

# Synthesis and characterization of novel Cellulose Nanocrystals-based Thin Film Nanocomposite membranes for reverse osmosis applications



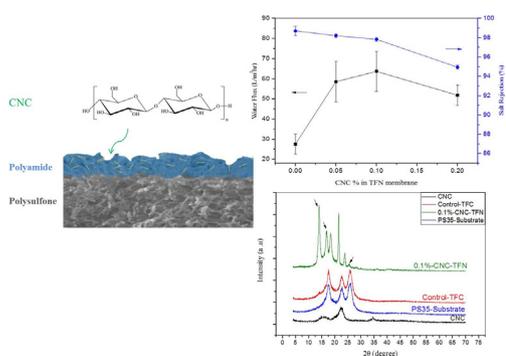
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## GRAPHICAL ABSTRACT



## ARTICLE INFO

### Keywords:

Cellulose nanocrystals (CNCs)  
Thin film nanocomposite membrane (TFN)  
Reverse osmosis  
Desalination  
Brackish water

## ABSTRACT

A novel TFN membrane was fabricated by embedding Cellulose Nanocrystals (CNCs) into the polyamide active layer. Membranes were synthesized by in-situ interfacial polymerization of *m*-phenylenediamine (MPD) and trimesoyl chloride (TMC) containing different amounts of CNCs. Successful incorporation of CNCs into the polyamide layer was confirmed by XRD analyses. Surface morphology of the membranes was characterized by SEM and AFM. Water contact angle measurements showed improved hydrophilicity of the TFN membranes. Desalination experiments with synthetic brackish water at 20 bar(g) revealed doubling of the water flux from 30 to 63 ± 10 L/m<sup>2</sup>h, without significantly compromising the salt rejection (97.8%) for 0.1% (w/v) CNCs loading. In the fouling/filtration experiments with 300 ppm of Bovine Serum Albumin (BSA) in the feed solution, the TFN membrane had 11% smaller water flux reduction compared to the control TFC membrane. The promising performance of the CNCs-based TFN membranes and the fact that CNCs are inexpensive and abundant nanoparticles indicates their potential for a large-scale use in water desalination. Moreover, since CNCs are safe and environmentally friendly nanoparticles their possible leaching out during membrane operation would not impose health and environmental concerns.

## 1. Introduction

Since the development of the Thin Film Composite (TFC)

membranes by Cadotte in 1970, reverse osmosis (RO) membranes have gone through several incremental improvements [1]. These advancements are mostly focused on overcoming the trade-off relationship

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between the membrane's permeance and selectivity, as well as enhancing membrane's antifouling properties and durability [2]. One of the breakthroughs in the fabrication of the composite membranes has been embedding nanoparticles (NPs) within the active polyamide layer. It is commonly reported that incorporation of NPs can enhance certain characteristics of the membrane such as increased hydrophilicity, surface charge density, and surface roughness, which lead to improvement in membrane performance [3]. Jeong et al., in their pioneering work in 2007, introduced zeolite NaA as nanoscale additives [4]. Since then various NPs such as TiO<sub>2</sub>, SiO<sub>2</sub>, Ag, Cu, carbon nanotubes (CNT), graphene oxides (GO), and other zeolites have been employed in the so-called Thin Film Nanocomposite (TFN) membranes [3,5,6]. However, the major challenge in commercialization of TFN membranes is leaching out of NPs into the downstream (permeate and retentate streams), which compromises the drinking water safety regulations. Besides, these commonly used NPs are typically quite expensive and difficult to produce [7–9].

The challenge imposed by the drinking water safety regulations can be addressed in two ways; (1) by preventing the leaching out of the NPs during the membrane operation; (2) by using biocompatible NPs. Ideally, the two approaches are combined. In this paper, we focus on the second approach by introducing cellulose nanocrystals (CNCs) to the TFN membranes. CNCs, also known as nanocrystalline cellulose (NCC), are natural rod/needle like particles, which are obtained from acid hydrolysis of native cellulose [10]. When biomass is treated in a harsh acid environment, the amorphous parts of the cellulose consisting of cellulose fibers, lignin, waxes, etc. dissolve, and a highly crystalline residue remains [10]. Fig. 1 represents the chemical structure of the cellulose chain. Depending on the source of the cellulose and the conditions of the acid treatment, the size of the CNCs varies, but in general, they are few hundred nanometers long and few nanometers in diameter [11,12]. They possess a high specific strength and Young's modulus, a large specific surface area, and a large negative zeta potential [12]. They also have a highly reactive surface due to the existence of the –OH groups, which make them favorable for chemical functionalization [11,12]. CNCs are biodegradable, renewable, have no/low environmental impact, and are typically known as non-toxic and safe particles [11–22]. According to the vendor, CelluForce Inc. (Montreal, QC, Canada), CNCs have been added to Canada's Domestic Substance List, which allows their unrestricted production and sale. Also, CNCs have received regulatory clearance for use in US and Europe as they fall into the least toxic classification in the Globally Harmonized System of Classification and Labelling of Chemicals [23]. These attractive properties of CNCs have harnessed a tremendous attention for a wide range of potential applications, for example, in nanocomposite polymers, protective coating, drug delivery, tissue engineering, solar cells, etc. [11,12]. Moreover, since cellulose biomass is abundant and the process of CNCs production is relatively simple, these NPs are very cost-effective, so if the performance of lab-scale CNC-based TFN membranes were favorable, their scale-up to an industrial level would not be prohibited by economic factors.

Although CNCs have been widely used in nanocomposite polymers, their application, particularly in hydrophobic polymeric membranes, has not been thoroughly investigated [20]. Bai et al. studied CNCs – polyvinylidene fluoride (PVDF) ultrafiltration (UF) membranes for water treatment [21]. They reported that in comparison to a control

membrane, incorporation of 0.1 wt% of CNCs in the membrane led to a 47.5% increase in the water flux without a significant BSA rejection decrease [21]. In another study, Li et al. prepared CNCs-polysulfone UF membrane for dialysis applications [22]. They reported that incorporation of 0.3 wt% CNCs in a composite membrane doubled its permeance (from 24 to 48.4 L/m<sup>2</sup>·h·mmHg), while the urea rejection increased by 44% (from 62.9% to 90.4%) [22]. Using SEM and AFM analyses, Li et al., reported that CNCs changed the structure of the UF composite membranes towards having larger finger-like pores and improved their connectivity. This was ascribed to the modified phase inversion kinetics, i.e., accelerated pervasion of water molecules into the casting solution [21]. Also, they noted a significant improvement of membrane wettability.

Lately, Daraie et al. fabricated the CNCs-polyethersulfone (PES) nanofiltration membrane by a phase inversion method [22]. They reported that CNCs-PES membranes (0.8% to 1.2% CNCs in the dope solution) showed two folds higher water permeability, and a 30% higher bivalent salt rejection than the pure PES control membrane. Moreover, they reported that CNCs-PES membranes had superior antifouling properties, showing a complete recovery of the water flux after a fouling test [22]. Although Daraie et al. did not study the leachability of CNCs from the membranes, they suggested that biocompatibility of CNCs allows them to “be safely employed for drinking water treatment” [22].

To our best knowledge, there is no article reporting the use of CNCs in the TFN membranes, fabricated by an interfacial polymerization method. Therefore, the main objective of this work is to study the possibility of incorporating CNCs, as safe and environmentally friendly NPs, in the thin film PA layer, and to investigate the effects of CNCs on the performance of the TFN membranes in comparison with the control TFC membrane.

Herein, the surface morphology of the synthesized membranes was characterized by SEM, AFM and contact angle measurements. Also, the presence and the effects of CNCs on the PA layer were investigated by FTIR and XRD. Antifouling properties of the best performing TFN membrane were examined and compared with the control TFC membrane. The permeance (water flux) and the salt rejection of the membranes were tested with a reverse osmosis system using synthetic brackish water as the feed solution.

## 2. Experimental

### 2.1. Materials

A polysulfone ultrafiltration membrane (PS-35) with the molecular weight cut off (MWCO) of 20 kDa, obtained from Nanostone Water, Waltham Massachusetts, USA, was used as a substrate. The reactant monomers *m*-phenylenediamine (MPD, purity of 99%) and 1,3,5-benzenetricarbonyl trichloride (TMC, purity > 98%) were obtained from Sigma-Aldrich. The organic solvent, *n*-hexane (purity > 99%), was also obtained from Sigma-Aldrich. CNCs were purchased from CelluForce Inc. (Montreal, QC, Canada). Sodium chloride (purity > 99.5%) was used as the solute in the feed. Bovine serum albumin (BSA) with the molecular weight of 66 kDa, used as the organic foulant to mimic proteins in the effluent organic matters (EfOM), was acquired from Sigma-Aldrich. All chemicals and CNCs were used as received, without

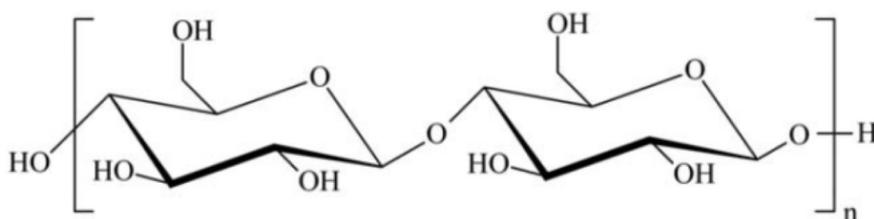


Fig. 1. Molecular structure of cellulose [11].

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