



The effect of membrane surface charges on demulsification and fouling resistance during emulsion separation



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ARTICLE INFO

Keywords:

Oil/water separation
Membrane fouling
Surface charge
Surfactant-stabilized emulsion
UV-initiated polymerization

ABSTRACT

The effect of membrane surface charge on demulsification and fouling resistance during emulsion separation is investigated. By grafting poly(2-dimethylaminoethyl methacrylate) (pDMAEMA) and poly(oligo(ethylene glycol) methacrylate) (pOEGMA) onto polypropylene (PP) membrane via ultraviolet (UV)-initiated polymerization, the obtained membranes PP-g-pDMAEMA and PP-g-pOEGMA present positive and negative surface charges in water, respectively. During the emulsion separation, PP-g-pOEGMA and PP-g-pDMAEMA membranes exhibit different separation efficiencies and pollutant resistant behaviors to the emulsions prepared from anionic surfactant (sodium dodecyl sulfate, SDS) and cationic surfactant (cetyltrimethylammonium bromide, CTAB). Comparing to PP-g-pOEGMA, the separation efficiency of PP-g-pDMAEMA was 15% (SDS emulsion) and 300% (CTAB emulsion) higher. But the flux recovery ratio of PP-g-pOEGMA was raised 30% (SDS emulsion) and 16% (CTAB emulsion). Hence, the membrane surface positive charges assist demulsification, but aggravate the membrane fouling. The mechanism obtained is beneficial to construct membranes with high separation efficiency and long-term durability.

1. Introduction

As water pollution caused by oil spill and oily wastewater discharge is one of the most serious environmental problems in the world, oil/water separation technology attracts more and more attentions in the last decades [1–4]. Recently, various kinds of efficient separating materials have been developed, mainly based on surface wettability [5–7]. Surface chemistry and surface roughness are key factors to generate surfaces with superhydrophobicity [8–10] or superhydrophilicity [11–13], which are beneficial to the oil/water separation. However, the separation of emulsified oil/water mixtures is still a challenge, especially for surfactant-stabilized microemulsions with a droplet size below 20 μm [14].

The emulsion separation is far more complex than the separation of layered oil/water mixture [15]. Some work has succeeded in separating oil/water emulsion by membrane surface construction [16–19]. For example, Yang et al. prepared superhydrophilic polypropylene (PP) membrane by dopamine/polyethyleneimine (PEI) co-deposition and achieved ultra-high water permeability and oil-in-water emulsion separation performance [19]. Shi et al. used TiO_2 nanoparticles to decorate PVDF membrane and produce high roughness. The as-prepared

superhydrophilic PVDF membrane showed a high separating efficiency (nearly 99%) and durability [20]. Although these membranes have been successfully applied to the separation of oil/water emulsions, further improvement is still required to enhance the separation efficiency and pollution resistance during long-term use [10].

The performance of membrane is significantly affected by the surface properties, such as wettability, charge, pore size and roughness [21–24]. During the membrane-assisted emulsion separation, it is found that electrostatic interactions play an important role in demulsification. In principle, enhancement of the interfacial interaction between oil droplets and surfaces is beneficial to the breaking and coagulation of emulsion particles [25,26]. As reported by literatures, the positively charged surfaces are in favor of emulsion demulsification [27,28]. However, the influence of surface charge to the demulsification process as well as the embedded mechanism is still unclear.

Membrane fouling is another critical issue limiting current membrane application. It will reduce the flux and the membrane life span, causing expensive maintenance during application [29,30]. Liu et al. demonstrated that both surface charge and surface hydrophilicity regulated the irreversible fouling of membranes [31]. Most foulants, such as oils and proteins, are hydrophobic in nature. Other soluble

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matters, such as surfactants, dyes, humates, bacteria and marine organisms, are negatively or positively charged [32]. The foulants are apt to adsorb onto surfaces by hydrophobic or electrostatic interactions [33]. Thus, hydrophilic and neutral surface is thought to be beneficial for antifouling performance [34]. Generally, poly(ethylene glycol) (PEG) [35,36] and zwitterionic polymers [37,38] are widely used for building antifouling surfaces. However, due to the ion adsorption from aqueous solution or ionization of hydrophilic polymers, hydrophilic membrane may not be neutral in aqueous environment [39]. For example, the relative strength of acid and base components in zwitterionic polymer may be different in a specific pH environment, leading to a negatively or positively charged surface [40].

Therefore, the surface charge plays a key role on both demulsification and antifouling behavior, which would further influence the overall performance of membrane during emulsion separation. To the best of our knowledge, few work systematically studied the effect of surface charge on the separation process of emulsions. In our investigation, two types of oil-in-water (O/W) emulsions were prepared using anionic surfactant (sodium dodecyl sulfate, abbreviated as SDS) and cationic surfactant (cetyltrimethylammonium bromide, abbreviated as CTAB). Two types of membranes were constructed by grafting poly(2-dimethylaminoethyl methacrylate) (pDMAEMA) and poly(oligo(ethylene glycol) methacrylate) (pOEGMA) onto polypropylene (PP) microfiltration membranes. The emulsion separation efficiency and pollutant removal performance of PP-g-pOEGMA and PP-g-pDMAEMA membranes towards two emulsions were investigated (Fig. 1). Finally, the strategy of constructing surfaces for efficient and durable emulsion separation was discussed.

2. Experimental

2.1. Materials

Polypropylene microfiltration membrane ($\Phi = 0.22 \mu\text{m}$) was purchased from Dalian Filter Membrane Technology Co., Ltd (Dalian, China). DMAEMA (purity 98%), OEGMA ($M_n = 360$), N,N'-methylene bisacrylamide (MBA, purity 99%) were purchased from Sigma-Aldrich Company. 2-Isopropylthioxanthone (ITX, purity > 98%) was purchased from Aladdin. Dopamine hydrochloride (DA) was purchased from J&K Scientific Company (Beijing, China). SDS and CTAB were provided by Hangzhou Gaojing Fine Chemical Company and Tianjin Kemiou Chemical Reagent Co., Ltd, respectively. Diesel was purchased from local gasoline station. All of the chemicals used as received without further purification.

2.2. Modification of PP membrane

PP membrane was firstly pretreated by ethanol, and then incubated in 2 mg/mL DA solution (dissolved in Tris buffer solution, pH = 8.5), followed by being shaken at room temperature for 12 h. Then, the as-prepared membrane (PP-pDA) was washed with ethanol and water and then dried in oven at 40 °C overnight.

The polymerization was taken on the surface of membrane via UV-initiated grafting polymerization technology. The preparation process followed two steps: Firstly, PP-pDA membrane was immersed into 0.4 mg/mL ITX/acetone solution and irradiated with UV light ($\lambda = 365 \text{ nm}$) for 5 min in UV curing machine (HWUV400X, Zhonghe Mechanical Equipment Limited, China). Then, the pre-irradiated membrane was immediately put into aqueous solution of monomer and exposed to the UV light again for 1 h. The concentration of DMAEMA was 10 mg/mL, and the mass ratio of DMAEMA: MBA (cross-linker): ITX was 50: 2.5: 2. During the grafting of pOEGMA, the monomer concentration was 20 mg/mL, and the mass ratio of OEGMA: MBA: ITX was 50: 5: 5. Finally, the as-prepared membranes (PP-g-pDMAEMA and PP-g-pOEGMA) were rinsed with deionized water to remove excess monomer and then dried in oven at 40 °C.

2.3. Characterization of modified membrane

The surface chemistry of resulting membranes was characterized by Fourier transform infrared spectroscopy (FT-IR, Bruker Vertex) under ATR mode. The surface chemical composition variation was confirmed by an X-ray Photoelectron Spectroscopy (XPS, K-Alpha, Thermo Scientific, UK). Surface morphology of modified membranes was observed by field emission scanning electron microscopy (FESEM, ALTRA55, Germany). The underwater oil contact angle (OCA) was carried out using a contact angle measuring instrument (DSA10, KRÜSS, Germany) using the captive bubble method. Diesel droplet was introduced onto the surface of membrane under water and the contact angle was analyzed. The surface zeta potential was measured by Zeta potential analyzer (SurPASS, Anton Parr, Austria). The distribution of pore size was measured by gas osmosis pore size analyzer (CFP-15WAE, Porous Materials Inc., US).

2.4. Oil/water emulsions separation experiment

Surfactant-stabilized diesel-in-water emulsions were prepared by mixing water and diesel in 100:1 (v/v) with addition of 0.5 mg/mL of emulsifier under magnetic stirring at high speed for 5 h. The emulsions were observed under optical microscope (LV100, Nikon, Japan). The particle size of the feed emulsion and filtrate was determined by dynamic light scattering particle size analyzer (Nano-S, Malvern,

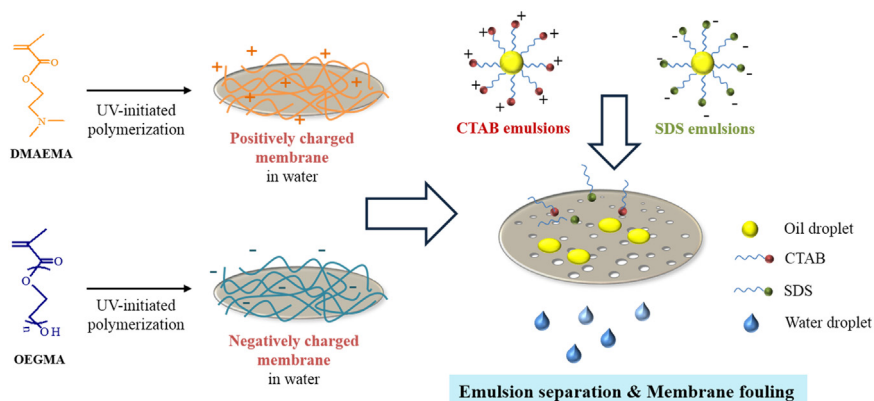


Fig. 1. The scheme of constructing PP-g-pDMAEMA and PP-g-pOEGMA membranes with different surface charges for the study of emulsion separation and pollutants removal. The emulsions were prepared with CTAB and SDS, respectively. The PP membranes were modified by pDMAEMA and pOEGMA grafting, respectively.

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