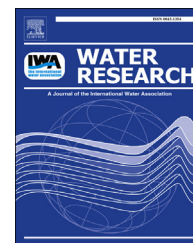




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Microalgae recovery by ultrafiltration using novel fouling-resistant PVDF membranes with *in situ* PEGylated polyethyleneimine particles

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ARTICLE INFO

Article history:

Received 26 June 2014

Received in revised form

28 October 2014

Accepted 3 December 2014

Available online 10 January 2015

Keywords:

Water treatment

Resource recovery

Mixed matrix membranes

Fouling resistant membranes

Microalgae harvesting

ABSTRACT

In this article, we report the preparation, characterization and microalgae recovery potential of a new family of fouling-resistant polyvinylidene fluoride (PVDF) ultrafiltration (UF) membranes embedded with hydrophilic and PEGylated polymeric particles. To optimize membrane performance for microalgae harvesting, we investigate the effects of three hydrophilic additives (Pluronic F-127, polyvinylpyrrolidone and polyethylene glycol) on the morphology, pore size, bulk composition, surface composition, wettability and surface charge, flux and fouling resistance of the mixed matrix PVDF membranes with *in situ* PEGylated polyethyleneimine (PEI) particles. Our filtration experiments show that a mixed matrix PVDF membrane with PEGylated PEI particles and Pluronic F-127 additive (PNSM-1) has an algae retention of 100% with a permeate flux of 96 L/m²/hr that is larger (by ~50%) than that of a commercial and hydrophilic PVDF UF membrane with a molecular weight cut-off of 30 kDa using a suspension of *Chlorella* sp. KR-1 microalgae with 1.2–1.4 g/L of dry biomass. The algae and water flux recovery rates of our new PNSM-1 are equal to ~94% and 100%, respectively, following a simulated membrane wash with deionized water and two subsequent water and microalgae filtration cycles.

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1. Introduction

The sustainable supplies of clean water, carbon-neutral energy and food are among the most challenging problems

facing society and the global economy in the 21st century (Diallo and Brinker, 2010; Diallo et al., 2013; Fromer and Diallo, 2013). The availability and supplies of water, energy and food are strongly coupled. Oil production and electricity generation by thermal/nuclear power plants require abundant amounts

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<http://dx.doi.org/10.1016/j.watres.2014.12.002>

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of water. Similarly, the production and delivery of clean water require a lot of energy. There is a growing realization that significant amounts of nutrients [e.g. nitrogen (N) and phosphorous (P)] will be needed in agriculture as the world faces the daunting challenge of doubling the amount of food it currently produces to feed around 9 billion people in the next 30 years while reducing the environmental footprint of agriculture (Diallo and Brinker, 2010). Microalgae biotechnology is emerging as a promising platform for achieving a sustainable water-energy-food nexus (Rosenberg et al., 2008; Christenson and Sims, 2011; Pitman et al., 2011; Razzak et al., 2013). Microalgae are micron-sized organisms (3–30 μm in diameter) that can be cultivated in non-agricultural lands (e.g. open ponds) or in closed photobioreactors using impaired water (e.g. wastewater, brackish water and seawater), carbon dioxide (CO_2), sunlight and dissolved nutrients (Razzak et al., 2013). They can capture and convert CO_2 into (i) valuable products (e.g. dietary supplements and pigments) and/or (ii) biomass with high lipid content for biofuel production (Rosenberg et al., 2008; Pitmann et al., 2011). Moreover, microalgae are very efficient at removing N and P from wastewater thus providing new opportunities to treat nutrient-rich wastewaters prior to their discharges into receiving water bodies (e.g. lakes, rivers and estuaries) while producing slow-release fertilizers for agriculture (Razzak et al., 2013). Although microalgal biomass has great potential to advance global sustainability, its efficient harvesting and subsequent downstream processing into useful products (e.g. biofuels and fertilizer substitutes) remains a major and unresolved challenge (Milledge and Heaven, 2013).

Harvesting commonly refers to the extraction and concentration of algal biomass from a dilute culture medium into a paste followed by dewatering and drying. Several solid–liquid separation processes are being evaluated for microalgae harvesting including sedimentation, centrifugation, flocculation, flotation and filtration (Milledge and Heaven, 2013). Pressure-driven membrane processes [microfiltration (MF) and ultrafiltration (UF)] are increasingly being investigated for microalgae harvesting due to their efficiency, ease of operation, scalability and reduced footprint (Zhang et al., 2010; Chen et al., 2012; Hwang et al., 2013; Sun et al., 2013). Due to its high thermal/chemical resistance and tensile strength, polyvinylidene fluoride (PVDF) has emerged as an important and widely utilized base polymer in the preparation of UF/MF membranes for a broad range of applications including (i) water treatment (Du et al., 2009), (ii) wastewater treatment (Chen et al., 2011) and (iii) microalgae harvesting (Hwang et al., 2013). However, pristine PVDF membranes are hydrophobic and thus can be easily fouled by the sorption, deposition and irreversible attachment of extracellular and hydrophobic macromolecules (e.g. proteins, polysaccharides and lipids) present in surface water, wastewater and microalgae culture media (Du et al., 2009; Chen et al., 2011; Sun et al., 2013). A broad range of strategies have been developed to prepare hydrophilic PVDF membranes with low adhesion/binding affinity toward hydrophobic macromolecules during the last two decades (Liu et al., 2011). Synthetic strategies that have been utilized to increase the fouling-resistance of PVDF membranes include (i) surface coating with hydrophilic monomers followed by curing/crosslinking (Stengaard, 1998;

Du et al., 2009), (ii) covalent surface grafting with hydrophilic polymer brushes and inorganic nanoparticles (Liu et al., 2007; Chang et al., 2009; Liang et al., 2013), (iii) *in situ* polymerization of acrylic monomers (Tao et al., 2012) and (iv) *in situ* dope blending with polyethylene glycol (PEG)-based amphiphilic copolymers (Hester and Mayes, 2002; Zhao et al., 2007; Lin et al., 2013) and hydrophilic inorganic particles (Yan et al., 2006; Li et al., 2009).

It is worth mentioning that surface coating has been successfully implemented in commercial PVDF membranes used in microalgae harvesting such as the ETNA UF membranes from Alfa Laval Naksov A/S (Sun et al., 2013). However, these membranes are “tighter” with lower water flux and pore rating [e.g. molecular weight cut-off (MWCO) of 10 kDa] (Stengaard, 1998; Liu et al., 2011). Although grafting has emerged as a versatile surface modification strategy for PVDF membranes (Liu et al., 2007; Chang et al., 2009), it also causes a decrease of membrane water permeability due to pore plugging (Liu et al., 2011). Moreover, surface grafting requires the utilization of synthetic procedures that are difficult to scale-up including surface-selective photo-initiated polymerization and surface-initiated atom transfer radical polymerization (Liu et al., 2011). In contrast, the modification of PVDF membranes via the addition and blending of PEG-based amphiphilic copolymers in the casting solutions is a much simpler strategy that can be applied to industrial production (Liu et al., 2011). However, this strategy is predicated upon the design, synthesis and scale-up of amphiphilic copolymers with the required structure (e.g. linear, comb or branched), molar mass and hydrophile-lipophile balance (e.g. number and length of PEG blocks) to induce the surface segregation of the PEG blocks during membrane casting (Hester and Mayes, 2002; Zhao et al., 2007; Lin et al., 2013). Similarly, the preparation of mixed matrix PVDF membranes with embedded inorganic and hydrophilic nanoparticles (e.g. TiO_2 and Al_2O_3) involves the dispersion of preformed and purified nanoparticles in a suitable polymer solution followed by membrane casting (Yan et al., 2006; Li et al., 2009). Moreover, the uniform dispersion of inorganic nanoparticles in mixed matrix membranes (MMMs) remains a major challenge that impacts membrane mechanical strength and performance (Liu et al., 2011). Hydrophilic polymeric particles could provide greater flexibility for the preparation of MMMs with improved particle-matrix compatibility, water flux and resistance to fouling by organic macromolecules/colloids and microalgae. In a recent article, we described a simple and facile route to the preparation of mixed matrix PVDF membranes with *in situ* synthesized polyethyleneimine (PEI) particles (Kotte et al., 2014). In this study, we show that the amino groups of the embedded PEI particles of these new membranes can be reacted with poly (ethylene glycol)diglycidyl ether (PEGDE) in the casting solutions to prepare mixed matrix PVDF membranes with uniform particle distribution and high loadings of *in situ* PEGylated PEI particles (>50 wt%). To optimize membrane performance for microalgae harvesting, we investigated the effects of three hydrophilic additives [Pluronic F-127 (PF-127), polyvinylpyrrolidone (PVP) and polyethylene glycol (PEG)] on the morphology, pore size, bulk composition, surface composition and charge, wettability, flux and fouling resistance of the mixed matrix PVDF membranes with *in situ* PEGylated PEI particles. The overall results

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