

Mixed matrix membranes for dye removal

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Abstract. Mixed matrix membranes (MMMs) can be a promising alternative for the solution of dye removal from coloured effluents. Polymeric membranes are widely used due to their good film-forming ability, flexibility, separation properties, and cost. However, they have low mechanical, chemical, and thermal resistances. Moreover, the fouling of polymeric membranes is high because of their hydrophobic nature. Hence, there is an increasing interest in organic-inorganic hybrid membranes as a new-generation membrane material. It has been shown that carbon nanotubes have the potential to increase the material properties of polymers with their low density, high strength, hardness, and exceptional aspect ratio. In this work, carbon nanotubes blended MMMs were prepared and methyl orange removal efficiency of them was investigated. Compared to the bare membranes, MMMs showed not only increased hydrophilicity, water content, and pure water flux but also increased methyl orange rejection and flux recovery

Keywords: carbon nanotubes; coloured effluent; dye removal; methyl orange; mixed matrix membranes (MMMs); nanofiltration

1. Introduction

One reason for groundwater pollution is industrial effluents containing dyes and heavy metal ions (Azimi *et al.* 2023). A large amount of coloured effluent is generated in the textile industry, which raises concerns about dye removal (Lau and Ismail 2010, Rashidi *et al.* 2020). Due to the lower cost, membrane-based separation techniques are widely used in dye removal from coloured effluents (Hassan *et al.* 2018, Reddy *et al.* 2022).

Membrane phenomena was first observed in the 18th century. Even though the elements of modern membrane science were developed in the 1960s, they were used only on a laboratory scale. At first, their cost, unreliability, and lack of selectivity made membranes impractical for widespread use as a separation process (Baker *et al.* 1991). Today, membrane filtration is widely used for water treatment because it is simple, efficient, and cost-effective (Bodzek *et al.* 2022, Yu *et al.* 2022). Among membrane processes, pressure-driven membrane processes are widely used in real-life applications (Yanar *et al.* 2021).

Polymeric membranes are widely used because of their good film-forming ability, separation performance, flexibility and low cost (Park *et al.* 2020). In addition, polymers are highly stable and have a low chemical interaction with the pollutants (Sepahvand *et al.* 2023). However, the thermal, mechanical and chemical resistances of polymeric membranes are low. Moreover, they tend to foul due to their relatively hydrophobic nature (Park *et al.*

2020). Fouling occurs because of the complex physical, chemical, and biological interactions between the membrane surface and the contaminants (Arhin *et al.* 2016). Therefore, there is growing interest in using organic-inorganic hybrid membranes as a potential future material.

Organic-inorganic hybrid membranes are expected to have the physicochemical stability of inorganic materials and the film-forming properties of polymers (Peng *et al.* 2007). Particularly, there is an increasing interest in membranes with nano-sized inorganic materials due to their properties, such as high permeability, hydrophilicity, high rejection rates and fouling resistance (Yang *et al.* 2007, Alambi *et al.* 2023). It has been shown that carbon nanotubes (CNTs) have a high potential to improve the material properties of polymers, with their exceptionally high aspect ratio, low density, high strength and stiffness (Gojny *et al.* 2004). CNT blended mixed matrix membranes (MMMs) exhibit high permeability, thermal stability, mechanical strength, and fouling resistance (Qu *et al.* 2013).

Choi and his colleagues (Choi *et al.* 2006) and Qiu and his colleagues (Qiu *et al.* 2009) showed that the morphology and filtration properties of CNT blended polysulfone MMMs depend on the amount of CNTs. Nechifor and his colleagues (Nechifor *et al.* 2009) showed successful removal of lead and mercury with CNT blended polysulfone MMMs. Celik and her colleagues (Celik *et al.* 2011a, b) demonstrated low protein adsorption and high fouling resistance of CNT blended polyethersulfone MMMs. According to the studies by Zhao and his colleagues (Zhao *et al.* 2014) and Kim and his colleagues (Kim *et al.* 2014), CNT blended polyamide MMMs have high salt removal performance and fouling resistance. Wang

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Table 1 Compositions of the MMMs prepared

Membrane name	Polymer ratio (wt %)	CNT ratio (wt %)	NMP ratio (wt %)
NF-C-0	25	0	75
NF-C-0.2	25	0.2	75
NF-C-0.5	25	0.5	75
NF-C-1	25	1	75

and his colleagues (Wang *et al.* 2015) demonstrated high permeability and salt removal of CNT blended polyethersulfone MMMs for nanofiltration. According to studies by Yang and his colleagues (Yang *et al.* 2016) and Celik Madenli and her colleagues (Celik Madenli *et al.* 2021), CNT blended polyethersulfone MMMs showed high antibacterial activity and suppressed biofilm growth. Studies have revealed CNT blended MMMs have improved properties like hydrophilicity, permeability, selectivity, and fouling and biofouling resistance. (Liu *et al.* 2014, Wang *et al.* 2015, Choi *et al.* 2016, Yang *et al.* 2016, Celik-Madenli *et al.* 2017, Celik Madenli and Cakmakci 2017, Celik Madenli and Ciftci 2022).

The aim of this study is to prepare CNT blended polyethersulfone MMMs to examine the performance of dye removal in coloured effluents. For this purpose, MMMs were prepared with different CNTs concentrations and characterisations were made using water contact angle, water content, pure water flux, and Fourier transform infrared (FTIR) spectroscopy. In addition, methyl orange removal tests were applied to determine the dye removal performance of the prepared MMMs.

2. Materials and methods

CNTs were functionalized by the method we reported in our previous studies (Celik *et al.* 2011a, b, Celik Madenli *et al.* 2017, 2021, Celik Madenli and Cakmakci 2017, Celik Madenli and Ciftci 2022). Briefly, CNTs were subjected to ultrasonication for 9 hours in a 3:1 ratio of HNO₃:H₂SO₄ acid mixture. After ultrasonication, CNTs were washed with distilled water to neutral pH and dried at 100 °C.

MMMs were fabricated by using the method described in our previous publications (Celik *et al.* 2011a, b, Celik Madenli *et al.* 2017, 2021, Celik Madenli and Ciftci 2022). Briefly, CNTs were sonicated in n-methyl-2-pyrrolidinone before dissolving 25 % polyethersulfone. Afterwards, the membrane solution was ultrasonicated to remove air bubbles before pouring it onto the glass plate using a casting knife. The glass plate was then quickly immersed in the coagulation bath prepared with distilled water. Membranes separated from the glass plate in the coagulation bath were washed with and stored in distilled water at 4°C for further testing. The compositions of the MMMs are given in Table 1. Note that MMMs marked as NF-C-0.2 refer to MMMs prepared in a casting solution in which the content of the CNTs with respect to polyethersulfone was 0.2 % by weight.

The functional groups of the MMMs were examined by FTIR. The surface hydrophilicity of the MMMs was determined using a contact angle goniometer. Water contact angles reported are an average of at least seven measurements. The water content of the MMMs (E, %) was determined by weighing the wet and dry MMMs and calculated using Eq. (1).

$$E = \frac{W_w - W_d}{W_w} \times 100 \quad (1)$$

where W_w and W_d are the wet and dry weights of the MMMs (g), respectively. The water content reported is the average of at least two measurements.

A cross-flow membrane test unit comprising a pump, a relief valve, and a pressure gauge was used for filtration tests. Filtration tests were carried out in the recycling mode, where the filtrate and concentrate lines were returned to the feed tank. During the filtration tests, pure water filtration was carried out for 2 hours under 6 bar pressure. Flux (J) was calculated using Eq. (2).

$$J = \frac{V}{A\Delta t} \quad (2)$$

where V is the permeate volume (L), A is the active membrane area (m²), and Δt is the time (h).

Methyl orange (0.02 mM) filtration was performed for 2 hours at 5.5 bar pressure to determine the methyl orange rejection rate of the MMMs. After 2 hours of filtration, the rejection rate (R, %) was calculated using Eq. (3).

$$R = \frac{C_f - C_p}{C_f} \times 100 \quad (3)$$

where C_p (mg/L) and C_f (mg/L) are the methyl orange concentration of the permeate and feed solutions measured at 460 nm with the UV-Vis spectrophotometer, respectively.

Finally, the pure water flux was measured after the MMMs were back-washed with distilled water at a high cross-flow rate for 20 minutes. The flux recovery rate (FRR) after back-washing was calculated using Eq. (4).

$$FRR = \frac{J_f}{J_v} \times 100 \quad (4)$$

where J_f and J_v are the flux of the membrane after back-washing and the virgin membrane flux, respectively.

3. Results and discussion

In terms of membrane selectivity, nanofiltration lies between reverse osmosis and ultrafiltration. The only difference between nanofiltration and reverse osmosis is that the network structure is denser in reverse osmosis. Even though the monovalent ion (e.g. Na⁺ and Cl⁻) rejection is weak with nanofiltration, divalent ion (Ca²⁺ and CO₃²⁻) rejection is very high (Foundation *et al.* 1996, Mulder 1996). The rejection rate of the nanofiltration to low molecular weight organic pollutants is very high (Ge *et al.* 2017). Nanofiltration is used in different areas like

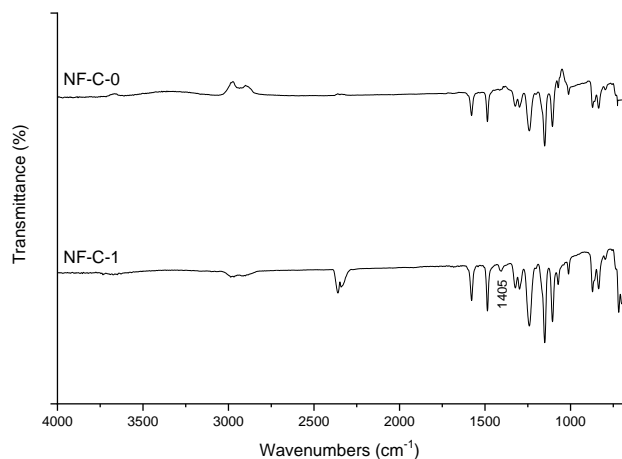


Fig. 1 FTIR spectra of the membranes

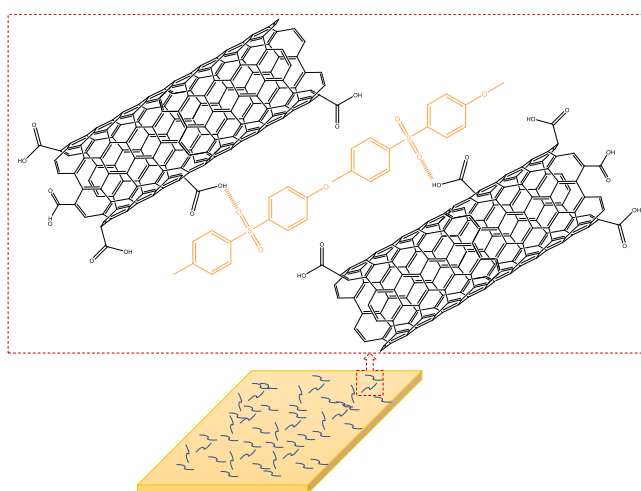


Fig. 2 The structure of polyethersulfone and CNTs

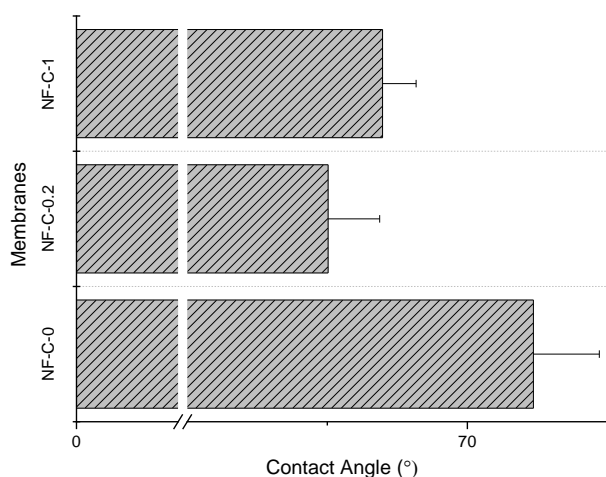


Fig. 3 Contact angles of the MMMs

removing pesticides, disinfection by-products, sulphate, and colour, and softening water (Cardew and Le 1998). In this study, functionalized CNT blended MMMs were synthesized and characterized. Then, methyl orange removal efficiencies were investigated in order to determine the efficiency of using these MMMs in coloured effluent treatment.

Table 2 Water contents of the MMMs prepared

Membrane	Water content (%)
NF-C-0	65.94 ± 1.03
NF-C-0.2	62.69 ± 1.46
NF-C-0.5	63.63 ± 0.23
NF-C-1	70.57 ± 0.70

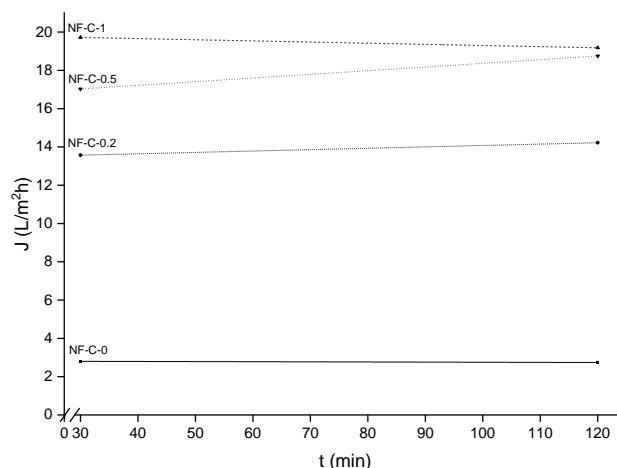


Fig. 4 Pure water flux of the MMMs

Fig. 1 shows the FTIR spectra of the fabricated MMMs. When the CNT blended MMMs are compared with the bare membrane, a new peak was identified at $\sim 1405 \text{ cm}^{-1}$ in the MMMs. This new peak corresponds to the carboxylic group (COOH) of the functionalized CNTs (Celik *et al.* 2011b). The formation of a new peak in MMMs shows a successful preparation of CNT blended MMMs.

Celik and her colleagues and Rong and his colleagues proposed that the CNT blended MMMs are formed by the hydrogen bonding interactions between the sulfonic groups of the polyethersulfone and the carboxylic groups of the functionalized CNTs (Fig. 2) (Rong *et al.* 2010, Celik *et al.* 2011b).

Contact angle measurements were used to determine the hydrophilicity of MMMs. The higher the contact angle, the more hydrophobic the surface. The lower the contact angle, the more hydrophilic the surface. Functionalized CNTs are hydrophilic due to the carboxylic groups. Fig. 3 shows the contact angle measurements of the MMMs. The presence of functionalized CNTs in the membrane increased its hydrophilicity. The functionalized CNTs likely moved to the membrane/water interface during the phase inversion process, which increased the hydrophilicity of the MMMs (Sun *et al.* 2010, Celik *et al.* 2011b).

The water content of the MMMs can be used as an indicator of membrane porosity. Table 2 shows the water contents of the MMMs. There is no statistical difference between the water contents of the NF-C-0, NF-C-0.2, and NF-C-0.5. However, 1% CNTs addition in the membrane solution caused an increase in the water content of the MMMs.

Fig. 4 shows the pure water flux of the MMMs. The pure water flux of the NF-C-0.2 membrane is five times

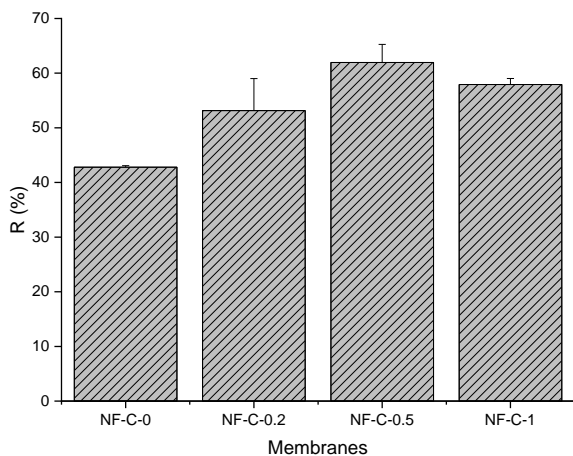


Fig. 5 Methyl orange rejections of the MMMs

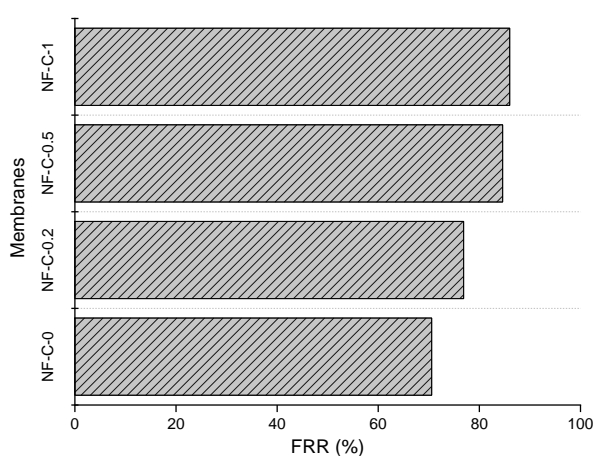


Fig. 6 Flux recovery ratios of the MMMs

higher than the pure water flux of the NF-C-0 membrane. In addition, the NF-C-0.5 and NF-C-1 membranes have seven times higher pure water flux than the NF-C-0 membrane. This is probably because of the increased hydrophilicity and porosity of the MMMs due to the addition of functionalized CNTs. This result is also consistent with the water content and hydrophilicity of the MMMs. More porous and hydrophilic MMMs exhibit higher filtration fluxes.

Methyl orange is a negatively charged dye with a molecular size of $17.93 \times 7.54 \times 6.02 \text{ \AA}^3$. The molecular weight of methyl orange is 327.34 g/mol (Jee *et al.* 2022). Hence, methyl orange was selected as a model dye to determine the filtration performance of the MMMs for coloured effluent filtration. Fig. 5 shows the methyl orange rejection performances of the MMMs. CNT blended MMMs provide approximately 25-45 % higher methyl orange removal than the bare membrane. Functionalized CNTs were negatively charged because of the carboxylic groups. CNT blended MMMs were also negatively charged because of the functionalized CNTs (Fig. 1). Hence, the addition of CNTs in the MMMs increased the methyl orange removal efficiency due to the electrostatic repulsion between the methyl orange and MMMs.

The increase in viscosity during membrane synthesis causes delayed phase separation, which causes smaller

pores (Han and Nam 2002, Celik *et al.* 2011b). The addition of CNTs in the membrane solution increased the viscosity of the membrane solution, which resulted in smaller pores. Even though ~40 % of the pores of the bare membrane were smaller than 1.79 nm, ~65 % of the pores of MMMs were smaller than 1.79 nm. The addition of functionalized CNTs to the MMMs led to higher rejection of methyl orange because of smaller pores and increased negative charge.

Flux recovery ratios were determined for determining the reusability of the MMMs. Pure water flux of MMMs after back-washing, which was used for methyl orange filtration, is given in Fig. 6. The flux recovery ratio of the bare membrane is about 70%, while the flux recovery ratios of NF-C-0.5 and NF-C-1 are about 85 %. CNT addition in the MMMs increased the flux recovery ratio. This result can be explained by the more efficient flux recovery of the hydrophilic MMMs after back-washing (Gray *et al.* 2008). The higher flux recovery ratio of the CNT blended MMMs shows they can be used for a longer time in methyl orange filtration without chemical back-washing. Longer usage of MMMs before chemical back-washing increases the membranes' lifetime. In addition, CNT addition increases the mechanical strength and the chemical stability of the MMMs (Nurazzi *et al.* 2021, Ali *et al.* 2023). Increased mechanical strength and chemical stability also increase the membranes' lifetime.

4. Conclusions

MMM with different CNT amounts were fabricated by the phase inversion method. Prior to fabrication, CNTs were functionalized in an acid mixture. It was shown in our previous publications that functionalized CNTs were shorter, and the carbons at the defect sites, and the tips of the CNTs were converted to carboxylic groups (Celik *et al.* 2011a, b, Celik Madenli *et al.* 2017, 2021, Celik Madenli and Ciftci 2022).

New peak formations in the FTIR analysis confirmed that CNTs successfully blended into the MMMs structure. CNT blended MMMs were more hydrophilic than the bare membranes. In addition, the water content of the CNT blended MMMs was also higher than the bare membrane. Hence, the flux of the CNT blended MMMs was higher than the bare membrane.

Dye removal efficiencies of the MMMs from coloured effluent removal was investigated by using methyl orange as a model dye. Methyl orange rejection of MMMs was 25-45 % higher than the bare membrane because of the electrostatic repulsion and size exclusion. In order to investigate the fouling resistance of the membranes, the flux recovery ratios of the MMMs were determined. Even though the flux recovery ratio of the bare membrane was 78 %, the flux recovery ratio of the MMMs was around 85 %. The higher flux recovery ratio of the CNT blended MMMs shows that they can be used for a longer time in methyl orange filtration without chemical back-washing. Longer usage of MMMs before chemical back-washing increases the membranes' lifetime.

As such, CNT blended MMMs can be an alternative for dye removal from coloured effluents with their increased rejection performances and flux recovery ratios.

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