Developing a continuous CNF spinning setup

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Abstract.

Introduction

The environmental issues regarding plastics such as carbon emissions and microplastics ending up in the ocean have pushed material research towards bio-based options (Wright et al. 2013) (Allwood et al. 2010). 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, also referred to as cellulose nanoparticles (CNs), obtained from lignocellulosic biomass (Lee et al., 2014, Moon et al., 2011). Nanocelluloses can be categorized in three groups; bacterial nanocellulose (BNC), nanocrystalline cellulose (NCC) and cellulose nanofibrils (CNF), although there exist many synonyms for these groups. Each of these groups of nanocelluloses possess different kind of properties (Klemm et al., 2011).

Cellulose nanofibrils (CNF) are lightweight, biodegradable and their stiffness is comparable to kevlar and steel (Lee et al., 2014; Lundahl et al., 2016). Nanocelluloses 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 of nanocellulose 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 in CNF spinning because of the low aspect ratio of CNF. (Baez, Considine and Rowlands, 2014; Sehaqui et al., 2012; Lundahl et al., 2016)

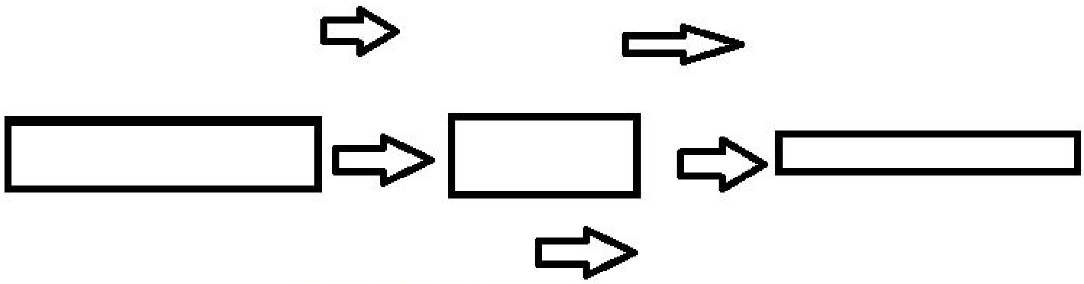
Four grand challenges in nanocellulose processing have been identified that, when solved, would greatly increase the potential for expanding nanocellulose usage in new composite materials. These challenges are: 1) decreasing the internal damage in CNs as a result of the extraction process, 2) narrow 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 of CNF, 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.

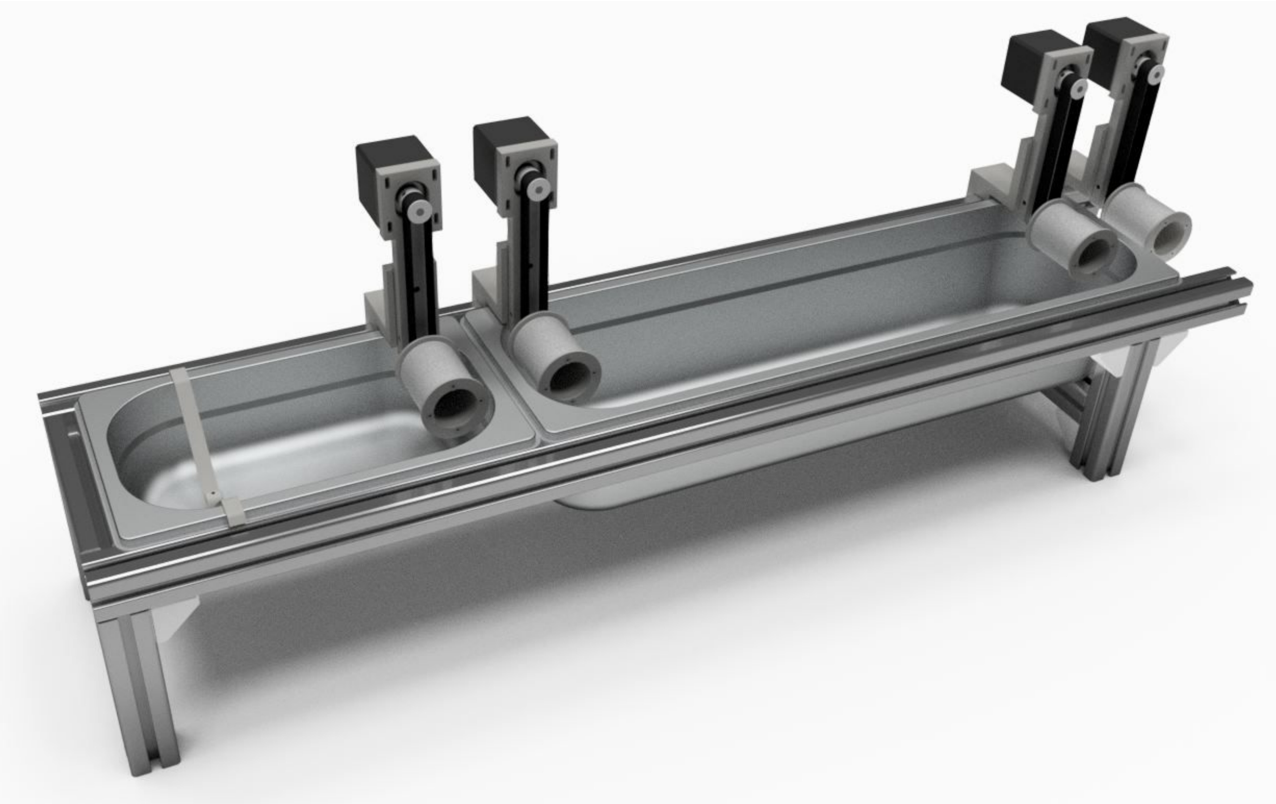


PICTURE OF THE PROCESS

The wet spinning process used in this study is a dual bath -approach where the CNF suspension is pumped to a coagulation bath containing anti-solvent, through a small diameter spinneret. In the coagulation bath, the CNF will slowly solidify and after solidification the CNF filament is transferred to a washing bath where the filament is washed from the salt solution or acid of the coagulation bath. After the washing, the filament is transferred from the washing bath so that the filament can be dried and spooled. Extra bath with acetone can be inserted after washing to increase the drying speed of the filament. Following requirements were set for the new spinning line setup:

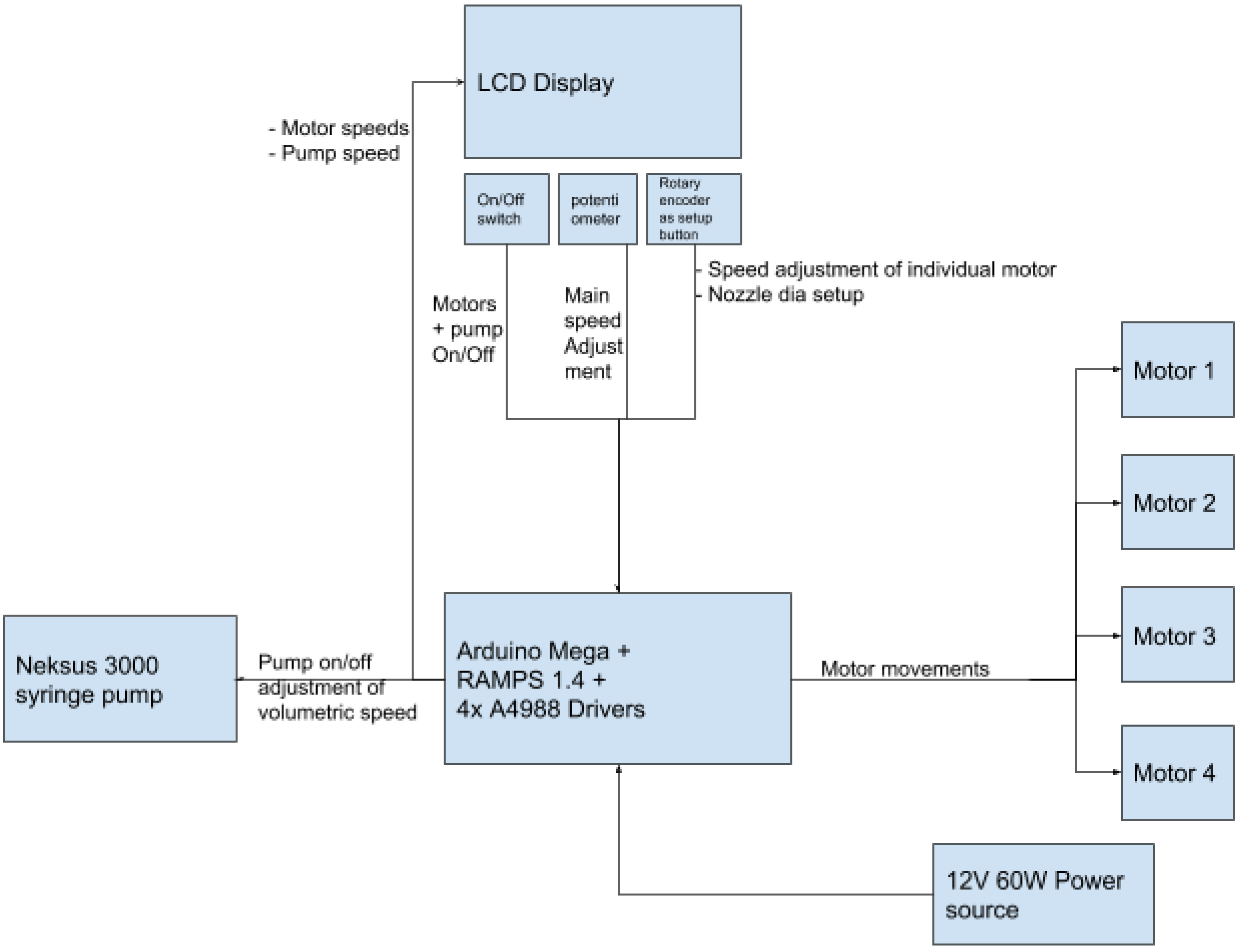
- Include one solidification bath, where the CNF suspension could be extruded and it would coagulate in that bath.
- Include one washing bath and a way to transfer the fragile CNF filaments from coagulation bath into washing bath without breaking.
- Support operation with Nexus 3000 syringe pump.
- Modular structure, 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.
- Fit in the 190x70cm (TARKISTA MITAT) fume cupboard
- Adjustable speed of the filament at each roller
- Simple and small user interface to enable the control of the spinning line. The operation should be possible when wearing lab equipment.

The build can be divided in mechanical structure and control system for further examination of the prototype.



Mechanical structure. 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 or PTFE plastic. Even though that was not necessary with current salt solution, this enables further development of various anti-solvents with this prototype.

Control. 1.5 A nema 17 Stepper motors was used to enable steady speed control of each roller. To achieve more seamless rotation and speed control, each motor was run in half stepping mode. An open-loop controller was built for the motors using Arduino Mega and A4988/DRV8825 drivers. Non-feedback control was used, since the forces and speeds in this setup were estimated to be relatively low, and therefore a reliable operation without any step skipping was achieved by only setting the currents on a sufficient level from the drivers. A RAMPS 1.4 shield was used on top of the Arduino, providing ready made slots for five stepper drivers, of which one slot had ready made pins for two motors and the rest had pins for one motor each. Therefore a total of six motors with five independent speeds could be controlled with this setup. The RAMPS board also supports attachment of Full Graphic Smart Controller including LCD, a beeper and relative rotary encoder, and therefore this was used as the base for the user interface. One on/off switch and a precision potentiometer was added beneath the Full Graphic Smart Controller to enable overall control of the line speed. The arduino is connected to the Nexus 3000 pump with a RS232, 9 pin serial connection, which is used for sending commands to the syringe pump from the arduino to control the volumetric speed and state (pump on pump off) of the syringe pump through the user interface of the spinning line setup. The schematics of the whole control system is visualised in the figure below.



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

How was starting of the test, straightforward, what problems, did the filament brake constantly or not?

How was the control of the setup, convenient, laggy, harsh or something else?

Actual testing results, how long filament samples were achieved with the spinning line setup? with what speeds and drawing ratios?

Discussion

Did it work as planned? why why not?

Is the spinning line produced a feasable setup to continue with. Should the work be continued by adding drying and spooling on the same setup?

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