Chapter 6

Summary, Conclusions & Future Prospects

The summary and conclusions of all the chapter are discussed here.

Chapter 1 introduces polymeric membranes and their preparation methods. It highlights the most commonly used polymers for membrane preparation, namely polysulfone and polyether sulfone, due to their thermal and chemical stability and high glass transition temperature. These polymers belong to the polysulfone family of polymers. The chapter further discusses mixed-matrix membranes, which are prepared by incorporating fillers into the polymeric matrix. The use of carbon nanotubes as a filler is highlighted due to their strong π-π stacking interaction with aromatic compounds and promising material for creating nanocomposite membranes. The chapter also covers surface modification methods, such as click reaction and simple reactions with trimesoyl chloride, cyanuric chloride, and polyethylenimine. It also emphasizes the importance of wastewater treatment and heavy metal rejection via membrane technology.

Chapter 2 of the research document serves as a crucial foundation for the study, presenting a clear and concise overview of the research objectives and goals. This section establishes the purpose and direction of the study, acknowledges the challenges, and outlines its objectives. In doing so, Chapter 2 lays a solid groundwork for the rest of the research, ensuring that the study is conducted with rigor and purpose.

In Chapter 3, the procedure for functionalizing multiwalled carbon nanotubes (MWCNTs) and preparing a mixed matrix membrane consisting of PES or PSU/f-MWCNTs using the phase inversion technique is discussed. This chapter also covers surface modification methods for the prepared membrane. In this chapter, the click reaction for modifying the PSU/azide-CNTs mixed matrix membrane is discussed, where the azide functional groups are converted into triazole rings. Additionally, modifications of PES or PSU/amine-MWCNTs using trimesoyl chloride and cyanuric chloride are described, as well as modifications using polyethylenimine (PEI) on PES or PSU/oxidized-MWCNTs membranes in the experiment section. Then described the characterization techniques for the prepared functionalized MWCNTs, prepared mixed matrix membranes and modified mixed matrix membranes via different methods. The functionalized MWCNTs were analyzed using FTIR and TEM, and the results confirmed the successful functionalization of the MWCNTs, with the TEM images showing no change in the structure of the carbon nanotubes. All the modified membranes were characterized using FT-IR and XPS, which confirmed the successful modification of the membrane surface. XPS analysis of the modified polysulphone/azide-MWCNTs membrane indicated the successful formation of a triazole ring on its surface. Similarly, the PES and PSU/amine-MWCNTs membranes modified with trimesoyl chloride displayed the formation of an amide linkage, while modification with cyanuric chloride resulted in the formation of an amide and the presence of a triazine ring on the membrane surface. PES and PSU/oxidized-MWCNTs membranes modified with polyethylenimine (PEI) also showed the formation of amide linkages and the presence of amine groups on their surfaces. The zeta potential study further confirmed the presence of functional groups on the surface of the membrane and revealed the surface charge of the membranes. The PSU/azide-MWCNTs membrane was initially positively charged; however, after the click reaction, it became negatively charged. The PES and PSU/amine-MWCNTs membranes are positively charged due to the presence of amine groups, but after treatment with trimesoyl chloride, they become negatively charged because of the presence of acyl functional groups that are prone to hydrolysis. However, treatment with cyanuric chloride positively charged the membrane owing to the presence of a nitrogen-containing triazine ring. Additionally, the PES and PSU/oxidized-MWCNTs membranes modified with polyethylenimine (PEI) showed a positive charge on the membrane surface owing to the presence of amine groups that protonate under acidic conditions. These surface charges facilitated the removal of heavy metal ions and foulants through electrostatic attraction or repulsion.

The focus of Chapter 4 was on evaluating the performance of unmodified and modified membranes through permeation studies. The study aimed to determine the permeability of the membranes by conducting permeation tests with pure water as the feed stream. Pure water flux of PSU/azide-MWCNTs membrane gives higher flux than the pristine PSU membrane, which further increases after the click modification, where triazole ring introduced on the surface of the membrane. The PES and PSU/amine-MWCNTs membrane exhibit a higher pure water flux compared to the pristine membranes because the incorporation of amine-MWCNTs in the polymeric matrix increases the porosity of the membrane. However, the treatment with trimesoyl chloride and cyanuric chloride results in a decrease in pure water flux due to either a reduction in pore sizes or the blockage of pores on the membrane surface. The pure water flux of the PES and PSU/oxidized-MWCNTs membranes is higher than that of the pristine membrane due to increased porosity. However, the pure water flux decreases after modification with polyethylenimine because of pore reduction or blockage. The antifouling behavior of the membrane was investigated by using bovine serum albumin (BSA). The study indicated that the incorporation of f-MWCNTs into the membrane improved its antifouling properties, and this improvement was further enhanced after the membrane was modified. The rejection of BSA by the membrane was also improved after modification, which was attributed to the surface roughness and surface charge of the membrane. BSA is negatively charged, and its rejection through the membrane is due to electrostatic attraction or repulsion, although in both cases, BSA does not accumulate on the membrane surface because of the presence of functional groups and the surface charge. As a result, the antifouling property of the membrane was enhanced after modification. The rejection of heavy metal ions by the unmodified and modified membrane also depends on the functional groups and surface charge of the membrane surface. This is because the rejection of heavy metal ions is because of both adsorption and complexation with functional groups present on the membrane surface. Additionally, the surface charge of the membrane affects the rejection of heavy metal ions through electrostatic attraction or repulsion. In our study, we used Cr(VI), Cu(II), Pb(II), Cd(II), and Hg(II) to investigate heavy metal rejection through membranes. The rejection of heavy metals can reach up to 96-99% after successfully modifying the membrane surface. This is because of the introduction of functional groups and increased functionalities on the membrane surface. For the PSU/azide-MWCNTs membrane, a triazole ring is introduced on the membrane surface, acting as a ligand to form a complex with heavy metal ions and reject them. The PES and PSU/amine-MWCNTs membrane is modified with trimesoyl chloride introduces a 1,3,5-benzenetricarbonyl ring and modified with cyanuric chloride introduces 1,3,5-triazine ring on the membrane surface, while the PSU/oxidized-MWCNTs membrane is modified with polyethylenimine, introducing multiple amine groups on the membrane surface to facilitate the formation of a complex with heavy metal ions. As a result, heavy metal rejection increases after surface modification.

The results and discussion in Chapter 5 focus on the morphological analysis of the membranes using FE-SEM, AFM, contact angle measurement, and SANS. The FE-SEM examined the cross-sectional structure of the membranes. The presence of fingerlike structures in the PES membrane and sponge-like structures in the PSU membrane was observed, and these morphologies remained unchanged even after the incorporation of f-MWCNTs and surface modification of the membranes. This suggests that the bulk morphology of the membrane was not affected by the surface modification. AFM (Atomic Force Microscopy) reveals the surface roughness of the membranes. Studies have shown that incorporating f-MWCNTs into the membranes leads to a decrease in surface roughness, resulting in a smoother surface because of the uniform distribution of the f-MWCNTs. However, after surface modification, the surface roughness slightly increases due to chemical reactions on the membrane surface. This increased roughness can impact the antifouling property of the membranes. The hydrophilic nature of the membranes that have been incorporated with f-MWCNTs is higher than that of the pristine membranes. This suggests that the functional groups on the membrane surface facilitate the interaction of water molecules, resulting in a higher pure water flux. However, after surface modification of the membranes, the hydrophilicity decreases and the contact angle increases, which further supports a decrease in pure water flux. SANS profiles provide information on the pore size and polydispersity of the membrane. The mixed matrix membrane with f-MWCNTs has smaller pores than the pristine membranes because of the introduction of f-MWCNTs. After surface modification, the pore size of all membranes decreases, which can be attributed to the modification process causing pore constrictions or blockages, resulting in a reduction of pore size. Despite this variation in pore size, all membranes exhibit the same level of polydispersity, indicating a similar degree of non-uniformity in their size distribution.

In Chapter 6, a summary of the work completed and the conclusions that can be drawn from the various studies conducted during the course of this project are presented.

Our research demonstrates that incorporating f-MWCNTs into the polymer matrix enhances the performance of the membrane. Specifically, it improves heavy metal rejection and antifouling properties. These improvements were further enhanced after surface modification of the membranes, which introduced functional groups and increased the number of functionalities on the surface. Additionally, the surface modification did not affect the bulk morphology of the membrane.

Future scope

Further research is needed for the accurate quantification of the functional groups on the membrane surface to facilitate easier and more precise modifications. The work regarding the mechanism of the separation of heavy metal ions through these surfaces modified membrane should be carried out, which is either because of the adsorption or complexation. Additionally, assessing the membrane efficiency for heavy metal rejection is crucial to understand the overall performance and potential applications of these modified membranes. This research could pave the way for designing advanced mixed matrix membranes with enhanced heavy metal rejection capabilities, contributing significantly to water purification and environmental protection efforts.