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Preparation of a Hollow Fiber Membrane Made of Antifouling PVDF/Zeolit using the Dip-Coating Technique

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Abstract. The practical technique for membrane modification is dip coating. This study coats a PVDF hollow fiber membrane-based composite with a coating of zeolite. The composite is made of a polyvinylidene fluoride (PVDF) membrane. During water filtering, the separation capabilities and propensities of composite membranes for organic impurities were examined. SEM and Fourier-transform infrared spectroscopy analysis of the findings demonstrated that the Zeolite coating was successfully deposited on the PVDF membrane. The flux recovery ratio increases from 69% to 80% while the relative flux drop decreases from 63% to 50%. A composite PVDF membrane dip-coating of Fe_2O_3/Zeolite with a GA and H_2SO_4 ratio of 1:2 is needed to remove about 75% of humic compounds from effluent. The results of this study show that the addition of Fe_2O_3/Zeolite with a GA and H_2SO_4 layer can greatly improve the hydrophilicity, selectivity, and anti-organic fouling of the PVDF hollow fiber membrane.

Keywords: *fouling, PVDF, dip-coating, membrane performance.*

1. Introduction

The industry has grown exponentially as a consequence of rising population growth, creating environmental issues and driving up demand for clean water [1-4]. The biggest obstacles to sustainable growth in the 21st century are water supply and waste management shortages. According to projections, the world's water consumption will rise from 4600 km^3/year to 6000 km^3/year by 2050, which will likely result in more wastewater being generated [5]. The membrane bioreactor is a substitute for conventional wastewater purification technology. (MBR). Microfiltration (MF) and ultrafiltration (UF) are two common membrane

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filtration techniques used in MBR [4, 6, 7]. MBR eliminates pathogens, organic matter, and suspended solids, and removal of COD (up to 90%) and TOC (more than 80%) while producing effluent with high effluent and biomass concentration, a smaller environmental impact, less reactor volume, and less sludge production [8–9].

Due to its excellent thermal stability, chemical resilience, and membrane-forming capacity, PVDF is a widely used material [3, 10, 11]. In microfiltration and ultrafiltration, PVDF membranes are frequently employed [12]. The semi-crystalline polymer PVDF, on the other hand, has repetitive units of -CH_2-CF_2, which can produce hydrophobic structures that make the membrane more prone to fouling [3, 12]. Hydrophobic species in fluids cause blockage, which lowers membrane permeability. A buildup of activated sludge can also shorten the lifespan of the membrane and increase running costs [3, 13]. Blockages may be both reversible and permanent [7, 14]. Reversible fouling is caused by contaminants that adhere to the membrane's surface, but persistent fouling is brought on by contaminants that securely attach to the membrane's pores [15]. Therefore, more effective antifouling membranes for MBR applications must be developed, as well as changes with the addition of hydrophilic component enhancements [12–13].

The membrane modification method aims to engineer the membrane's surface to make it more hydrophilic, increasing the hydrophilicity, antibacterial properties, and performance of the membrane and producing more effective wastewater treatment results [2, 16]. A few modification techniques include coating, grafting, covalent coupling, irradiation, plasma treatment, coating adsorption, and coating [16]. The coating approach, which also has a straightforward procedure and is less expensive, is the way that is most adaptable [2]. The substrate surface is coated with a coating solution (liquid phase) in the dip-coating technique, which is then applied to another surface and dried [17]. The method of dip-coating allowed the PVDF surface to have a maximum permeate flow and hydrophilic qualities. The dip-coating method is easy to use, has high efficiency for industrial applications, and does not require special conditions (high pressure and temperature) [2, 18]. In some earlier studies, coating techniques employing polydopamine (PDA), titanium dioxide and silver nanoparticles (TiO_2-NP and Ag-NP), thin-film nanofibrous composite-cellulose nanofiber (TFNC-CNF), and other materials were also employed [3, 16, 19]. One of the most well-liked and effective methods is the inclusion of inorganic nano- and micro-particles, such as Al_2O_3, SiO_2, and several compounds with polar groups [2, 10, 20, 21].

According to Evangeline's 2018 study [22], adding iron oxide nanoparticles can reduce the low flux produced by PES and CA membranes. Iron oxide serves as an absorbent for ionic pollutants, is biocompatible, has minimal toxicity, and helps to maintain the membrane's mechanical stability [22–23]. According to a study by Hafizah et al. from 2019 [24], the mixed matrix membrane had more pores and was clearer than pure PVDF membranes. Demirel et al. 2017 found that adding Fe_2O_3 to PVC causes flow and FRR to be 782 L/m^{2}h and 91.5 percent higher than when Fe_2O_3 is not added [25]. Adding Fe_2O_3 to the mixed PVDF/membrane matrix results in fluxes of 46.72 L/m^{2}h and 33.18 L/m^{2}h, respectively, according to Hafiza et al. 2019 [24].

Aluminum (Al), silica (Si), and oxygen are the molecular constituents of zeolite, an inorganic crystal. (O). It enhances the surface area for the development of biofilms and has high adsorption and ion exchange capacities as well as the capacity to filter molecules and recycle waste [26–27]. Zeolites have many commercial applications, including the separation of linear from non-linear hydrocarbons, the decrease of excess ammonium, gas adsorption, the removal of heavy metals, and water softening [28]. The material of choice for membranes with superior oleophobicity and heavy metal ion adsorption capabilities is natural zeolite, a porous aluminosilicate mineral with exceptional ion exchange capabilities [21]. Produced the FRR, RIrr, and RRev flows in the PSf matrix at rates of 8.4 L/m^{2}h, 40%, 84%, and 2 L/m^{2}h, 41%, 78%, and 19.5%, respectively [29]. Vatanpour et al. (2016) found that adding SAPO-34 to the PVDF membrane resulted in fluxes known as RRev (47 kg/m^{2}h, 66.4%, 24.98%, and 7) and fluxes known as FRR, RIrr, and RRev (63.5 kg/m^{2}h, 89.4%, 7%, and 19%) [30].

The surface of the PVDF hollow fiber membrane will be altered using a dip-coating of Fe_2O_3/Zeolite in this research. In addition, analyses of the membrane's performance (flux and antifouling) as well as tests on the quality of the treated water were performed. The modified membrane is then applied to industrial and municipal wastewater treatment systems.

2. Materials and Methods

2.1 Materials

Materials used in this research included distilled water, 70% alcohol, Fe_2O_3, glutaraldehyde (GA), 98% H_2SO_4, hollow fiber membrane (PVDF), poly (vinyl alcohol) (PVA), and zeolite. The tools used in this research included stir bars, glass funnels, beaker glasses, measuring cups, glassware, volumetric flasks, digital balances, ovens, glass plates, tongs, tweezers, pipettes, spatulas, ultrasonicators ROHS-CSBJZQFS-150N0001V2.

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2.2 Solvent Preparation for Coatings

100 millimeters of distilled water were prepared with 0.15 gr of PVA. When the mixture was homogeneous, the components were combined and stirred with a hot plate stirrer at a speed of 200 to 300 rpm [34, 35]. The mixture was then stirred for 30 minutes using a hot plate stirrer before Fe_2O_3 and Zeolite were added to it in four amounts (0.1, 0.2, 0.3, and 0.4 g). Then the solution was ultrasonically sonicated for 30 minutes to produce a consistent Fe_2O_3/Zeolite dispersion [3]. The membranes were immersed in the dope solution for 5 hours and then desiccated at room temperature.

2.3 Crosslinking Solutions Creation

Use 0.5 gr of GA, 100 ml of distilled water, and 5% prepared H_2SO_4 for each component. Once homogeneous, the mixture is stirred and mixed once more. To catalyze the reaction, H_2SO_4 is introduced [36]. The cross-linking solution was made by mixing GA and H_2SO_4 according to the proportions. (1:1; 1:2; and 2:1). The desiccated membrane was then immersed in the crosslinking solution for two minutes. The membrane was placed on a glass plate and dried in a 45 °C oven for two hours. Crosslinking solutions aim to maintain PVA's stability in the aqueous phase by incorporating elements that may reduce PVA's solubility in water (like GA) and increase the tensile strength of thin film composites [33].

2.4 Performance of Membranes

Flux Recovery Ratio (FRR) analysis:

$$FRR = \frac{Jr}{Jw} \times 100\%$$

Where (Jr) is water flux following a fouling test and (Jw) is pure water flux [25]. The FRR calculation aims to determine the membrane's antifouling capacity [29].

Analysis of Fouling Resistance:

$$R_{Rev} = \frac{(Jp - Jr)}{Jw} \times 100\%$$

Fouling resistance analysis was carried out by computing the total R, RRev, and RIrr. The reversible blockage fraction (RRev) is obtained from the following equation [30.35]. The irreversible blockage fraction (R_{Irr}), however, employs the following formula:

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$$R_{Irr} = \frac{(Jw - Jr)}{Jw} \times 100\%$$

The level of total flow loss due to obstruction (RTtotal) can be computed using the following equation:

$$R_t = \frac{(Jw - Jp)}{Jw} \times 100\%$$

3. Result and Discussion

3.1. FTIR analysis

Analysis of chemical properties and identification of functional groups was carried out by the FTIR test shown in Figure 1. Several additional peaks were shown by the composite membrane compared to the PVDF membrane. In the range of 2000-3000 cm^{-1}, asymmetric C=O and CH_2 functional groups were formed which indicated the presence of Fe_2O_3 and Zeolite attached to the surface of the membrane. The range of 1100-1200 cm^{-1} indicates the presence of sulfonate groups in the composite membrane, while the peak of 1280 cm^{-1} indicates the presence of PVDF material. The IR spectrum on the composite membrane showed that Zeolite and Fe_2O_3 were successfully coated on the surface of the PVDF membrane. In addition, the results also show that through the dip-coating method, a composite UF membrane can be produced [3].

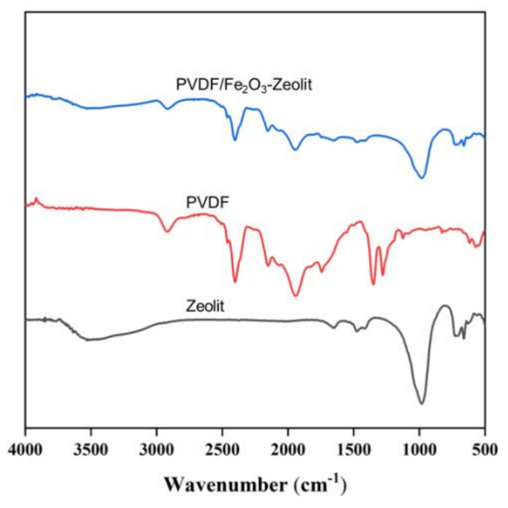

Figure 1. FTIR spectrum of composite PVDF membrane

3.2. SEM analysis

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Figure 2 shows the SEM image of the modified PVDF membrane. The surface of the PVDF membrane is depicted in Figure 2 (a). PVDF membrane showed a new coating on its surface after being coated with Zeolite and Fe_2O_3. Figure 2 (b, c, and d). SEM image of the PVDF membrane with a 1:1 ratio of GA and H_2SO_4 can be seen in Figure 2(b). As shown in Figure 2(c) there is a 1:2 ratio between GA and H_2SO_4. A 2:1 comparison of GA and H_2SO_4 is shown in Figure 2(d). The surface formation may be relatively denser because there is more available H_2SO_4 than GA. The surface densities appear to be more similar to the membrane ratio of GA and H_2SO_4 which is 1:1.

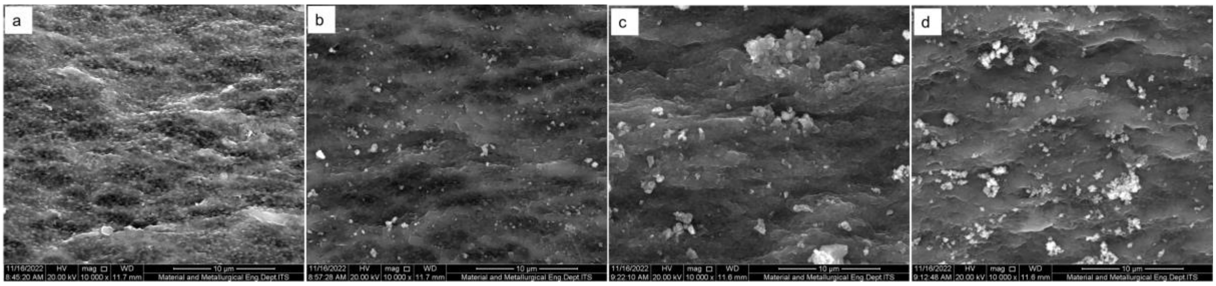

Figure 2. SEM image with ratio of GA and H_2SO_4 (a) 0, (b) 1:1, (c) 1:2, and (d) 2:1

3.3. Membrane performance

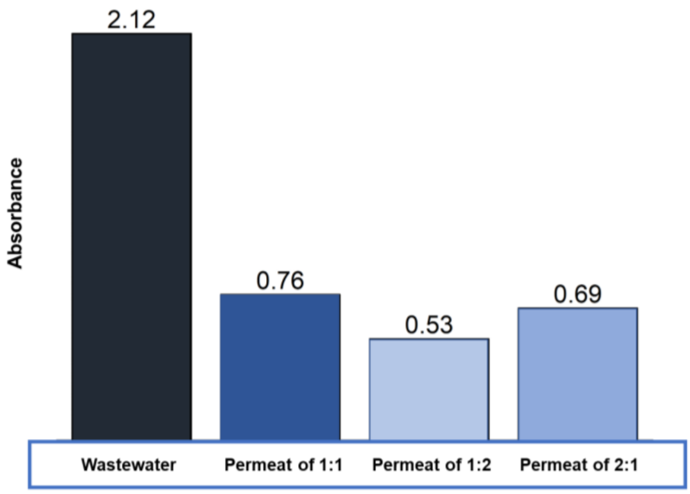

Figure 3. Performance of Composite Membranes for Wastewater Filtration at a Wavelength of 254 nm, Wastewater Absorption, and Permeate Ultrafiltration

Composite membranes are applied in industrial wastewater filtration to assess separation characteristics and the presence of organic impurities. Figure 3 shows the decrease in the amount of humic compounds is associated with a decrease in the absorbance value. Composite membranes can achieve a lower absorbance of up to ~75%. Based on this, it can be stated that the composite membrane can remove around ~ 75% of humic compounds contained in wastewater. The ability of the composite membrane to purify wastewater can also be seen in. The removal of humic compounds increased dramatically in composite membranes with a ratio of GA and H_2SO_4 (1:2) compared to membranes without modification. Likewise, the composite membrane with a ratio of GA and H_2SO_4 (2:1) has the ability to remove almost the same humic compounds as in Figure 3. These findings indicate that the combination of Zeolite and Fe_2O_3 can produce better permeability and selectivity.

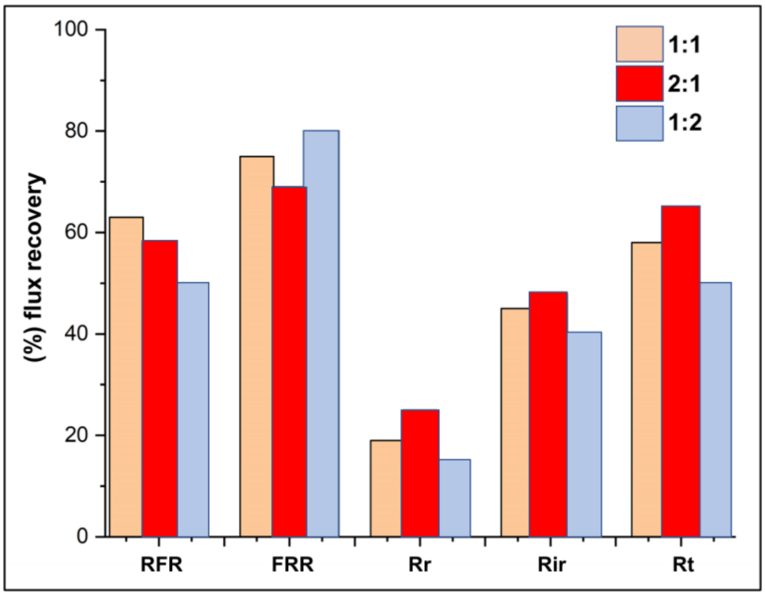

Figure 4. Blockage Parameters

Figure 4 shows the blocking parameters of the PVDF membrane that has been coated with Fe_2O_3/Zeolite with crosslinker ratios in the form of GA and H_2SO_4 of 1:1, 1:2, and 2:1. PVDF membrane that has been coated with Fe_2O_3/Zeolite with a ratio of 1:2 has a higher FRR value compared to 1:1 and 2:1. However, membranes with a 1:2 ratio have lower clogging parameters (RFR, Rir, Rr, and Rt) than 1:1 and 2:1. Foulant on the surface of the PVDF membrane with a 1:2 ratio of GA and H_2SO_4 is easier to remove because the adhesion of organic matter is lower than that of 1:1 and 2:1 ratios. The hydrophilic membrane surface allows interaction between the membrane surface and less foulant. Therefore, the addition of

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hydrophilic Fe_2O_3/Zeolite can increase the anti-organic fouling on PVDF membranes that have been modified using the dip-coating method with a 1:2 ratio of GA and H_2SO_4.

4. Conclusions

Based on the research that has been done, the tendency of clogging by organic substances on the surface of the composite PVDF membrane is reduced. FTIR and SEM test results prove that the dip-coating method was successfully used to coat the surface of the PVDF membrane with Zeolite/Fe_2O_3. Fe_2O_3 particles increase the hydrophilic properties of the membrane so that greater hydrophilicity can reduce the accumulation of clogging organic matter on the surface of the membrane during wastewater treatment (FRR: from 69% to 80% and RFR: from 63% to 50%). In addition, the modified membrane using the Zeolite/ Fe_2O_3 dip-coating method with a 1:2 ratio of GA and H_2SO_4 was able to remove humic compounds in wastewater about 75%.

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