

PREPARATION AND APPLICATION OF STIMULI-RESPONSIVE PET TEMS FOR WATER-OIL EMULSION SEPARATION

Oil's pivotal role in the global economy is undeniable, yet its extraction, processing, and utilization have significant environmental repercussions, including the formation of water-oil emulsions. These emulsions pose threats to ecosystems and human health, prompting the need for effective remediation methods [1]. Traditional methods like flotation, coagulation, and extraction have limitations, highlighting the necessity for advanced separation technologies. Membrane technology, particularly using modified poly(ethylene terephthalate) (PET) track-etched membranes (TEMs), offers promising solutions due to their precise pore size control and stability [2].

In this study, pH-sensitive membranes were obtained by UV-initiated RAFT graft copolymerization of hydrophobic styrene (ST) and hydrophilic acrylic acid (AA) on the surface of PET TEMs. Comprehensive characterization was conducted using AFM, SEM-EDX, TGA, FTIR, and CA measurements.

AFM and SEM-EDX analysis revealed a uniform grafting layer and the pore diameter decreases from 1.65 ± 0.08 to 1.51 ± 0.04 μm , while TGA indicated the thermal stability from 380 °C of the grafted membranes. FTIR spectra showed characteristic peaks corresponding to PS and PAA, confirming the chemical composition of the grafted layers. Absorption peaks of PS: for bending C-H out-of-plane 699 cm^{-1} and 759 cm^{-1} , for stretching C=C aromatic 1601 cm^{-1} , 1492 cm^{-1} and 1452 cm^{-1} . After PAA grafting, the intensity of the peak corresponding to the absorption of C=O groups increases and shifts to 1713 cm^{-1} . Also, the broadening of the peak at 2845 cm^{-1} (PS-g-PET TEMs) corresponding to the stretching of C-H aliphatic groups was observed at 2862 cm^{-1} after PAA grafting.

The pH sensitivity is due to the presence of carboxyl groups of PAA, which contribute to the contact angle (CA) in response to changes in the environmental pH above or below the pKa of PAA (4.8). pH responsiveness is pronounced at: distance from the UV source - 10 cm, molar ratio of RAFT agent to initiator - 1:10, monomer concentration - 430 mM, irradiation time - 60 minutes. In a basic environment at pH 9, the membrane surface is negatively charged, which leads to the straightening of PAA chains in the composition of PET TEMs-g-PS-g-PAA, consequently decreasing the CA to 65°. Water with pH 9 begins to seep through the pores of the membrane while preventing the penetration of oil. In an acidic environment at pH 2, the CA increases to 97° as carboxyl groups are not ionized, and PAA remains in a coiled state, exposing the hydrophobic links of PS. Consequently, oil passes through the pores, and water with pH 2 is retained on the membrane surface.

PET TEMs-g-PS-g-PAA with switchable hydrophilicity/hydrophobicity were successfully tested in the separation of several water-in-oil emulsions with efficiency of more than 90% and a flux rate of 2500 $\text{L m}^{-2} \text{h}^{-1}$ for direct emulsions and 1700 $\text{L m}^{-2} \text{h}^{-1}$ for reverse emulsions. The membranes exhibit antifouling properties, with a 96% chloroform flux recovery and only a 12% decrease in total flux reduction factor during water-in-chloroform reverse emulsion separation.

Thus, in this work, the properties of moderately hydrophilic PET TEMs have been enhanced by a simple method leading to controlled wettability by changing only the pH of the environment. These membranes show potential for the separation of two types of water-oil emulsions.

References:

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