

Slot die coating of nanocellulose on paperboard

VINAY KUMAR, DOUGLAS W. BOUSFIELD, AND MARTTI TOIVAKKA

ABSTRACT: Constantly growing interest in nanocellulose usage and applications is pushing the scientific community to search for suitable production and processing techniques. One example is use of nanocellulose as a barrier material in packaging applications, which requires processing of nanocellulose into films and coatings. It is challenging to coat nanocellulose suspensions using conventional pigment coating techniques due to their yield stress behavior and very high viscosity, even at low solids concentration.

This work demonstrates a novel roll-to-roll coating method of nanocellulose on paperboard using slot die geometry. The rheology of nanocellulose suspensions in pressure-driven flow is found to play a critical role in enabling their coating using the said process. In this work, the slot die is used in an unconventional manner where the shearing and metering elements are combined to aid wet film formation and application of nanocellulose suspension onto the paperboard. Barrier performance of the nanocellulose coated paperboards against air, water, heptane vapor, grease, and oils was found to be reasonable and warrants further research on the subject.

Application: The modified slot die coating method reported here could be utilized for roll-to-roll coating of web materials with a large variety of highly viscous materials such as cellulose nanofibers.

Replacement of oil-based materials with those produced from renewable material sources has attracted considerable interest in recent years. An example is the use of bio-based barriers in food packaging [1-3]. One candidate material for renewable barrier applications is nanocellulose, which has been found to possess excellent impermeability, especially against grease, oils, and oxygen. Numerous reviews [4-17], doctoral theses [18-21], and book chapters [22-25] have covered various fundamental aspects of nanocellulose manufacturing, properties, and applications. For packaging applications, large scale production of nanocellulose-based films and coatings is needed. However, most of the research related to potential future applications that has been presented so far is based on small, batch-produced freestanding films [26,27] and coatings [28-38]. Reports on continuous processing into films or coatings, which is required for large-scale and low-cost production, are few [39-41]. One such example is coating of co-ground pigment-microcellulose suspension on a wet paper web in the wet end of a paper machine, as demonstrated recently by Svending et al. [42]. For the successful operation of a coating process, the rheology of the coating material needs to be understood and controlled. Rheology studies based on boundary driven flow of nanocellulose suspensions have been very extensive and are comprehensively covered in a recent review by Nechyporchuk et al. [43] and a book chapter by Naderi and Lindström [25]. However, high deformation rate rheology of nanocellulose, which is relevant for high speed coating operations, has received very little attention [44]. The lack of work on large scale processing of nanocellu-

lose suspensions into coatings and relevance of their high shear rheology in the same calls for more research effort on the subject.

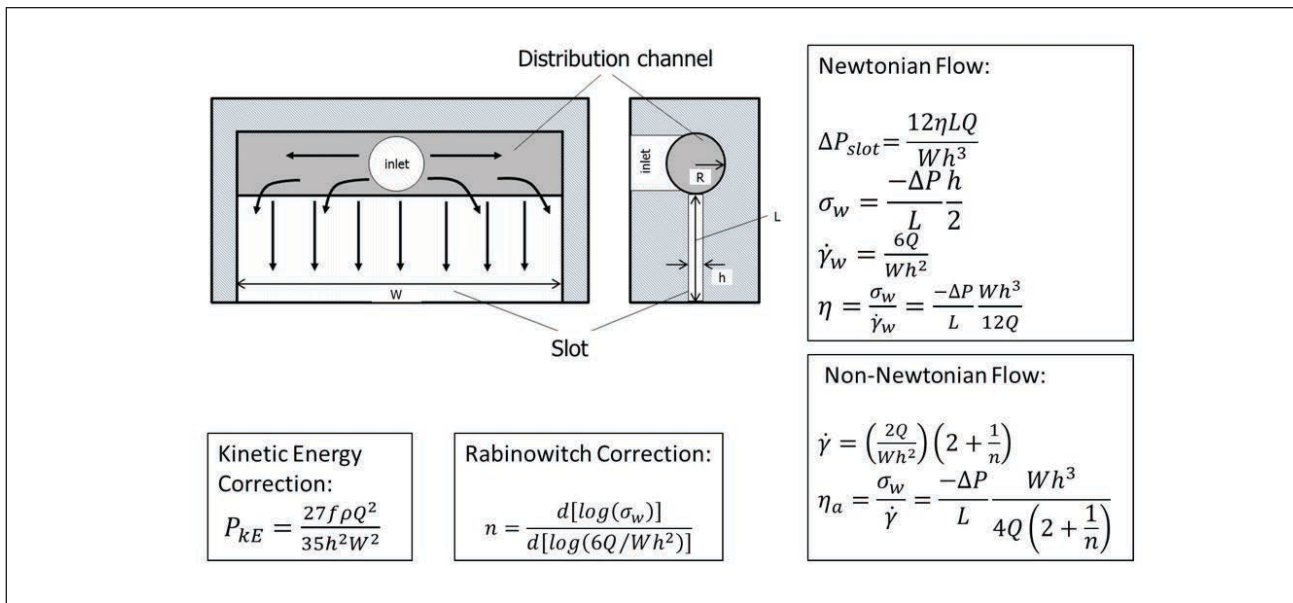
The slot die coating technique is well known in production of, for example, magnetic tapes, pressure sensitive adhesives, photovoltaics, and functional coatings. Slot die has also been demonstrated to be useful for coating of shear-thinning suspensions [45,46]. Fundamentals of the technique can be found, for example, in the book chapter by Durst and Wagner [47]. However, slot die coating has not become a common technique for paper coating, and one potential reason is the swelling of base paper fibers, which can lead to non-uniform coatings for thin coating layers. This is less of a problem when coating with low solids content fluids, as is the case with nanocellulose suspensions. Nanocellulose suspensions have been reported as highly shear thinning in many previous studies [25,44,48,49], and their shear-thinning behavior was utilized to process them into coatings in a roll-to-roll manner in earlier work by the authors [39,40].

The objectives of the current work are to demonstrate the usefulness of slot die geometry for determining high shear rheology of nanocellulose suspensions and enabling their coating on paperboard in a roll-to-roll process. Barrier properties of the nanocellulose-coated paperboard, including oil, grease, water, and heptane vapor barriers, are reported to illustrate potential future applications.

EXPERIMENTAL

The nanocellulose produced by mechanical treatment of cellulose fibers is commonly called micro- or nanofibrillated cellulose (MFC or NFC). Other terms, such as cellulose

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1. Slot die geometry and equations to calculate viscosity.

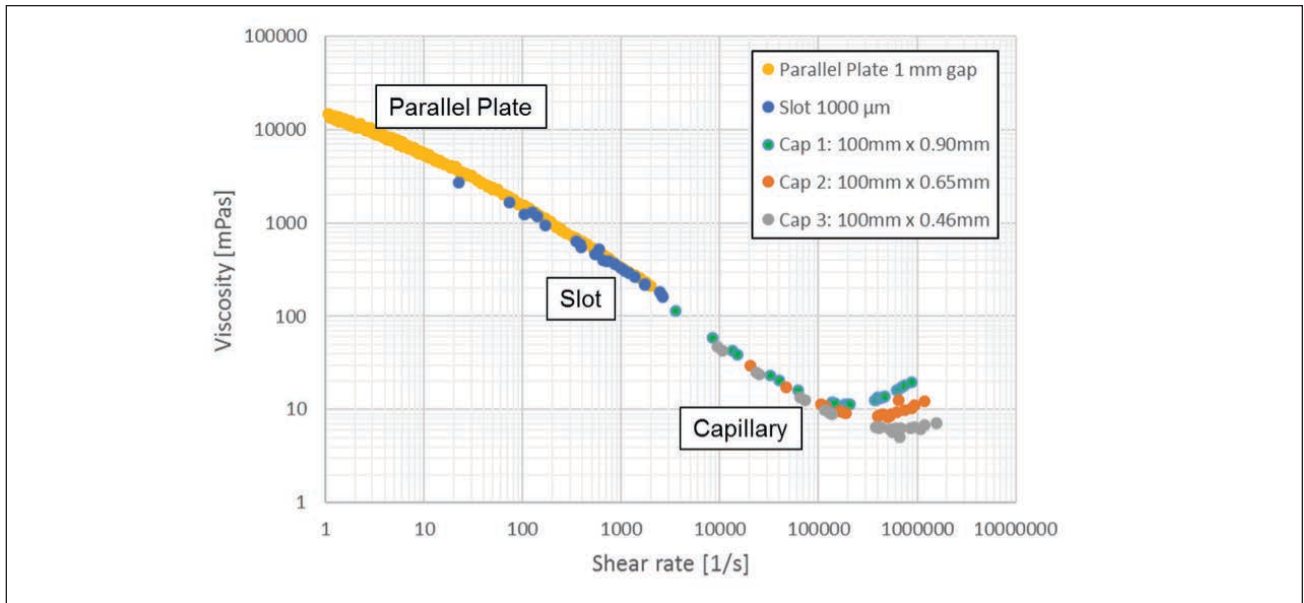
nanofibers (CNF) and micro-nanofibrillated cellulose (MNFC) are also in use. Turbak et al. [50] and Herrick et al. [51] were first to report production of this type of nanomaterial. The mechanical defibrillation process can be combined with chemical [52-54] and/or enzymatic [55] pretreatments to reduce the energy consumption in MFC production, and/or to provide higher fibrillation level with finer material. Nanocellulose, referred to as CNF in this work, was produced at the University of Maine's Process Development Center (Orono, ME, USA) using mechanical treatment with a refiner as described in detail in Kumar et al. [26]. CNF suspension was diluted to 3% solids for rheology determinations and coating with slot die. A JEOL JEM-1400 Plus (JEOL; Tokyo, Japan) transmission electron microscope (TEM) operating at 80 kV acceleration voltage was used to obtain electron micrographs of CNF. The suspension (0.05% w/v) was drop cast on Form-var/carbon-coated 400 MESH copper grids and negatively stained with 1% (w/v) uranyl acetate in water for 40 s before imaging.

Due to experimental problems, viscosity of CNF suspensions has usually been reported only for very low shear rates, typically up to 1000 s⁻¹. Sample ejection from the measurement gap, potentially due to repeated shear, is a common problem when measuring with rotational viscometers. In order to reach higher shear rates, the current work utilizes slot viscometry with a simple, custom-built slot die, as shown in **Fig. 1**. By measuring the inlet pressure and the outflow, the viscosity of the test material can be calculated with well-known equations for narrow gap flow as detailed in Kumar et al. [44]. Corrections to account for fluid acceleration, slot end/exit effects, and non-Newtonian behavior can be applied in a manner similar to capillary viscometry. Correct operation of the slot viscometry device was verified by measuring Newtonian oils and honey water solutions of known viscosities and

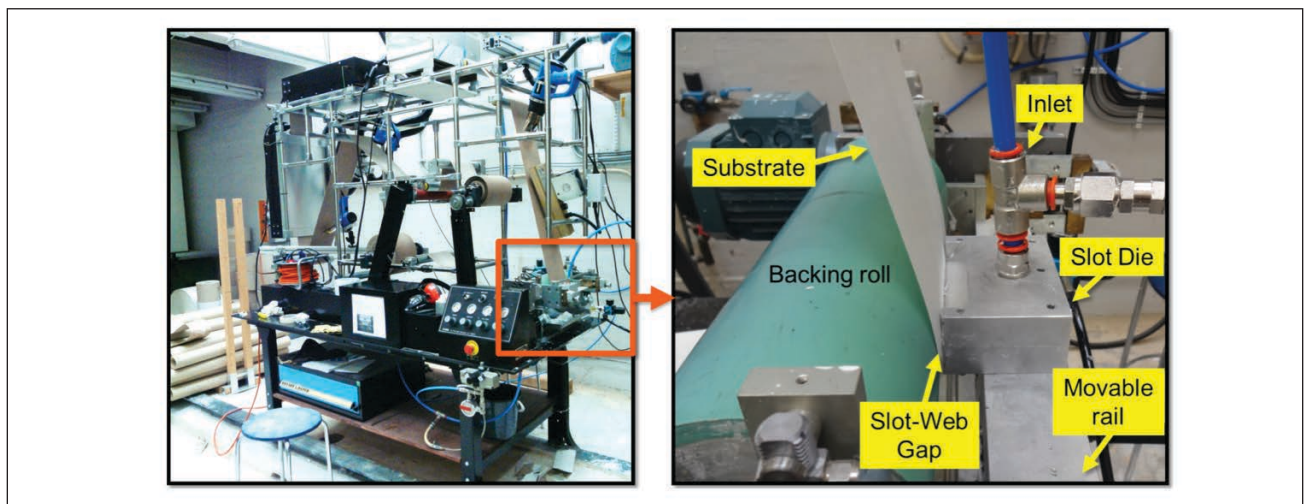
by comparing to flow curves for a non-Newtonian carboxymethyl cellulose (CMC) solution measured with rotational parallel plate and capillary viscometry as shown in **Fig. 2**.

Figure 3 shows the experimental setup for slot die coating of CNF on paperboard used in the current work. The custom-built slot die (slot width = 74 mm, slot length = 34 mm, slot gap = 500–1000 μm, lip lengths = 5 mm) is placed at the three o'clock position on a movable rail against the backing roll. The nanocellulose suspension is fed into the coating head from an air-pressurized vessel. For a chosen slot gap, the coating process can be controlled by changing the web speed (1–50 m/min), feeding pressure (0–10 bar), and the slot-web gap (SWG; 0–2000 μm). Utilizing the slot die geometry, a recycled fiber paperboard (grammage 178 ± 4 g/m² and thickness 190 ± 5 μm) was coated with CNF suspension at 2% solids concentration using 5 pph CMC (FINNIFIX 4000G, CP Kelco; Atlanta, GA, USA) as an additive. All the CNF-coated paperboards were calendered with a laboratory soft nip calender, keeping the back side towards the soft roll, using a line load of 100 kN/m and temperature of 60°C. Samples were conditioned (23°C, 50% relative humidity [RH]) for at least 24 h before testing.

CNF-coated paperboards were characterized for coating coverage and barrier properties. Surface images of the coated paperboards were obtained using a Leo (Zeiss) 1530 Gemini scanning electron microscope (SEM; Carl Zeiss, Oberkochen, Germany) operating in secondary electron mode. A working distance of 11 mm and an acceleration voltage of 10 kV were used to obtain surface images at different magnifications. The specimens were sputtered with a thin layer of gold before imaging. For barrier performance of CNF-coated paperboards, water vapor transmission rate (WVTR) tests were performed according to the ASTM Standard Test Method E96/E96M-05 "Standard test methods for water vapor transmission of mate-



2. Comparison of slot, rotational, and capillary viscometry using a 2% carboxymethyl cellulose (CMC) suspension.

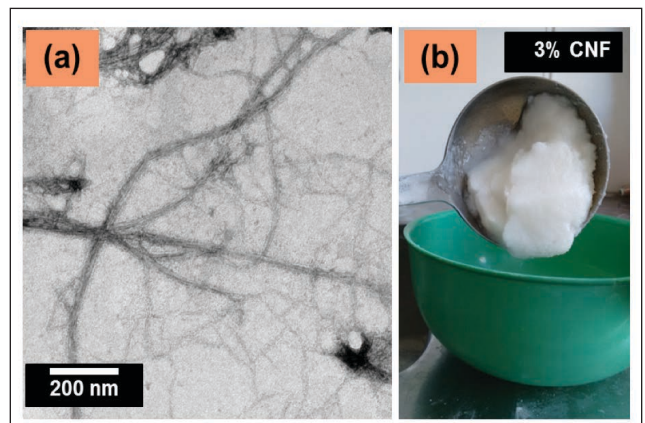


3. Experimental setup for roll-to-roll slot die coating of nanocellulose (CNF) suspension.

rials.” Heptane vapor transmission rate (HVTR) was determined using a similar method and has been described in detail by Kumar et al. [39]. Air permeability tester SE-166 (L&W; Kista, Sweden) with a measurement range of 0.003–100 $\mu\text{m}/(\text{Pa}\cdot\text{s})$ was used to determine the air permeance. Five parallel measurements were performed on different areas of each sample. Grease resistance was determined using the KIT Test following the TAPPI Standard Test Method T 559 “Grease resistance test for paper and paperboard.”

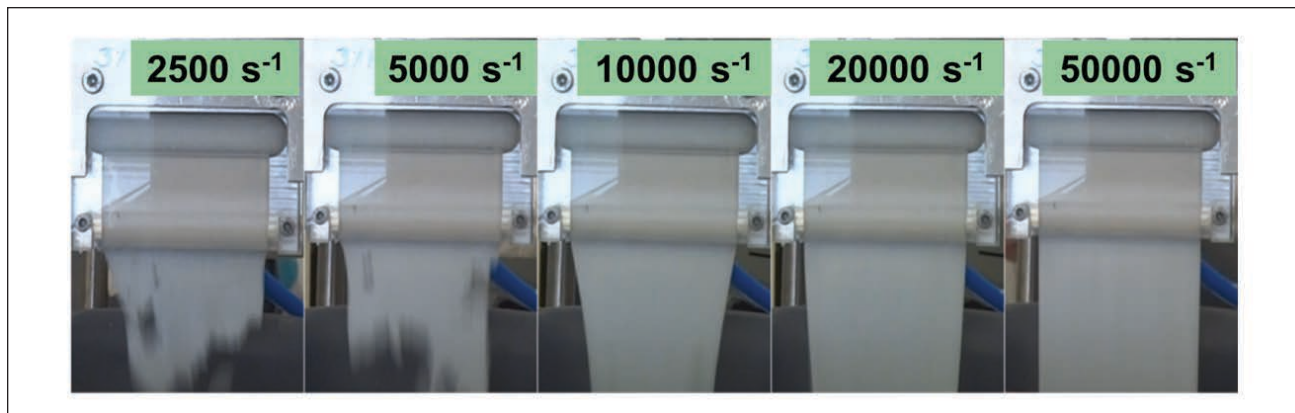
RESULTS

CNF used in the current work was produced without any chemical pretreatment. Therefore, the fibril diameter varies greatly, from less than 100 nm to microns, and the length can reach tens of microns, as shown in the electron micrograph in Fig. 4a. Much smaller sized nanocellulose can be pro-

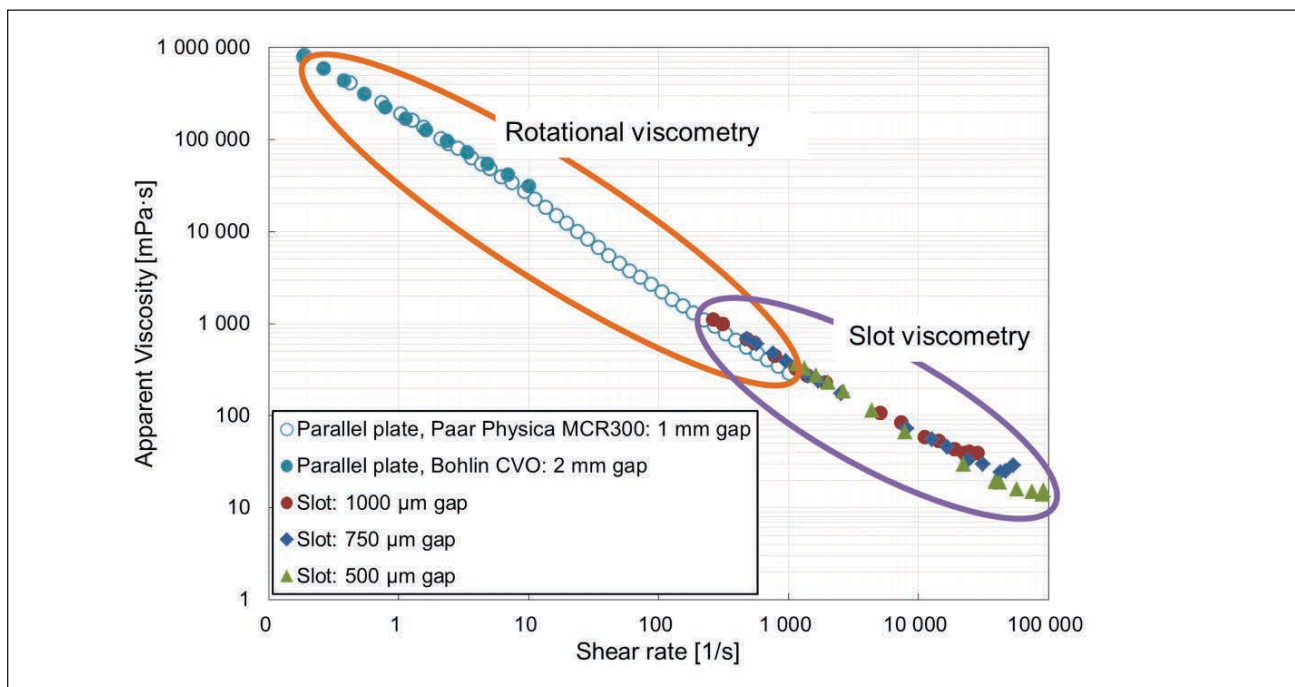


4. (a) Transmission electron micrograph (TEM) of CNF and (b) CNF suspension at 3% solids.

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5. A 3% CNF suspension flowing through the slot die at different shear rates.



6. Apparent viscosity of 3% CNF suspension with parallel plate and slot geometries. Data reproduced from Kumar et al. [44].

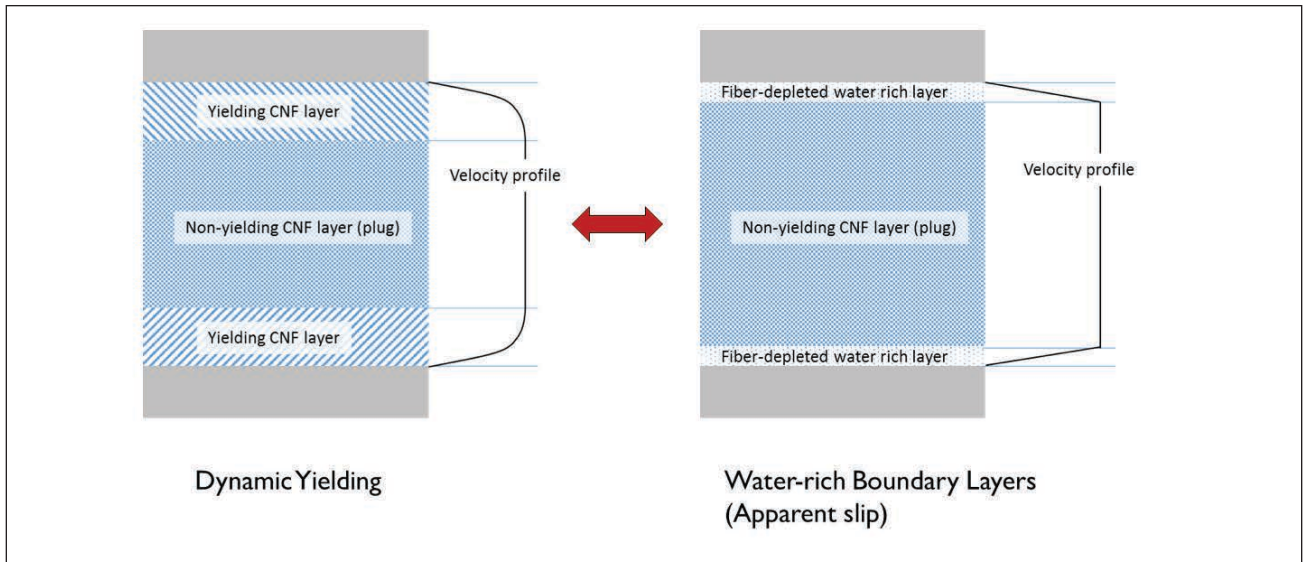
duced by using, for example, TEMPO-mediated oxidation or acid hydrolysis prior to defibrillation. CNF suspensions are typically gel-like, and demonstrate very high viscosities and yield stress already at low solids concentrations, as exemplified in Fig. 4b. The high viscosity, the large amount of water to be dried, and water absorption into paper during coating challenge the CNF coating on paper.

Rheology

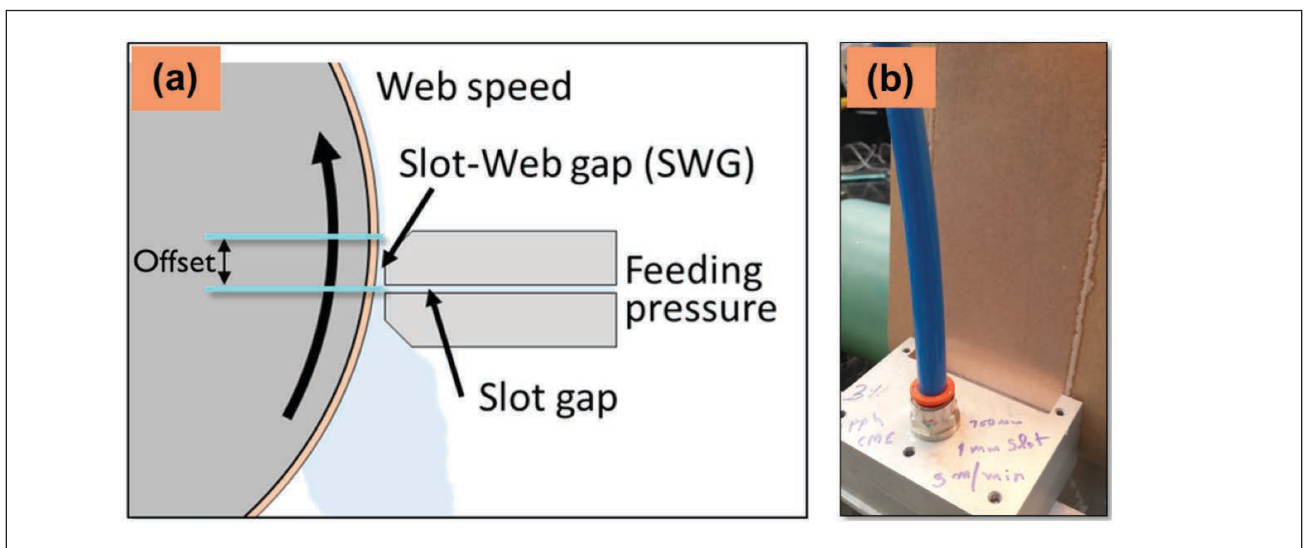
Figure 5 shows the 3% CNF suspension at different flow rates in the slot die. One can observe the highly shear-thinning behavior as the deformation rates increase. At shear rates 50000 s⁻¹ and beyond, the CNF suspension seems to flow without much resistance. With slot geometry, it is possible to reach high deformation rates, as shown in **Fig. 6**, which plots the apparent viscosity of 3% CNF suspension over six decades

of shear rate. The viscosity from the boundary-driven parallel plate devices at low shear rates agrees reasonably with that from the slot geometry at high shear rates. The three different used slot gaps—500 μm, 750 μm, and 1000 μm—give the same result. Noteworthy is the extension of the apparent shear thinning to high shear rates, where the apparent viscosity approaches 10 mPas. The shear thinning can be explained by dynamic yield behavior, where a non-yielding center plug is surrounded by yielded, flowing suspension layers at the boundaries as illustrated to the left in **Fig. 7**.

An alternative explanation for the apparent shear thinning can be provided by apparent slip at the boundaries (Fig. 7, right). Optical coherence tomography measurements for Couette flow of nanocellulose by Haavisto et al. [48] provide experimental evidence for existence of fiber-depleted watery boundary layers. Assuming a non-deforming center plug sur-



7. Alternative mechanisms for apparent shear-thinning behavior in slot flow of CNF.



8. (a) Position of the slot die relative to the backing roll, and (b) slot die in operation.

rounded by water layers, the thickness of the water layer calculated for 3% CNF suspension in the current experiments varies from less than one micron up to 5 microns. While the current work cannot separate the two mechanisms causing the high apparent shear thinning, the low flow resistance does suggest the possibility of using slot die coating for processing of CNF suspensions into coatings and films.

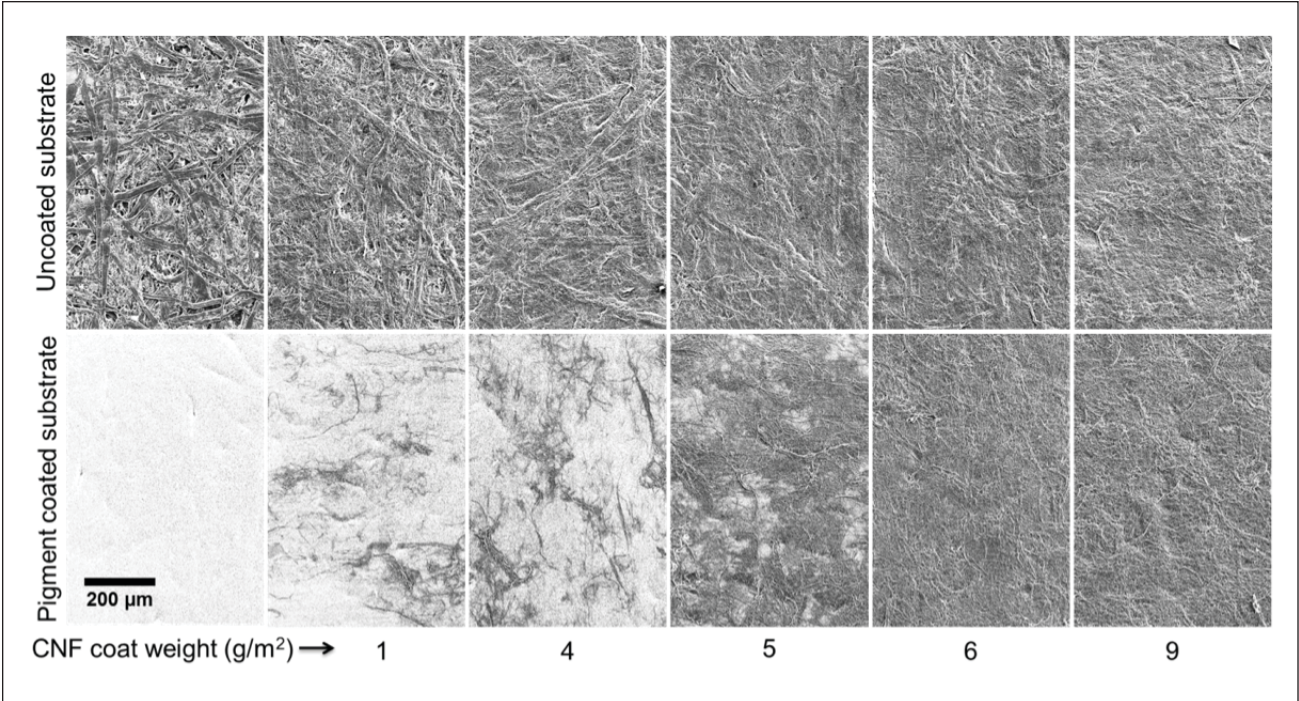
Slot die coating of CNF suspension

Several process parameters, such as slot gap, SWG, feeding pressure, and web speed, affect the slot die coating process. Occasional large aggregates in the CNF material forces one to use relatively large slot gaps to avoid clogging of the slot entrance. At the same time, high flow rates through the slot are needed to produce low apparent (process) viscosity and uni-

form flow. Therefore, to obtain low coat weights at relatively low coater speeds (required by slow drying) the slot die has to be run in an unconventional mode in which part of the coating material is metered off.

For well-controlled metering, it was found to be beneficial to offset the slot die 6 mm below the center line of the backing roll, as shown in **Fig. 8a**. This creates a converging geometry between the substrate and the slot die lips, which enables coat weight control by changing the SWG. The split (i.e., the percentage of CNF coated on paperboard vs. being metered off) is determined by the balance between the pressure-driven extrusion flow forcing the coating both upward and downward, and the shear drag of the moving paper web pulling the coating along upwards. In the converging narrow gap geometry, the pressure-driven flow scales to the third

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9. SEM images of CNF coatings with increasing coat weight on recycled fiber paperboard (top) and pigment coated paperboard (bottom).

power of the gap distance, whereas the shear drag upward is directly proportional to the inverse of the gap distance. Closing the SWG, increasing the feeding pressure, and lowering the web speed all reduce the *relative* proportion of coating transferred to the paper. The final coat weight, which depends on the web speed and the amount of coating that is not metered off, increases with increasing SWG, feeding pressure, and lowering of the web speed. The coat weight was controlled by adjusting the web speed, the CNF suspension feeding pressure, and the SWG to obtain dry CNF coat weights

varying from 1 to 16 g/m². The option to control the coat weight with both the SWG and the feeding pressure was found useful, because the flow profile in the slot-paper gap seemed to influence the visual coating quality of the fibrous nanocellulose. Furthermore, CMC addition was found to improve the CNF dispersion and increase the water retention, leading to overall improvement in the final coating quality as observed earlier by Kumar et al. [39]. The quality of wet CNF film formed on the paperboard can be seen in Fig. 8b, which shows the slot die in operation.

Coat Weight, g/m²	Air Permeance, μm/Pa*s	Water Vapor Transmission Rate, g/m²/day	Heptane Vapor Transmission Rate, g/m²/day	KIT Number**
0	1.72 ± 0.0321	688 ± 43	9031 ± 417	0
1	0.3680 ± 0.0304	502 ± 9	6852 ± 315	0
4	0.0567 ± 0.0129	376 ± 9	858 ± 304	1
5	0.0159 ± 0.0029	323 ± 31	338 ± 249	4
6	0.0030*	216 ± 3	23 ± 16	8
9	0.0030	115 ± 10	0	9
11	0.0030	92 ± 3	0	10
13	0.0030	103 ± 15	0	10
16	0.0030	120 ± 12	0	10

* Detection limit of the instrument.
** Grease resistance measurement.

I. Barrier properties of the nanocellulose (CNF)-coated paperboard.

Coating coverage and barrier properties

Figure 9 shows surface SEM images of the CNF-coated paperboards with increasing coat weight. Because it is difficult to visualize the fibrous CNF coating on top of the uncoated paperboard, pigment-coated board was also used as a substrate to improve the coating coverage visualization. The SEM images suggest that full coverage is reached at coat weights higher than 5 g/m². The incomplete coverage at low coat weights is also apparent in the barrier properties shown in **Table I**. Air permeability values indicate a fully closed surface by CNF at 6 g/m². Both the high surface roughness of the substrate (PPS 7.3 μm) and the coarse CNF quality used in the current work contribute to the insufficient coverage when applying thin coatings. Once the paperboard surface is fully covered, beyond 6 g/m², the barrier properties improve considerably. At 9 g/m², the water vapor barrier reaches a constant value of approximately 100 g/m²/day, a reasonable value considering the hydrophilic nature of the CNF material. At high coat weights, the coating provides a grease barrier with KIT values of 9 or more and no detectable heptane vapor transmission. No vegetable oil penetration was detected within 4 weeks, after which the test was aborted. Similarly, no penetration of a non-polar solvent, dichlorobenzene, was found within the tested 1-week time period. The high solvent barrier suggests potential uses in paper-based electronics such as supercapacitors and batteries. Oxygen barrier was measured, but most samples did not show any oxygen barrier, probably due to pin holes in the relatively large tested areas.

CONCLUSIONS

The current work measured high shear rate viscosity of CNF suspensions utilizing slot die geometry. The results agree with those obtained with parallel plate devices and demonstrate that, in pressure-driven flow, the highly shear-thinning behavior observed with rotational instruments at low shear rates extends to high shear rates. The high apparent shear-thinning behavior can be explained by dynamic yielding or fiber-depleted water-rich boundary layers (apparent wall slip) controlling the flow. The highly shear-thinning process viscosity of CNF suspensions in slot flow enables roll-to-roll coating of these viscous, high-yield stress, gel-like suspensions on paper. Offset slot die placement enables easy metering with slot-to-paper gap control. Provided there is a full coverage of the paper by the CNF material, the barrier properties of paperboard improved considerably. The insufficient coverage issue at low coat weights could potentially be addressed by using a substrate with lower surface roughness and nanocellulosic material with smaller fiber size. Attention needs to be paid to possible pinholes in the coating. Potential solutions include use of multilayer coatings and finer grade nanocellulose. Future work should address moisture sensitivity of the CNF. The current results were obtained at 50% RH, and it is well known that at high humidity the swelling of CNF deteriorates the barrier properties.

Although this work demonstrates a successful use of slot die for coating of nanocellulose suspensions, there are still process challenges that need addressing. For example, higher concentrations of nanocellulose suspensions may lead to air entrainment and difficulties in their pumping to the slot die. Also, an even distribution of these highly viscous and high-yield stress suspensions in wide slot dies at industrial scale may become challenging. **TJ**

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ABOUT THE AUTHORS

We chose to study this topic because continuous roll-to-roll coating on paperboard can potentially enable commercialization of nanocellulosic materials. For large-scale and, therefore, low-cost production, a roll-to-roll coating is needed. However, previous work by others has been limited to small, batch processes.

The most difficult aspect of this study was finding a suitable coating technique, as well as the practical operation of it. The most interesting finding was the very low apparent viscosity of nanocellulose at high deformation rates in pressure-driven flow. Through this research, we gained a better understanding of flow properties for high-yield stress, gel-like materials, such as nanocellulose.

Mills might benefit from the information in this study by gaining a better understanding of the potential to use nanocellulose as a coated barrier. Our next step is to use other types of nanocellulose and to find end-use demonstrators.



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