



Research paper

Properties and ultrafiltration efficiency of cellulose acetate/organically modified Mt (CA/OMMt) nanocomposite membrane for humic acid removal



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ABSTRACT

In this study, asymmetric nanocomposite membranes based on cellulose acetate and organically modified montmorillonite (OMMt) were prepared by a combination of solution dispersion and phase inversion methods. The effects of OMMt on the properties and performance of prepared nanocomposite membranes were investigated. The prepared nanocomposite membranes were characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), contact angle, porosity measurement, tensile strength techniques, and humic acid adsorption. Pure water flux (PWF) and humic acid rejection were studied using an ultrafiltration experimental setup. The results showed that the PWF significantly increased from 3.8×10^{-5} to $6.6 \times 10^{-5} \text{ m}^3/\text{m}^2 \cdot \text{s}$ with increasing OMMt concentration from 0 to 5 wt.%. By increasing the OMMt concentration, rejection decreases from 94.54% for pure cellulose acetate membrane to 91.85% for 2 wt.% OMMt/CA nanocomposite membrane, while rejection of 95.05% was obtained for 5 wt.% OMMt/CA membranes.

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1. Introduction

One of the most important challenges the world faces today is how to provide clean water for human consumption. Demand for potable water has increased because the population exceeds 7 billion and will reach 10 billion by 2050. One of the main contaminant species in surface waters is natural organic matter (NOM), which primarily consists of humic compounds (Gutman and Semiat, 2010). These are plentiful in the environment and can be classified into humic acid (HA), which is insoluble at a pH < 2, fulvic acid (FA), which is soluble at any pH, and humin, which is completely insoluble (Lowe and Hossain, 2008; Gutman and Semiat, 2010; Hamid et al., 2011). HA can lead to the formation of disinfection byproducts, including trihalomethanes (THM) and haloacetic acids (HAA), which are dangerous for human health (Costa and de Pinho, 2005; Weng et al., 2006; Lowe and Hossain, 2008; Rajesh et al., 2013). Hence, effective removal of HA from surface or ground water has prompted many efforts in the current development of water purification technologies.

The most important processes in the removal of HA in drinking water treatment plants are chemical oxidation, using chlorination or ozonation, activated carbon adsorption, coagulation, and membrane process (Alborzfar et al., 1998). Among these processes, membrane

technology has received the most attention because of its low energy consumption, simple process, low cost, and compatibility with the environment (Rajesh et al., 2013). Most recently, nanocomposite membranes have been applied to the membrane technology for removal of pollutants from water and wastewater. Polymer-inorganic nanocomposite membranes (PINM), also called mixed matrix membranes (MMM), are formed by incorporation of inorganic nanoparticle fillers into polymer matrix (Jaafar et al., 2009; Garg et al., 2011; Ma et al., 2012). Among the various inorganic fillers, clays have received specific attention due to their proper dispersion in the polymer matrix, high surface area (up to 750–800 m²/g), low cost, non-toxicity, and availability in nature (Ghaemi et al., 2011a, 2011b; Rhim, 2011; Daraei et al., 2013). The most common methods used in clay-polymer nanocomposite (CPN) technology are classified as: in-situ polymerization, melt intercalation, and solution dispersion methods (Anadão et al., 2010). Depending on the strength of polymer-silicate interaction, intercalated or exfoliated nanocomposite structures can be obtained as shown in Fig. 1. In intercalated structure, polymer chains are inserted into the layered silicate clays, and while in exfoliated structure, the individual clay layers are separated in a continuous polymer matrix that depends on clay loading (Ray and Okamoto, 2003). Montmorillonite (Mt) is a well-known layered silicate composed of silica tetrahedral and alumina octahedral layers. The layered structure of Mt can be modified by using the chemical and physical treatments in order to improve dispersion and compatibility of nanoparticles (Xing et al., 2011). Organically

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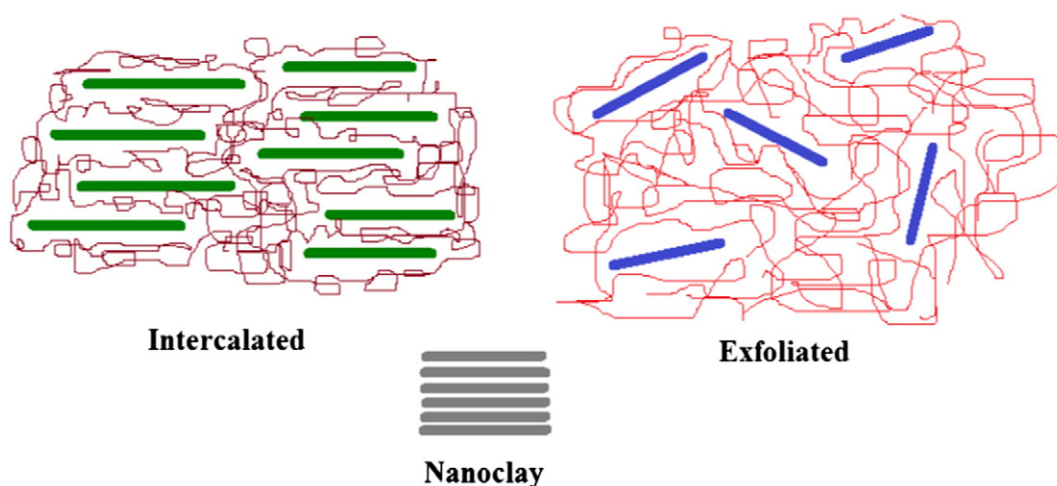


Fig. 1. Arrangement of layered silicates in polymer matrix.

modified Mt (OMMt), such as Cloisite, is very promising filler in CPN membranes and is expected to enhance the performance of polymeric membranes (Jaafar et al., 2009).

Cellulose acetate (CA) is nearly the first polymer that is applied as membrane for aqueous-based separation, i.e. reverse osmosis (RO) and ultrafiltration (UF) (Sivakumar et al., 2006). CA membrane has shown high hydrophilicity and good resistance to chlorine; however, this membrane usually contains a dense skin layer and a low porous sub-layer resulting in an extremely low flux (Ghaemi, 2011b). Hence, to improve the performance of CA membranes, modifications of polymer by proper additives seem to be essential.

In recent years, the effects of adding nanoclay, especially OMMt, on the performance and properties of nanocomposite membranes, have been researched. Modification of nanocomposite membrane by using OMMt on sulfonated poly phenyl ether sulfone/PTFE for fuel cell application (Xing et al., 2011), sulfonated poly (ether ether keton) (Jaafar et al., 2011), and sulfonated poly vinyl alcohol (Sanglimsuwan et al., 2011) for direct methanol fuel cell (DMFC) application, poly lactic acid for gas permeation (Koh et al., 2008), PVDF membranes for survey the effect of OMMt nanoclay on membrane properties (Hwang et al., 2011a, 2011b), PVDF-HFP membrane for lithium ion secondary batteries (Zhang et al., 2014), and poly(dimethyl siloxane) for pervaporation (Garg et al., 2011) are examples of the aforementioned literatures. There are a few studies in the field of water treatment that use nanocomposite membrane made with OMMt. Ghaemi et al. (2011a, 2011b) fabricated nanofiltration poly ether sulfone/OMMt membrane to be used for the removal of pesticides. They found that the addition of OMMt improved the hydrophilicity, mechanical, and thermal properties of prepared nanocomposite membrane. Additionally, PWF, permeation, and rejection of pesticide were enhanced.

However, to the best of our knowledge, the preparation and characterization of CA/OMMt nanocomposite membrane has not been studied previously. In addition, there is no research that explains the influence

of OMMt on performance of ultrafiltration membranes. Accordingly, in this work, nanocomposite membranes on the base of cellulose acetate in different loadings of Cloisite 15A were prepared via a combination of solution dispersion and wet-phase inversion methods. The morphology, structure, hydrophilicity, thermal, and mechanical properties of as-prepared nanocomposite membranes were studied. Furthermore, performance of prepared membrane was evaluated using PWF, HA adsorption, and separation efficiency of humic acid solution, and results compared with those of pure CA membrane.

2. Experimental

2.1. Materials

Cellulose acetate ($M_n = 30000$), was used as the basic polymer for the preparation of the asymmetric nanocomposite membrane and was supplied by Sigma-Aldrich (Germany). OMMt clays (Cloisite 15A) with a cationic exchange capacity of 1.2 meq/g were provided by Southern Clay Products Inc. (USA). N,N-Dimethylformamide (DMF) from MP biomedical (Netherlands) was used as solvent. Deionized water was used as the non-solvent in coagulation bath.

2.2. Preparation of nanocomposite membranes

Neat cellulose acetate membrane and cellulose acetate/OMMt nanocomposite membranes for ultrafiltration were prepared using the wet-phase inversion method. At first, a certain percentage of OMMt (0, 0.5, 1, 2, 3, 4 and 5%) was added to DMF and stirred for 4 h in ambient temperature. The solutions were further sonicated for 3 h to ensure proper dispersion of the nanoparticles. Next, CA dissolved in DMF was added to the prepared OMMt dispersion and the solution was mixed and vigorously stirred for 15 h. The prepared solutions were kept for at least 24 h to remove air bubbles that may exist in the solutions. After degassing, the solutions were cast uniformly on a glass substrate using an adjustable casting bar (Neurtek2281205) with a thickness of 250 μm . Subsequently, the solvent present in the casted film was allowed to evaporate for 30 s before immersion in a water coagulation bath (22 °C). After 12 h, all the membranes were removed from the coagulation bath and then squeezed in two filter papers for 24 h at ambient temperature for drying. The resultant membranes are further referred to CA (neat polymeric membrane), CA-0.5 (0.5%), CA-1 (1%), CA-2 (2%), CA-3 (3%), CA-4 (4%), and CA-5 (5%) based on the OMMt dosage. The conditions under which the membranes were prepared are listed in Table 1.

Table 1
Composition of casting solution.

Membrane	Composition of casting solution (wt.%)		
	CA	DMF	OMMt
CA	18	82	0
CA-0.5	18	81.5	0.5
CA-1	18	81	1
CA-2	18	80	2
CA-3	18	79	3
CA-4	18	78	4
CA-5	18	77	5

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