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Study on the fouling behavior of silica nanocomposite modified polypropylene membrane in purification of collagen protein

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ABSTRACT

In this study, microporous polypropylene (PP) membrane was fabricated via thermally induced phase separation (TIPS) method. Using Stober method, hydrophilic SiO₂ nanoparticles (NPs) were synthesized by *in situ* and *ex situ* approaches and covalently connected to the membrane via hydrolysis-condensation reactions with hydroxyl (–OH) groups, created by Piranha solution (3:1, v/v, H₂SO₄ and H₂O₂) in pretreatment step. The average sizes of SiO₂ NPs synthesized by *in situ* and *ex situ* approaches were about 250–300 nm and 15–20 nm, respectively. Incorporation of SiO₂ NPs on the membrane surface improved surface hydrophilicity, pure water flux and mechanical properties and increased the portion of reversible fouling ratio (RFR) which consequently resulted in higher flux recovery (FR) of the membrane due to the hydrophilic property of SiO₂ NPs. The flux recovery (FR) increased from 64.5% for unmodified membrane to 71.8%, 79.2% and 88.6% for PP-SiO₂ composite membranes prepared by *in situ* and two *ex situ* routes with 6 and 12 h immersion time intervals, respectively. Application of combined fouling models revealed that the “cake filtration-standard blockage” fouling mechanism is prevailing fouling mechanism for all membranes.

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1. Introduction

The membrane is at the heart of every membrane process and can be considered as a permselective barrier or interface between two phases (Mulder, 1996). Membrane separation processes are currently applied in various fields such as water and wastewater treatment, medicine, pharmacy, food and beverage industries (Pabby et al., 2009; Zhu et al., 2013; Xia et al., 2003; Bottino et al., 2002). Recently, membrane-based processes gained importance in biotechnology due to their ability for size and/or charge based protein separation. Pressure-driven processes of microfiltration, ultrafiltration and nanofiltration are usually applied for protein separation and purification (Saxena et al., 2009; Shen et al., 2009;

Jafarzadeh and Yegani, 2015; Jafarzadeh et al., 2015a). Collagen based proteins have recently attracted great attention due to their extensive applications in biotechnology, tissue engineering and biomedical science (Xiong et al., 2009; Duan et al., 2009). However, there are few reports about Collagen proteins purification using membranes (Shen et al., 2009; Jafarzadeh and Yegani, 2015; Jafarzadeh et al., 2015a). Despite the advantages of membrane processes, membrane fouling is one of the major limitations for practical applications of membrane processes. Membrane fouling is the accumulation of substances on the surface and/or within the pores which results in the deterioration of the membrane performance. Fouling is a complicated phenomenon which is efficiently influenced by operating conditions such as trans-membrane pressure,

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cross-flow velocity, and temperature; feed characteristics such as the type, size and concentration of foulant, pH of feed and also membrane properties including hydrophilicity, roughness and the type as well as the size of pores (She et al., 2009; Wang and Tang, 2011). The interaction between membrane surfaces and solution components plays an important role in the extent of membrane fouling (Nady et al., 2011). It is commonly accepted that a hydrophilic membrane surface provides better performance than a hydrophobic membrane surface against fouling (Hilal et al., 2005; Liang et al., 2012). Unfortunately, most polymeric materials; used for membrane fabrication, are weakly hydrophilic such as Polyethylene (PE), Polyvinylidene fluoride (PVDF), Polysulfone (PSf), Polyethersulfone (PES) and polypropylene (PP).

PP is superior to many other polymers in terms of mechanical strength, chemical stability, thermal and chemical resistance and low cost. Therefore, PP is a very promising material for separating membranes (Xu et al., 2013; Yu et al., 2007; Hu et al., 2006). However, PP is a hydrophobic material since it lacks any polar functional group and consequently PP membranes can be easily fouled and cause a series of problems in practical application. Hence, lots of efforts have been devoted to enhance the hydrophilicity of the polymeric membranes (Zhu et al., 2013; Xu et al., 2013; Yu et al., 2005; Song et al., 2014; Yan et al., 2008).

Membrane modification can be divided into six main groups: coating, blending, grafting, chemical treatment, composite and combination of aforementioned methods (Nady et al., 2011).

Among all modification methods, organic–inorganic composite membranes demonstrate promising performances and are predicted to gather the intrinsic properties of both polymeric and inorganic membranes and give interesting advantages of the hybrid membrane such as great thermal and chemical resistances, good separation performances and excellent adaptation to the severe operating conditions (Xu et al., 2009).

In general, the composite membranes can be fabricated by two methods; the first method is blending the inorganic filler or its precursors with dope solution and the second method is direct incorporation of an inorganic phase into the polymeric membrane substrate (Jafarzadeh and Yegani, 2015; Jafarzadeh et al., 2015a; Liang et al., 2012; Zhang and Wang, 2013; Liang et al., 2014). Regarding the first method, Jafarzadeh et al. fabricated ZnO/PE composite membrane by dispersing hydrophilic ZnO nanoparticles into the polyethylene casting solution and showed that the antifouling property of the composite membrane has considerably improved (Jafarzadeh et al., 2015a). Linag et al. (2012) fabricated anti-irreversible fouling PVDF membrane by adding ZnO nanoparticles with different dosage as an additive into the membrane matrix for the modification of the internal surfaces of membrane pores. The multi-cycle filtration experiments showed that the modified PVDF membranes demonstrated significant anti-irreversible fouling property. All the modified membranes achieved almost 100% water flux recovery after physical cleaning, whereas the raw membrane only reached 78% recovery (Liang et al., 2012). Liang et al. (2014) prepared PVDF/SiO₂ hybrid membranes by the thermally induced phase separation followed by an *in situ* sol–gel process. The surface hydrophilicity, pure water flux and mechanical properties were obviously improved with the formation of SiO₂ particles in the hybrid membranes. Moreover, the hybrid membranes possess excellent anti-compression

property due to the supporting effect of inorganic SiO₂ particles.

It seems that the second method would be more effective in modifying the membrane surface property since the inorganic phase is in exposure without the cover and interference from the polymeric matrix (Zhang and Wang, 2013). Regarding this method, there are successful reports (Xu et al., 2013; Zhang and Wang, 2013, 2014). Zhang and Wang (2014) fabricated hydrophobic organic–inorganic composite hollow fiber membrane by incorporating the fluorinated silica (fSiO₂) inorganic layer on the polyetherimide (PEI) organic substrate using sol–gel method. The incorporation of the fSiO₂ inorganic layer not only offered high hydrophobicity that is beneficial in membrane contactors, but also protected the polymeric substrate from the attacks of chemical absorbents, giving the longer lifespan for membrane. To improve the separation performances of polypropylene membranes, Xu et al. (2013) deposited TiO₂ on the membrane pore surface using the atomic layer deposition strategy with and without a plasma pretreatment step on the membrane. The TiO₂-deposited membranes showed improved anti-fouling property compared to the unmodified membranes due to enhanced hydrophilicity.

In this study, we report a novel method to fabricate a hydrophilic organic–inorganic nanocomposite membrane using direct incorporation of the silica (SiO₂) nanoparticles on the PP membrane substrate via sol–gel process for Collagen protein purification. PP membrane was pre-treated by Piranha solution (mixture of H₂O₂ and H₂SO₄) to create hydroxyl functional group (–OH) on the membrane surface (Raveshian et al., 2014). The existence of hydroxyl group ensures the chemical grafting of silane coupling agents on to the membrane surface.

A set of experiments including Scanning Electron Microscopy (SEM), Field Emission Scanning Electron Microscopy (FE-SEM), Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) spectroscopy, Energy dispersive X-ray (EDX) spectroscopy, pure water flux, static contact angle, porosity and mechanical strength measurements were carried out to characterize the structural and operational specifications of unmodified and modified PP membranes. Moreover, in order to investigate the impact of membrane modification on the fouling behavior of modified PP membranes, dead-end filtrations of Collagen protein using unmodified and modified PP membranes were carried out and fouling mechanisms during protein filtration were investigated using combined models (Bolton et al., 2006).

2. Theory of fouling and combined models

Four mechanisms are introduced for membrane fouling during membrane filtration, consisting of complete blocking, intermediate blocking, standard blocking and cake filtration (Hermans and Bredée, 1936). In the case of cake filtration model, foulants form a layer on the membrane surface. This layer grows with time and causes further flux decline (Bottino et al., 2002). Complete blocking assumes that particles seal off pore entrances and prevent flow. Intermediate blocking is similar to complete blocking but assumes that a portion of the particles seal off pores and the rest accumulates on top of other deposited particles (Bolton et al., 2006). Standard blocking, also called internal pore blocking, occurs when small size solutes deposit or adsorb onto the pore walls in the membrane (Bowen and Jenner, 1995). These mechanisms occur either

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