Dispenser system for nanocellulose 3D printing

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KEYWORDS: 3D printing, nanocellulose, dispenser, syringe

ABSTRACT

A 3D-printed stepper motor dispenser assembly for a 10ml plastic syringe was constructed. This dispenser assembly was used to run a set of calibration experiments to evaluate its suitability to dose nanocellulose mass. The control of the dosing was done with a Labview software along with an Arduino Uno board. A set of dosing trials was conducted with three different dosing speeds and two different dosing volumes to verify the accuracy and repeatability of the constructed system in the nanocellulose mass dosing. The average dosing accuracy of the system was estimated to be at acceptable level for the application.

1 INTRODUCTION

Three dimensional (3D) printing has recently started to replace conventional industrial manufacturing techniques. This applied especially for objects that conventional manufacturing techniques come up with special challenges, e.g. 3D-objects from cellulose [1]. Cellulose nanofibrils (CNF) and cellulose nanocrystals (CNC), generally known as nanocellulose [2], are promising low-cost, strong, porous, lightweight, solution processable, biocompatible, biodegradable materials, which can be used, e.g., as stem cell culturing scaffolds for a growth of bio-implants [3]. The aqueous CNF dispersion appears as a high viscosity gel with is compatible with printing and coating processes, allowing low-cost and high-throughput manufacturing of nanocellulose structures.

Commercially available 3D-printers set strict requirements for printable materials, and are thus limited mostly to specially formulated and designed plastics. There are some tailored laboratory scale approaches for nanocellulose printing, but those printing systems are not commercially available. Thus, there is a need for development of cheap and viable technology for nanocellulose printing.

Recently, CNF have also shown its suitability for fabrication of electronic components, for example piezoelectric sensors [4] and supercapacitors [5, 6, 7]. This work aims at development of scalable technology for 3D-printing of nanocellulose platforms and objects. Aqueous CNF gel is compatible with extrusion printing technique, which can be combined with an in-house 3D-printer after certain modifications are performed. In this paper, a CNF gel dispenser for laboratory scale 3D-printing system was developed, and its accuracy and repeatability were tested.

3 DISPENSER ASSEMBLY

The dispenser assembly described in this work is aimed to be used with a 3d- printing system capable of printing nanocellulose hydrogels. These printed hydrogels are to be processed further to form nanocellulose aerogels with three dimensional structures. These processed aerogels are to be used in studying cell cultivation in aerogel/hydrogel environments.

A 10ml dispenser manufactured by Becton Dickinson S.A. with a plunger diameter of 14.43mm was used in the dispenser arrangement. Along with the dispenser was a M3 threaded rod with a thread pitch of 0.5mm. A stepper motor (24BYJ-48) was used for moving the syringe piston through the threaded roc. The stepper motor was geared to yield 1600 steps per revolution. The stepper was driven in full-step mode. This resulted a theoretical dose of 51.10nl per step. It was decided to target two volumes in the test setup: 10ml and 25ml which correspond to 10mg and 25mg masses respectively with reasonable accuracy as the nanocellulose used in this study contains ~98% water. Theoretical calculations led to requirement of driving the stepper 196 and 490 steps to get the desired volumes.

The practical implementation of the dispenser consisted of four 3D-printed parts: a plunger, a threaded rod motor shaft coupler, a dispenser attachment plate and a motor mount attachment plate. Lulzbot 3D-printer with PLA plastic was used to 3d print the parts. The CAD drawing of the parts is shown in Figure 1a. The actual dispenser assembly is shown in Figure 1b. At the plunger tip a rubber seal that was taken from the original syringe plunger was attached.



Figure 1. a) CAD drawing of dispenser building blocks, and b) photograph of the nanocellulose filled syringe assembled into the dispenser system.

4 CONTROL ELECTRONICS

A simple Labview software was programmed for driving the stepper motor via Arduino Uno microcontroller board. The software utilised two open source libraries in Arduino Uno: Accelstepper and Labview interface For Arduino (LVFA). The LVFA software was not modified in anyway and used in standard configuration in Arduino Uno Board. The LVFA software library contains a AccelStepper library which was used in generating the actual driving signals to the stepper motor driver. The stepper motor was used in unipolar mode and therefore, an ULN2003 based darlington driver was found to be suitable selection for this purpose.

Similarly to Arduino, two freely available software packages in Labview software were used. The communication between Arduino and Labview is based on serial port (RS-232) hardware and a Labview VISA serial interface was used as a basis for the software. Also the software package which realises a rather simple protocol between Arduino and Labview was done with a Labview side of the LVFA library.

Further, a Precisa scale interface was programmed to the Labview software in order to realise automated scale reading. The scale interface utilised also the serial port Visa interface with a simple poll protocol.

5 USER INTERFACE

A Labview user interface presented in Figure 2. was built using Labview standard tools. The user interface enabled to set motor driving parameters such as number of steps, stepping speed, acceleration and number of doses to be run in a single measurement run. Furthermore, the measurement results were logged to a data file for further analysis of the data.

Stepper_dispense	r_scale_file.vi			
File Edit View	Project Operate	Tools Windo	w Help	?
Simple s Log file pai & p:\logfil Scale port & COMB Uno stepp & COM9 Init Initialise Exit	tepper driv	er with scal	e interface Set Speed (Steps per Second) 100 # of Steps to Move 100 Set Acceleration (0 to disable) 10 Stepper # 1 Scale stabilisation 1000 Rounds to be run 5 Rounds run 0	Steps Remaining?
timeout 10000	(10sec)		Scale output	

Figure 2. Labview software user interface.

6 MEASUREMENT SETUP

The stepper motor controlled 10ml (brand) dispenser syringe was filled with 1.96 wt-% aqueous dispersion of CNF (obtained from Aalto University, Finland). CNF fabrication has been described elsewhere [4]. A 1mm diameter syringe needle was attached to the dispenser and the dispenser arrangement was placed onto Precisa XR-205SM-DM digital scale in a such way that neither the dispenser nor the needle was touching the scale. Few doses were pushed from the syringe with the stepper motor arrangement to ensure that there would not be immediate air bubbles in the outcoming CNF mass. The parameters used in the set of experiments are reported in Table 1. The aim for this test setup was to figure out whether the stepping speed or the dosing volume will contribute to the accuracy and repeatability of the nanocellulose dosing volume.

Experiment	1	2	3	4	5	6
Steps	192 (10ml)	192 (10ml)	192 (10ml)	490 (25ml)	490 (25ml)	490 (25ml)
Speed (steps/s)	25	50	100	25	50	100
Speed (ul/s)	1.28	2.56	5.11	1.28	2.56	5.11
Doses	40	40	40	40	40	40

Table 1. The parameters used in dosing experiments.

7 RESULTS

Table 2. presents the summary of experimental results, showing the obtained dose volumes from six different rounds of measurements. The results are characterised in terms of min, max, 25%, 75%, mean and standard deviation of dose volumes. A visual representation of the dose volume measurements is presented in Figure 3.

1							
Experiment	1	2	3	4	5	6	
Min (ul)	5.11	-2.06	0.31	-1.17	18.42	13.10	
1st quartile (ul)	7.66	7.27	8.01	22.39	22.36	21.65	
3rd quartile (ul)	10.61	12.30	13.73	27.08	26.35	26.29	
Max (ul)	14.52	19.69	18.33	38.43	32.21	37.56	
Mean (ul)	9.24	9.74	10.57	24.04	24.62	24.23	
Standard deviation (ul)	2.12	4.39	4.42	5.92	3.14	5.08	

Table 2. Results of of the test rounds in quartiles and mean values.



Figure 3. Accumulated dose of CNF dispersion using volume of a) 10ul and b) 25ul. Boxplot presentation of c) accumulated dose and d) dosing error.

8 ANALYSIS AND CONCLUSIONS

After the experiments, it can be concluded that the linearity of the dosing itself is on acceptable level for 3Dprinting of CNF dispersion. The accuracy of selected dispensed dose volumes (10 and 25 μ l) appeared to be very close to theoretical predictions. Achieved average dose volumes were 9.24, 9.74 and 10.57 μ l in the in former case, and 24.04, 24.62 and 24.23 μ l in the latter case, when dosing rates of 1.28, 2.56 and 5.11 μ l/s were used, respectively. These represent dosing errors of 5.7 and 3.9% for 10 and 25 μ l doses, respectively. The measurement error contributed by the used volume measurement method itself was estimated to be negligible when compared to these values. The standard deviation of the dose volumes were from 2.12 to 4.42 μ l for 10 μ l dose, and from 3.14 to 5.92 μ l for 25 μ l dose (see Table 2). The values represent about ±40% dosing error for 10 μ l doses, and about ±20% for 25 ul doses, where $\frac{2}{3}$ of the observations falled into this range.

The step motor stepping speed and the dose size does not seem to affect the overall linearity of the dispenser. When considering the individual dose volumes, the error in the dose size does not seem to correlate with the stepping speed and individual dose size. However, there are significant variations between individual dose volumes. One possible contributor to this is the residual air bubbles in CNF gel in the syringe, when observed by eye. The volume of the air bubbles was not characterised in this study. To conclude, the average dosing accuracy of the developed dispenser system was on acceptable level for 3Dprinting of CNF dispersion, as the dispenser is designed for constant speed extrusion. The average dose volume accuracy is the driving parameter in this study, and it was found to be consistent. After the upcoming CNF printing trials using developed dispenser with the 3D-printer system, further experiments may be required for fine tuning the dosing properties.

9 REFERENCES

- Håkansson, K. M., Henriksson, I. C., de la Peña Vázquez, C., Kuzmenko, V., Markstedt, K., Enoksson, P., & Gatenholm, P. Solidification of 3D Printed Nanofibril Hydrogels into Functional 3D Cellulose Structures. Advanced Materials Technologies, 1.7 (2006).
- [2] Moon, R. J., Martini, A., Nairn, J., Simonsen, J., & Youngblood, J. Cellulose nanomaterials review: structure, properties and nanocomposites. *Chemical Society Reviews* 40(7) (2011) 3941.
- [3] Kalia, S., Dufresne, A., Cherian, B. M., Kaith, B. S., Avérous, L., Njuguna, J., & Nassiopoulos, E. Cellulose-based bio-and nanocomposites: a review. International Journal of Polymer Science, 2011 (2011).
- [4] S. Rajala, T. Siponkoski, E. Sarlin, M. Mettänen, M. Vuoriluoto, A. Pammo, J. Juuti, O. J. Rojas, S. Franssila and S. Tuukkanen, Cellulose Nanofibril Film as a Piezoelectric Sensor Material, ACS Appl. Mater. Interfaces 8(24) 15607 (2016).
- [5] K. Torvinen, S. Lehtimäki, J. T. Keränen, J. Sievänen, J. Vartiainen, E. Hellén, D. Lupo, S. Tuukkanen. Pigment-Cellulose Nanofibril Composite and Its Application as A Separator-Substrate in Printed Supercapacitors. *Electronic Materials Letters* 11(6) (2015) 1040. doi: 10.1007/s13391-015-5195-6.
- [6] S. Tuukkanen, S. Lehtimäki, F. Jahangir, A.-P. Eskelinen, D. Lupo, and S. Franssila. Printable and disposable supercapacitor from nanocellulose and carbon nanotubes 5th Electronics System-Integration Technology Conference (ESTC 2014), Helsinki, Finland. doi:10.1109/ESTC.2014.6962740.
- [7] J. Virtanen, A. Pammo, J. Keskinen, E. Sarlin, and S. Tuukkanen. Pyrolysed cellulose nanofibrils and dandelion pappus in supercapacitor application. (submitted for publication).