

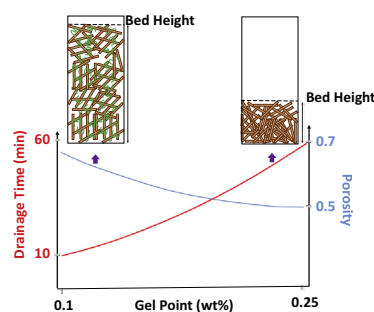
## Effect of cationic polyacrylamide on the processing and properties of nanocellulose films



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### GRAPHICAL ABSTRACT



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### ABSTRACT

The use of high molecular weight cationic polyacrylamide (CPAM) was investigated to accelerate the drainage of nanocellulose (Microfibrillated Cellulose) suspensions into films. The mechanism was quantified and optimized by measuring the gel point, the lowest solids concentration at which a continuous network is formed. The flocculation of MFC was analysed as a function of the polyelectrolyte dosage, charge density and molecular weight as well as process parameters (drainage time) and material properties. The adsorption isotherms of CPAMs on nanocellulose and their zeta potential curves were also analysed as a function of CPAM charge and dosage. Measured CPAM adsorption capacities for the 50% and 10% charged 13 MDa CPAM onto MFC were 5 mg/g and 8 mg/g, respectively, corresponding to adsorption coverage on cellulose of 0.14 mg/m<sup>2</sup> and 0.22 mg/m<sup>2</sup>. The floc strength and drainability of MFC suspensions were quantified with the gel point as a function of CPAM properties. For all combinations of polyelectrolyte molecular weight and charge density, the gel point of a nanocellulose suspension goes through a minimum with increasing polymer dosage. The minimum gel point was independent of the polyelectrolyte charge density at constant molecular weight. However, it reduced with decreasing CPAM molecular weight, at a constant addition rate. The drainage time of a nanocellulose suspension into a film is reduced by 2/3 by halving the gel point from 0.2 to 0.1 kg/m<sup>3</sup>; this is due to the more flocculated suspension facilitating drainage between flocs. Nanocellulose films of increased porosity also result from reducing the gel point, signifying that the more open 3D structure of the flocculated cellulose suspension is retained upon drying the 2D film cellulose film structure.

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### 1. Introduction

Nanocellulose films made from micro fibrillated cellulose, cellulose nanofibres or nanocellulose crystals are being developed for

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many new applications including diagnostics, transparent conductive films for electronics [1–3], smart clothing [1], optically transparent electronic displays [4] and membrane applications which comprise fuel cells [5], tissue engineering [6] and liquid purification and filtration [7,8]. Nanocellulose composites are excellent materials for optically transparent flexible electronic displays due to their low coefficient of thermal expansion [4] and their optical properties which prevent light scattering as the fibre diameter is less than one-tenth of the visible light wavelength. Cellulose nanopapers have high mechanical strength, low density, controlled permeability and high surface area which are important for the proposed membrane applications [9].

A central issue with non-woven materials made from nanofibre suspensions is the impractically long preparation time which ranges from 45 min [10] to 4 h [11]. Recently, drainage time has been reduced to 3 min for nanofibre suspensions alone by using a high solids content in sheet forming [12]. The drainage time was further reduced by controlling the nanocellulose flocculation using polyelectrolytes [13]; however, the colloidal mechanisms behind this improvement were not explored. Cellulose fibre films are made by draining a fibre suspension over a moving wire, producing a continuous flexible material from discontinuous fibres. This process is a robust, flexible and efficient process which is widely available all over the world to produce high performance materials at very low cost. Polyelectrolytes are often used as retention aids [14–16] and to control paper structure: pore size, colloidal structure and fibre distribution [17–20].

The adsorption of cationic polyelectrolytes onto cellulose fibres and their flocculation to increase process efficiency and paper quality are well understood [21–26]. Much less is known on the effect polyelectrolytes plays on the physics of the nanocellulose colloids, their flocculation and their effect on materials and process engineering. Furthermore, by changing the charge density and molecular weight of the polyelectrolyte, it is reasonable to assume that the nanocellulose flocculation mechanism can range from pure bridging to charge reversal. But does it alter the nanocellulose processing and material properties and can it be optimized and engineered?

In this study, we analysed the flocculation of cellulose nanofibres using a series of cationic polyacrylamides varying in charge density and molecular weight. Their adsorption isotherms and their zeta potential curves were analysed as a function of CPAM dosage and charge. We relied on the gel point as a methodology to quantify and engineer the floc properties. The gel point is the solids concentration at which all the primary flocs form a self-supporting network [27,28]. It describes the compressibility and structure of sediments [27]. Micro fibrillated cellulose (MFC) is selected as model fibres for its convenience and well-understood properties. MFC is a heterogeneous material with dimension distribution that is a function of the processing conditions and starting material. The terms MFC and cellulose nanofibres are used interchangeably in this paper. It is the objective of this study to engineer the process and properties of nanocellulose films by controlling the structure of flocs using linear high molecular weight Cationic polyacrylamide (CPAM).

## 2. Experimental method

### 2.1. Materials

Micro fibrillated cellulose (MFC) purchased from DAICEL Chemical Industries Limited (grade Celish KY-100G) with 75% moisture content was used. The cationic polyacrylamide (CPAM) polymers were kindly supplied by AQUA + TECH Switzerland from their SnowFlake Cationics product range, and used as received.

**Table I**

Cationic polyacrylamide properties [25] as provided by the manufacturer. Charge density was measured by titration as described.

CPAM	Charge density (wt%)	Molecular weight (MDa)	Measured charge density (eq/g)
50C13M	50	13	$1.30 \times 10^{-3}$
40C13M	40	13	$1.00 \times 10^{-3}$
30C13M	30	13	$0.77 \times 10^{-3}$
20C13M	20	13	$0.68 \times 10^{-3}$
10C13M	10	13	$0.30 \times 10^{-3}$
40C15M	40	15	–
40C8M	40	8	–
40C6M	40	6	–
80C4M	80	4	$2.2 \times 10^{-3}$

These are copolymers of uncharged acrylamide with cationic dimethylaminoethylacrylate methyl chloride with charge densities and molecular weights shown in Table I. Specific charge density for each CPAM was measured by titration with oppositely charged polyelectrolyte (PES-Na) of known concentration (0.001 eq/g) (Table I) [25,29]. The granulometry (particle size distribution) of all the CPAMs ranged from 0.125 mm to 1.120 mm, as specified by the supplier. Ultrapure water purified with a Millipore system (18 M $\Omega$  cm) was used in all aqueous solutions and rinsing procedures.

### 2.2. Preparation of MFC and CPAM suspension

A 3L Mavis Engineering (Model No. 8522) disintegrator was used to disperse fibres in water evenly using 15,000 revolutions. A fresh 0.5 mg/mL of CPAM solution was prepared at least 8 h prior to the experiment.

### 2.3. Surface area determination

Mercury intrusion measurements for MFC were made using an Autopore IV 9500 (Micromeritics Instrument Corporation). The maximum applied pressure of mercury is 60 kPsia. The equilibration time at each of the increasing applied pressures of mercury was set to 10s.

### 2.4. Particle and colloid charge

The zeta potential measurements were performed with a Nano-brook Omni (Brookhaven Instruments) in a cuvette cell at 25 °C. The zeta potential was calculated with the supplied software by determining the electrophoretic mobility from an electrophoresis experiment using laser Doppler velocimetry and applying the Smoluchowski equation. CPAM of 0.5 mg/mL concentration was added at different dosages to 0.5 wt% MFC suspension, and then mixed using a hand stirrer for 2 min. MFC–CPAM suspension was centrifuged at 4400 rpm for 20 min to remove big aggregates and retains colloids. The supernatant containing colloidal nanocellulose was then used to measure the zeta potential.

### 2.5. Polymeric flocculant adsorption

Following the method employed by Peng and Garnier [30], the amount of polymer adsorbed onto MFC was determined by subtracting the concentration of CPAM in the supernatant after centrifugation from that of the dosed polymer. The quantification of CPAM was made through polyelectrolyte titration, which used the streaming potential of the particle charge detector to identify the point of zero charge. The streaming potential measurements were performed with the particle charge detector (Mutek PCD-03, BTG Instruments). An automatic titrator continuously added

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