

## Developing a continuous CNF spinning setup

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### Abstract.

### Introduction

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The environmental issues regarding plastics such as carbon emissions and microplastics ending up in the ocean have pushed material research towards bio-based options (Klar et al., 2018). Also the consumers, industry and governments are increasingly demanding for products that are made from renewable resources with low environmental impact (Moon et al., 2011).

A promising substitutive material is nanocellulose obtained from lignocellulosic biomass (Lee et al., 2014). Cellulose nanofibrils (CNFs) are lightweight, biodegradable and their stiffness is comparable to kevlar and steel (Lee et al., 2014; Lundahl et al., 2016). Nanocellulose nanoparticles are ideal materials for composite products and their potential applications include flexible displays, polymer fillers, biomedical implants, fibers and textiles, and many others (Moon et al., 2011).

Continuous fibers with a desired length can be formed by various spinning techniques, such as dry-spinning, wet-spinning or dry-jet wet-spinning. The only techniques applied to CNF spinning without added polymers are wet- and dry-spinning. (Lundahl et al., 2016)

Mechanical properties of man-made cellulosic materials depends on the cellulose crystallite alignment, which can be enhanced by drawing during the spinning. However, this can be difficult to implement because of the low aspect ratio of CNF. (Baez, Considine and Rowlands, 2014; Sehaqui et al., 2012; Lundahl et al., 2016)

Four grand challenges in cellulose nanoparticle processing have been identified that, when solved, would greatly increase the potential for expanding cellulose nanoparticle usage in new composite materials. These challenges are: 1) decreasing the internal damage in CNs as a result of the extraction process, 2) narrow the particle size range for a given CN processing methodology, 3) decrease the cost of the extraction process and 4) scaling up production to industrial quantities. (Moon et al., 2011)

In this paper, we focus on the challenge of scaling up production, which is challenging especially if controlled structure and characteristics of the material are demanded (Lundahl et al., 2016) and we present a prototype of a wet spinning line for CNF that would allow the production to be scaled up to industrial quantities.

The process used in this study is conducted in room temperature and salt solution is used in the solidification bath. The aqueous CNF suspension is pumped into the salt solution and a set of two rolls is used to transfer the CNF from solidification bath into the washing bath without breaking. Third roll is used at the end of the washing bath to allow the CNF transported into drying. The rotating speed of both rolls is interrelated to the extrusion speed of the spinneret and drawing speed at both baths is adjustable.

## Methods

In this paper, we present a wet spinning line, that could enable more reliable manufacturing of CNF filaments, also allowing the manufacturing of longer filaments, as the previous setup only enabled the production of XX meters of filament at the time, and required constant supervision and manual operating. The setup was required to have a solidification bath, where the CNF suspension could be extruded and it would coagulate in that bath. The setup also was required to have a washing bath and a way to transfer the fragile CNF filaments from coagulation bath into washing bath without breaking. The setup was required to work with the Nexus 3000 syringe pump and it was also supposed to be modular, meaning that the bath lengths and roller diameters should be changeable and the line should support adding a drying and spooling section after the baths. The speed of the filament at each roller was also required to be controllable through a simple and small user interface, that could be operated when wearing the lab equipment. The build can be divided in physical built and control system for further examination of the prototype.

**Physical built.** The prototype was required to have a maximum dimensions of XX-XX-XX. This was required, because in the future, the anti-solvent in the solidification bath may be switched into acid that would require the setup to be in a fume cupboard. The frame of our prototype was built from aluminium profile and as for the baths, stainless steel Gastronorm sized catering containers were used to eliminate the need for custom part manufacturing in case of later development of our prototype. The prototype has three XX mm diameter rollers in total, which were all knurled to reduce the agglutination of the filaments into the rollers. Attachments for the rollers were custom made, mostly from aluminium because of aluminium's weight, price and easiness to machine. The attachments for the rolls were all made identical and they can be used to wet spin filaments in different roller sizes ranging from XX mm to XX mm. All parts that have a possibility of touching the solidification bath's liquid were made from acid proof stainless steel. Even though that was not necessary with current salt solution, this enables further development of various anti-solvents with this prototype.

“What were the special/limiting features of the described components. What are the specifications for key components (actuators, sensors, etc.)(brands are usually not mentioned)” Add more justification, for ex. no big forces to the roller but accurate speed control is important

**Control.** Nema 17 Stepper motors was used to enable steady speed control of each roller. The motors were controlled using Arduino Mega and A4988/DRV8825 drivers. A RAMPS 1.4 shield was used on top of the Arduino, providing ready made slots for five stepper drivers and six motors. The RAMPS board also supports attachment of Full Graphic Smart Controller including LCD, a beeper and switch, and therefore this was used as the user interface. The arduino is connected to the Nexus 3000 with an RS232 cable, which is used to sync the speed of the spinning device with the volumetric speed from the syringe pump.

**Testing conditions.** For the testing of the wet spinning line, a CNF suspension with a water content of XX% was used and the salt solution in the coagulation bath was XX and pure water was used for the washing bath. (add more info about CNF suspension, maybe how its made). The testing

was conducted with varying CNF extrusion speeds from XX to XX ml/min from a XX mm diameter spinneret, and drawing ratio of 1:XX to 1:XX between extrusion and washing, as well as drawing ratio of 1:XX to 1:XX during the washing. The operation reliability of the machine was evaluated by means of the length of filament without interruptions.

## Results

## Discussion

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$$c^2 = a^2 + b^2. \tag{1}$$

### Literature References

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## References

Baez, C., Considine, J. and Rowlands, R. (2014). Influence of drying restraint on physical and mechanical properties of nanofibrillated cellulose films. *Cellulose*, [online] 21(1), pp.347-356. Available at: <http://web.a.ebscohost.com.libproxy.aalto.fi/ehost/detail/detail?vid=0&sid=eb3dbcdc-2d25-474f-9ff4-b20dc26823f3%40sessionmgr4010&bdata=JnNpdGU9ZWWhvc3QtbGl2ZQ%3d%3d#db=teh&AN=93894121> [Accessed 3 Feb. 2019].

Klar, V., Orelma, H., Rautkoski, H., Kuosmanen, P. and Harlin, A. (2018). Spinning Approach for Cellulose Fiber Yarn Using a Deep Eutectic Solvent and an Inclined Channel. *ACS Omega*, [online] 3(9), pp.10918-10926. Available at: <https://pubs.acs.org/doi/10.1021/acsomega.8b01458> [Accessed 3 Feb. 2019].

Klemm, D., Kramer, F., Moritz, S., Lindström, T., Ankerfors, M., Gray, D. and Dorris, A. (2011). Nanocelluloses: A New Family of Nature-Based Materials. *Angewandte Chemie International Edition*, [online] 50(24), pp.5438-5466. Available at: <https://onlinelibrary-wiley-com.libproxy.aalto.fi/doi/full/10.1002/anie.201001273> [Accessed 2 Feb. 2019].

Lee, H., Hamid, S., Zain, S.(2014). Conversion of Lignocellulosic Biomass to Nanocellulose: Structure and Chemical Process. *The Scientific World Journal*, pp.1-20. Available at: <http://search.proquest.com/docview/1560860676/abstract/967744725B0F4D82PQ/1> [Accessed: 3 February 2019].

Lundahl, M., Klar, V., Wang, L., Ago, M. and Rojas, O. (2016). Spinning of Cellulose Nanofibrils into Filaments: A Review. *Industrial & Engineering Chemistry Research*, [online] 56(1), pp.8-19. Available at: <https://pubs.acs.org/doi/10.1021/acs.iecr.6b04010> [Accessed 2 Feb. 2019].

Moon, R., Martini, A., Nairn, J., Simonsen, J. and Youngblood, J. (2011). Cellulose nanomaterials review: structure, properties and nanocomposites. *Chemical Society Reviews*, [online] 40(7), p.3941. Available at: <https://pubs-rsc-org.libproxy.aalto.fi/en/content/articlepdf/2011/cs/c0cs00108b> [Accessed 2 Feb. 2019].

Sehaqui, H., Ezekiel Mushi, N., Morimune, S., Salajkova, M., Nishino, T. and Berglund, L. (2012). Cellulose Nanofiber Orientation in Nanopaper and Nanocomposites by Cold Drawing. *ACS Applied Materials & Interfaces*, [online] 4(2), pp.1043-1049. Available at: <https://pubs-acsc-org.libproxy.aalto.fi/doi/abs/10.1021/am2016766> [Accessed 3 Feb. 2019].

Tang, H., Butchosa, N. and Zhou, Q. (2015). A Transparent, Hazy, and Strong Macroscopic Ribbon of Oriented Cellulose Nanofibrils Bearing Poly(ethylene glycol). *Advanced Materials*, [online]

27(12), pp.2070-2076. Available at:  
<https://onlinelibrary-wiley-com.libproxy.aalto.fi/doi/full/10.1002/adma.201404565> [Accessed 3 Feb. 2019].

Torres-Rendon, J., Schacher, F., Ifuku, S. and Walther, A. (2014). Mechanical Performance of Macrofibers of Cellulose and Chitin Nanofibrils Aligned by Wet-Stretching: A Critical Comparison. *Biomacromolecules*, [online] 15(7), pp.2709-2717. Available at: <https://pubs-acrs-org.libproxy.aalto.fi/doi/abs/10.1021/bm500566m> [Accessed 3 Feb. 2019].

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[1] J. van der Geer, J.A.J. Hanraads, R.A. Lupton, The art of writing a scientific article, *J. Sci. Commun.* 163 (2000) 51-59.

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[2] W. Strunk Jr., E.B. White, *The Elements of Style*, third ed., Macmillan, New York, 1979.

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[3] G.R. Mettam, L.B. Adams, How to prepare an electronic version of your article, in: B.S. Jones, R.Z. Smith (Eds.), *Introduction to the Electronic Age*, E-Publishing Inc., New York, 1999, pp. 281-304.

[4] R.J. Ong, J.T. Dawley and P.G. Clem: submitted to *Journal of Materials Research* (2003)

[5] P.G. Clem, M. Rodriguez, J.A. Voigt and C.S. Ashley, U.S. Patent 6,231,666. (2001)

[6] Information on <http://www.weld.labs.gov.cn>