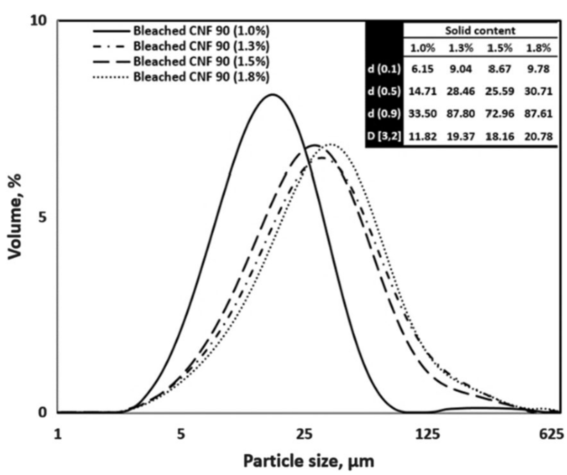

Fig. 16 Comparisons of particle size distributions of bleached cellulose nanofibrils with different solids contents

No difference between 100 and 90% fines level samples was observed, while the fines level of 80% led to a much larger CED. Thus, setting the fibrillation level to 90% fines is also preferable for a successful production with saving some energy during the fibrillation process. Figure 16 shows the size distributions of bleached CNF samples at different suspension solids contents before spray drying. No significant difference was observed among 1.3, 1.5, and even 1.8 wt.%, except the one at 1.0 wt.%. Even though the bleached CNF with the lowest solids content of 1.0 wt.% shows the smallest mean CED value, the mean CEDs of 1.3 and 1.5 wt.% are smaller than other commercial cellulose powder products (e.g., microcrystalline cellulose (MCC)). The SEM images of the bleached CNF samples show similar particle sizes in all samples with different solid contents, except the sample with the 1.8 wt.% solids content (Fig. 17). Formation of larger fibrillar fragments were observed in the SEM images of 1.8 wt.% solids content samples compared to that of lower solids contents. This result

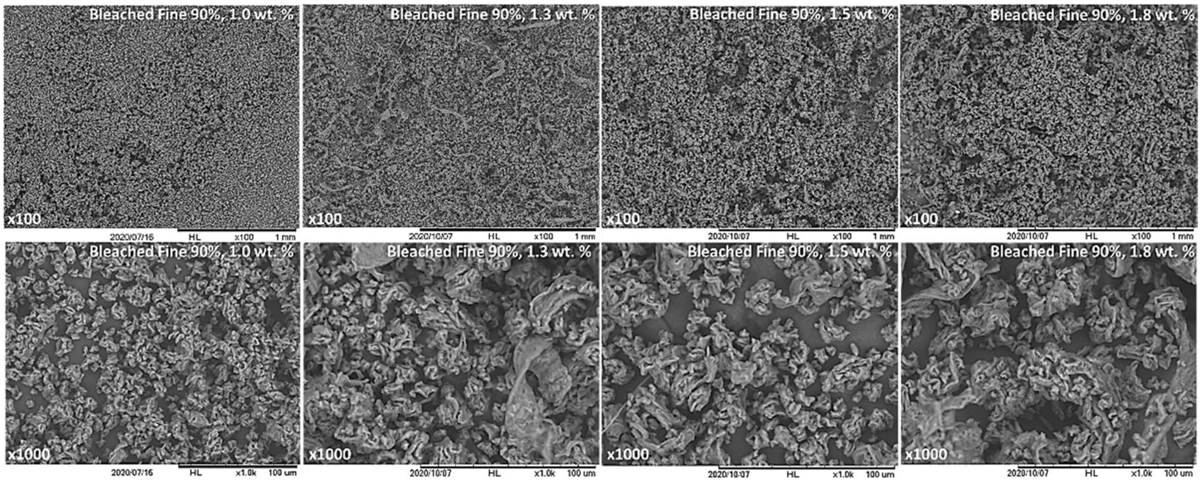

Fig. 17 SEM micrographs of bleached cellulose nanofibrils with different solids contents

may be mainly caused by the relatively high viscosity of 1.8 wt.% solids content (Patel et al. 2009). In the spray drying process, centrifugal forces cause the liquid sheet to break up into ligaments. As atomization continues, these ligaments further disintegrate into droplets. The high viscosity and surface tension of the solution increase the inertia of the ligaments, thereby delaying their disintegration. This delay often results in the formation of larger droplets. As a result, the larger droplets encapsulate a greater amount of CNF fragments (Dafsari et al. 2019).

Conclusions

The conclusions based on the findings of this experimental work, are summarized below;

1. The efficiency of CNF production via spray drying can be improved by increasing the solids content of the CNF suspensions from 1.0 to 1.5 wt.%.
2. The fines level of the CNF suspension affects its production yield but not significantly. Energy savings can be expected from the fibrillation process by targeting the 90% fines level rather than 100%.
3. Spray drying of CNFs produced from different feedstocks resulted in different size distribution of dry CNF powder samples. CNFs produced from bleached feedstock resulted in the smallest particle sizes of dry powder with more uniform size distribution.
4. Fines levels and solids content of the CNF suspensions significantly affected the final size distributions of the dried CNF powders. Higher fines levels resulted in smaller CNF particles, while the higher solids content led to the opposite. The solids contents, ranging from 1.3 to 1.5 wt.%, of CNF suspensions showed excellent potential for better economic viability in terms of manufacturing and achieving smaller CNF particle sizes.

If it is confirmed that the reinforcing effects of these CNF samples are not inferior to the ones with the smallest CED, the higher solids content CNF suspensions can contribute significantly to higher production efficiency, leading to lower manufacturing costs.

This experimental work suggests valuable information regarding the optimization of the spray drying process for manufacturing dried CNF particles.

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Declarations

Competing interests I declare that the authors have no competing interests.

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