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Formation of cellulose nanocrystal membranes by controlled deposition

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Introduction. Controlling membrane performance by tuning simple fabrication parameters is highly desirable. We used cellulose nanocrystals (CNCs) to form the selective layer of composite membranes via tangential flow filtration of CNC suspensions over a porous support layer. Shear rate, which affects the alignment of the nanorod-shaped CNCs in the flow direction and the suspension's ionic strength and pH were used as tuning parameters to vary the effective pore size of the CNC membranes.

Experimental/methodology. CNCs were formed by acid hydrolysis of cotton linter [1]. After depositing CNCs on a porous support layer made of cellulose acetate, physical stabilization was done by permeating a 0.2 M AlCl₃ solution through the layer. Alternatively, chemical crosslinking of TEMPO-oxidized CNCs was done via Ag(I)-catalyzed oxidative decarboxylation [2,3]. Polarized optical microscope images were obtained between crossed polarizers to demonstrate birefringence of CNC layers due to nematic or cholesteric alignment.

Results and discussion. CNCs align in the flow direction beyond a certain shear rate, as verified by polarized optical microscopy (Fig.1a). Increasing shear rate to align the nanorods increases the rejection of the membranes (Fig.1b).

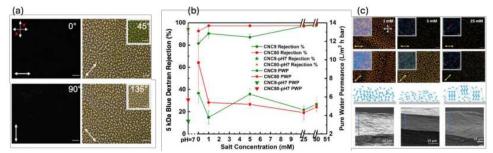


Figure 1. (a) The membrane with aligned CNC layers under crossed polarizers. Flow direction at 0, 45, 90 and 135 degrees to the polarizer. (b) The rejection and permeance of CNC membranes prepared under high shear (CNC80) and low shear (CNC9) conditions. (c) CNC membranes prepared using suspensions containing 1, 5 and 25 mM NaCl between crossed polarizers (top), schematic explanation of the morphology (middle) and SEM images of cross-sections With increasing salt concentration in the CNC suspension, rejection increases both at low and high shear rate,

accompanied by a drop in permeance, due to screening of electrostatic repulsion between nanorods allowing their tighter packing (Fig.1b) [4]. Polarized optical microscopy indicates presence of tactoids, or aggregates with cholesteric order, of varying size as a function of shear rate and salt concentration (Fig.1c).

CNCs obtained by sulfuric hydrolysis have sulfate half esters on their surface. TEMPO oxidation of these CNCs adds carboxyl groups onto the surface which can then be crosslinked in a solution of AgNO₃ and potassium persulfate [3]. While both physical and chemical crosslinking of TEMPO-CNCs yield membranes with good stability, chemical crosslinking improves drying of the membranes which is important for module preparation and scale up (Fig.2) [2].

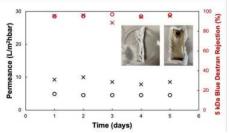


Figure 2. Permeance and rejection of TEMPO-CNC membranes formed by physical crosslinking (circles) and chemical crosslinking (crosses). Inset: Dried membranes. Left: physically crosslinked, right: chemically crosslinked.

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