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Adhesion forces between humic acid functionalized colloidal probes and polymer membranes to assess fouling potential



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ABSTRACT

Fouling of membrane surfaces by organic, inorganic and biological materials is a significant cause of increased operational costs and power consumption for membrane separation processes including reverse osmosis, nanofiltration, membrane bioreactors and membrane distillation. Novel polymeric membrane surfaces are being developed by numerous research groups to counter foulant attachment and build up. One such type of membranes under development are ones which contain an active surface created using polymeric bicontinuous microemulsions (PBM) [1]. In this work we use humic acid functionalised colloidal probes to simulate organic foulants to investigate the adhesion forces between polymer membranes modified with a PBM coating, both in high purity water and model textile dye wastewater (MTDW). Membrane performance varied considerably when comparing different preparations and with the commercial PES membrane which was used as the base-layer. When making measurements in the two liquid media it was found that the behaviour was very different, with some membranes showing low adhesion in water showing high adhesion in the textile dye wastewater and vice versa. Water contact angle measurements of clean membranes showed good correlation with measured adhesion forces. Conversely, contact angle measurements made with membranes after prolonged exposure to MTDW showed a strong inverse correlation with the measured adhesion forces in MTDW, with more hydrophilic surfaces showing greater adhesion with the functionalised probe. Measurements of contact angle of humic acid before and after exposure to MTDW showed a change from a hydrophobic substance (129°) to a moderately hydrophilic substance (29°). These measurements suggest that the modification of both membranes and probe by components in the MTDW changed the adhesion forces experienced by the HA probe from favouring hydrophobic interactions to hydrophilic ones.

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1. Introduction

With a growing global population, access to clean water is due to become an increasingly critical issue, both for the production of safe drinking water, as well as for providing supplies for agricultural and industrial consumption [2]. As many as 3.9 billion people worldwide are expected to be living in severely water stressed areas by 2030 according to the OECD [3]. Deficits may be reduced by the recycling and reuse of municipal, domestic and industrial wastewater and by the desalination of brackish and saline water

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http://dx.doi.org/10.1016/j.memsci.2015.03.018 0376-7388/© 2015 Elsevier B.V. All rights reserved. leading to a reduced demand on natural freshwater sources. One family of technologies which is of crucial importance in the production of safe water from contaminated or non-potable sources are membrane filtration based technologies including nanofiltration (NF), reverse osmosis (RO), membrane bioreactors (MBR) and membrane distillation (MD) [4].

A major problem with membrane separation technologies is the fouling of membranes by inorganic, organic and biological foulants which are a major barrier to their efficient operation. Fouling of membranes can lead to a substantial decrease in membrane flux causing a need for increased trans-membrane pressures to maintain water flow rates, pre-treatment of feed-water, and system downtime to allow membrane cleaning all of which serve to increase operating costs considerably. Fouling by microorganisms is considered to be the greatest cause of flux decline and loss of retention in membranes

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[5]. For seawater RO alone the global market for pre-treatment chemicals to prevent biofouling has been estimated at \$500 million in 2011 and is expected to rise to almost \$1 billion by 2016 [6]. For MBR it has been estimated that 30–50% of power consumption costs are due to the need to aerate the membranes to mitigate biofouling [7], a significant amount if this technology is to be more widely used to treat industrial and domestic effluent. All of these effects serve to increase the operating costs and power consumption of water treatment facilities using membrane based technology and to a decrease in the operating lifetimes of the, usually expensive, membranes used.

Atomic force microscopy (AFM) is a versatile technique which is incredibly useful for characterisation of surfaces, particularly as it is able to operate within a fluid environment, which can be made to mimic process conditions [8]. Amongst other capabilities, its ability to measure adhesion forces between different materials is of great value, particularly when the sharp scanning tip is replaced by a microscale particle, a "colloidal probe" made from or functionalised with a material of interest [9–11]. This approach has an advantage of requiring very small amounts of sample compared to those needed for lab or pilot scale tests of membrane filtration, allowing for the possibility of screening a relatively large number of small samples for low adhesive properties prior to upscaling of favourable samples to pilot scale tests.

The use of colloidal probes was first reported by Ducker and colleagues [12,13] who used a $3.5\,\mu m$ diameter silica sphere to measure its interactions with a polystyrene surface as a function of solution ionic strength and pH. By measuring the long range Derjaguin-Landau-Verwey-Overbeek (DLVO) forces under different conditions it is possible to estimate the ability of a membrane surface to reject attachment of a colloidal foulant to the surface, whilst measurement of adhesion forces can give information about the strength of foulant-membrane attachments. This is of particular use when modifying membrane surfaces to resist fouling. From adhesion measurements it can be seen whether a membrane is likely to be fouling-resistant using only a small sample of membrane without the need for fabrication of larger amounts of membrane which may be expensive. For example Hilal et al. [14] measured adhesion forces for poly-ether sulphone (PES) membranes which were either unmodified or modified with 2-dimethyl-aminoethylmethacrylate (qDMAEMA) in order to reduce fouling. It was found that the modification reduced the adhesion forces experienced with using a silica sphere from 39.5 mN/m to 24.8 mN/m, suggesting that the modified membrane would be more resistant to fouling by silicate particulates. Other probes can be used to assess attachment of other foulants. For instance Guillen-Burrieza et al. [15] reported interaction force measurements between a probe comprising a CaCO₃ crystal and PVDF and PTFE membranes developed for membrane distillation applications, as part of a study of scaling of these membranes during normal operation. A similar approach was earlier used to measure interactions of CaCO₃ with stainless steel surfaces, allowing assessment of effects of anti-scalant and anti-foaming additives on inorganic fouling of desalination equipment [9].

Humic acid (HA) is a heterogeneous collection of chemical species which are a major component of natural organic matter in soil and river water, formed through the natural decay of biological matter, particularly plant matter [16]. It is also a major foulant of filtration membranes [17]. HAs have been used by many investigators as a model foulant in studies of fouling of membranes by NOM including microfiltration [18], ultrafiltration [19] and membrane distillation membranes [17]. HA comprises both aromatic and aliphatic compounds with three major functional groups commonly found including carboxylic acids, phenolic alcohols and methoxy carbonyls, amongst many others [18].

A PBM consists of thermodynamically stable continuous and interconnected domains of immiscible materials (typically oil and water) separated by a monolayer of polymer surfactant, as opposed to a simple emulsion which consists of disassociated droplets of one material suspended in another to which it is immiscible [20,21]. PBMs are of increasing interest due to their possible application in catalysis, use as drug delivery systems as well as coating materials in separation processes [22]. This latter includes use as a coating for surface modification previously fabricated membranes to improve their performance through surface modification [23–25]. Recently Galiano *et al.* [1] studied the modification of commercial membranes using PBM layers and found them to produce smooth and relatively hydrophilic surfaces which showed lower biofouling fouling than the unmodified membranes.

This work follows on from the experiments reported by Galiano *et al.* [1]. We have developed a novel colloidal probe consisting of humic acid covalently attached to a polystyrene latex microsphere, to serve as an analogue for organic fouling particulates. The membranes of interest are a number of membranes created from the PBM modification of a commercial PES membrane under slightly different conditions. The major objective of this work is to use these humic acid functionalised colloidal probes to assess the comparative adhesion forces between different PBM preparations both in water and in a model textile dye wastewater, to study the effects of foulant exposure on membrane and foulant behaviour.

2. Material and methods

2.1. PBM membrane preparation

Commercial membranes were PES ultrafiltration membranes manufactured by Microdyn-Nadir (Germany). Other membranes were prepared by production of a polymeric bicontinuous microemulsion as a top layer onto the commercial PES membrane. The fabrication of these PBM membranes has been described by Galiano *et al.* [1], with a schematic of their basic structure outlined in Fig. 1. The basic composition of the microemulsions used to create the PBM modified membranes examined in this study includes:

- 1. Methyl methacrylate (MMA), comprising the oil phase of the microemulsion.
- 2. 2-hydroxylethyl methacrylate (HEMA).
- 3. Water, comprising the aqueous phase.
- 4. The zwitterionic surfactant acryloyloxyundecyltriethyl ammonium bromide (AUTEAB) – used to decrease the surface tension of the microemulsion.
- 5. Ethylene glycol dimethacrylate(EGDMA) cross-linking agent.
- 6. Ammonium persulfate (APS) and N,N,N',N'-tetramethylethylenediamin (TMEDA).

For PBM membrane preparations A, B, and C, the chemical compositions were identical, with changes to preparation temperature only. The chemical composition of these samples and



Fig. 1. Schematic cross section of a PES membrane modified with a PBM layer, showing the basic structure of the modified membranes.

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