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Improving performance in algal organic matter filtration using polyvinylidene fluoride–graphene oxide nanohybrid membranes

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ABSTRACT

This study investigated the characteristics of various graphene oxide (GO) nanohybrid membranes and their performance in algal organic matter (AOM) filtration. The membranes were fabricated by phase inversion method. The effect of GO and its nanohybrids embedded in membranes was investigated in terms of wettability, porosity, pore size, surface charge, composition, morphology, permeability, fouling resistance and antimicrobial ability. In addition, the rejection of protein and carbohydrate as critical foulants in AOM was studied. Based on the findings, all the composite membranes showed lower flux decline than PVDF membrane. Composite membranes maintained higher protein (81–86%) and carbohydrate (77–83%) rejection compared with PVDF membrane (64% for protein and 63% for carbohydrate). However, the reversible to irreversible fouling ratio of PVDF, ZnO/GO-PVDF, Ag/GO-PVDF and GO-PVDF membranes was 3.07, 1.53, 0.86 and 1.09, respectively. This scenario implied that more hydrophilic substances in small molecular weight (MW) contained in AOM had plugged the composite membranes' pores and resulted in irreversible fouling. On the other hand, ZnO/GO-PVDF and Ag/GO-PVDF membranes exhibited superior antimicrobial ability and showed great potential in anti-biofouling mitigation.

1. Introduction

The scarcity of global water and energy supply has increased scientists' concern to search for solutions to achieve sustainable global development. Algal biofuel has been identified as a potential alternative to replace fossil fuel in energy generation via the production of biohydrogen from its biomass [1]. Therefore, microalgae have been widely cultivated and harvested for this purpose. As microalgae cultivation consumes a large amount of nutrients like nitrates and phosphates, the idea of cultivating microalgae in nutrient-rich wastewater seems promising. This integration not only produces algal biomass for biofuel, but also recovers nutrients from the wastewater [2,3]. On the other hand, eutrophication in water sources like seas, lakes and rivers results in frequent algal blooms. The occurrence of red tides (harmful algal blooms) in the ocean has severely affected the operation of desalination plants [4]. The algal biomass could enter water treatment plants, causing serious fouling on membranes and deteriorating the treated water quality. Toxins produced by these microalgae could be retained in treated water and have chronic effects on human beings [5,6]. Therefore, the complete removal of microalgae and algal organic matter (AOM) should be the focus in the development of filtration technology in water treatment plants.

Various separation methods have been employed in the harvesting of microalgae in algal ponds as well as separating microalgae from the water source in municipal water treatment plants. Most common are sedimentation, centrifuge and dissolved air filtration (DAF) [1,7]. However, centrifugation consumes a lot of energy while sedimentation and DAF require a higher footprint in large-scale application. Thus,

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membrane technology is greatly preferable in algal biomass harvesting and water treatment due to its relatively small footprint, low energy consumption and high quality product [8]. A few studies were performed in recent years to investigate the characteristics of microalgae species and their impact on membrane performance to achieve complete removal of microalgae in the filtration process. Qu et al. [9] characterized the microalgae solution as dissolved extracellular organic matter (dEOM) and bound extracellular organic matter (bEOM). The dEOM comprised humic-like substances, polysaccharide and protein with mainly hydrophilic compounds. Meanwhile, bEOM consists of only protein with a few polysaccharides, hence it is more hydrophobic. Huang et al. [10] observed that hydrophilic substances were dominant during algal blooms while hydrophobic biopolymer was dominant in the absence of algal blooms in Taihu Lake, China. Furthermore, most of the hydrophilic substances could be removed in the coagulation process, but the rejection of hydrophobic biopolymer was low and hence resulted in severe membrane fouling. In addition, small and medium MW fractions of protein and carbohydrate have been identified as the critical foulants in the algal filtration process [10,11]. Marbelia et al. [12] found that hydrolyzed PAN membrane with negative zeta potential exhibited a higher permeation flux in microalgae filtration in comparison to neutral charge PAN membrane with the same pore size. This is because the surface charges of microalgae suspension are always negative; the electrostatic repulsive force existing between the membrane and microalgae reduced the contact between the microalgae and the membrane surface. Hence, membrane fouling was successfully mitigated [12].

Modification of the treatment process has been performed to reduce the membrane fouling in algal suspension filtration. The effectiveness of various pretreatment processes was tested to remove large MW organic matter before entering membrane filtration. Yan et al. [13] removed fluorescent components of EOM with the aid of permanganate ($KMnO_4$)-aided aluminium (Al) coagulant. Meanwhile dosing of manganese oxide (MnO_2)-aided Al coagulant showed the greatest improvement in both membrane permeability and irreversible fouling by retaining in situ formed MnO_2 particles on the membrane surface, which adsorbed the hydrophilic fraction of EOM. Another study found that pre-ozonation showed a limited membrane fouling mitigation effect as the ozonation broke down large MW compounds into a smaller fraction, at the same time oxidizing the hydrophobic biopolymer to hydrophilic, which contributed to irreversible fouling [14]. The axial vibration ultrafiltration (UF) membrane system increased the critical flux significantly in a submerged membrane filtration system due to the existence of a long-range repulsive force which prevented the deposition of microalgae on the membrane surface [15].

de Baerdemaeker et al. [16] identified PVDF as the most suitable membrane for microalgae filtration as it possesses higher permeability and fouling resistance compared with PVC and PES-PVP membranes. Hwang et al. [17] coated PVDF membrane with PVA to increase the membrane hydrophilicity. As a result, the permeation flux of algal suspension improved by 36% and the fouling events were retarded due to the lesser contact of foulants with the membrane surface. Besides, a PVDF hollow fibre membrane embedded with TiO_2 nanofillers was also applied in an integrated algae cultivation and wastewater polishing system [3]. As the membrane surface hydrophilicity increased, the fouling resistance improved by 50% compared with the control membrane. In another study by Venault et al. [18], parameters like flux decline, flux recovery rate, reversible fouling and irreversible fouling of bovine serum albumin (BSA) and microalgae filtration showed great improvement when PVDF/PS-b-PEGMA membrane with an improved hydration property was applied. Although the overall throughput of the modified membranes in microalgae filtration was improved, the filterability of dissolved algal organic matter (dAOM) and the rejection of protein and carbohydrate as the main foulants with hydrophilic membranes were not investigated.

In recent years, GO incorporated membranes have received much attention due to their high reliability and outstanding filtration performance [19,20]. Zhao et al. investigated the reliability and performance of GO-PVDF membrane in a submerged MBR system [21]. The composite membrane not only increased the critical flux up to 50%, but also reduced the cake thickness of the composite membrane to 80 μm, which was lower than the PVDF membrane. Furthermore, the tightly and loosely bound polysaccharides content of GO-PVDF membrane was five times lower than PVDF membrane. As a consequence, the filtration of GO-PVDF membrane could last 3 times longer and was eventually able to lower the system operating cost. Recently, metallic oxides such as ZnO [22], TiO_2 [23] and Ag [24,25] were impregnated on GO, forming nanohybrids to enhance both the membrane's permeability and its antimicrobial ability. As GO and its nanohybrids possess an abundance of oxygen functional groups, the addition of these fillers will increase the hydrophilicity of the PVDF membranes and at the same time increase the membranes' overall negative surface charge. However, the characteristics and performance of these composite membranes have not been compared.

This is the first study to investigate the potential of PVDF membranes blended with GO and GO nanohybrids in microalgae filtration. The physical characteristics of the composite membranes in terms of their hydrophilicity, permeability, surface charge, elemental composition and morphology were first compared. AOM which contained protein and carbohydrate as the main foulants in the microalgae solution was extracted and used as the feed solution to investigate the membranes' filtration performance. As the characteristics of PVDF membranes were altered with the addition of different fillers, the effect of the surface charge and hydrophilicity of the membranes on AOM filtration, and protein and carbohydrate rejection were investigated. Lastly, the membranes' antimicrobial properties were compared in order to study their biofouling mitigation ability.

2. Experimental

2.1. Materials

Fine graphite (50 μm, Merck Co.), potassium permanganate ($KMnO_4$, Accot), sulphuric acid (H_2SO_4, Accot) and hydrogen peroxide (H_2O_2, Sigma Aldrich) were used for the synthesis of GO using the Hummers method [24]. Silver nitrate ($AgNO_3$, Sigma Aldrich) and sodium borohydride ($NaBH_4$, Sigma Aldrich) were used for the synthesis of Ag/GO nanohybrids. Zinc nitrate ($Zn(NO_3)_2$, 98%, VWR), polyvinylpyrrolidone (PVP, 160,000 g/mol, R & M Chemicals) and sodium hydroxide (NaOH, Merck) were used to synthesize ZnO/GO nanohybrids. Sodium hypochlorite (NaOCl, John Kollin) was used for membrane cleaning.

For membrane solution preparation, *N*,*N*-dimethylacetamide (DMAc, ≥ 99.5%, Sigma Aldrich) was used as solvent to dissolve the PVDF (Solef 6010, Solvay) powder. Microalgae (*Chlorella vulgaris*) were cultivated in culture media (initial pH 7) [26] for two weeks in an airlift photobioreactor, under a 55 W fluorescent lamp intensity of 6500 Lux on the surface of the photobioreactor with 3 L/min aeration using an air pump. Sartorius arium pro ultrapure water was used throughout the experiment.

2.2. Synthesis of GO, Ag/GO and ZnO/GO nanohybrids

The synthesis of GO and Ag/GO nanohybrids and their characteristics were described in detail in a previous study by Mahmoudi et al. [24].

For synthesis of ZnO/GO nanohybrids, initially 0.2 g of GO powder was dispersed in absolute ethanol (C_2H_5OH, 99.8%, Systerm). The powder was stirred rigorously with a magnetic stirrer followed by 30 minute sonication to achieve a homogeneous solution. $Zn(NO_3)_2$ (10 wt% of Zn with respect to GO) with dissolved PVP was added slowly into the GO solution. After rigorous stirring for 1 h, 0.20 M of

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