SYNTHESIS AND EVALUATION OF FUNCTIONALIZED CARBON NANOTUBES (CNT) BASED POLYMER COMPOSITE NANOFILTRATION MEMBRANES FOR DESALINATION

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ABSTRACT

Composite membranes were fabricated by incorporating Carbon Nan tubes (CNT), both single walled carbon nanotubes (SWCNTs) and multi walled carbon nano tubes (MWCNTs) in Polyvinylidene Fluoride (PVDF) membranes. Some amounts of CNT were dispersed (30 minutes of sonication) in Dim ethyl Sulfoxide (DMSO) solvent for 0.5 mg/ml. This suspension, after vacuum filtration dispersed over PVDF membrane of pore size 0.22µm. Excess DMSO was removed by washing using Ethanol followed by Deionized water. Normal and various functionalized (Hydroxyl (-OH), Carboxylic (-COOH), Amine (-NH₂)) SWCNTs and MWCNTs were purchased and the CNT based polymer composite nanofiltration membranes were fabricated by vacuum filtration method at a uniform loading of 10mg/cm². The experimental results were used to evaluated the TDS removal efficiency of the fabricated membrane modules, which are normal and functionalized (4 composites in both SW and MW combinations) CNT. The TDS removal efficiency of all the 8 composite membranes were observed under varied operating conditions viz., varying flow rate (100,150,200 and 250 ml/hr) and influent TDS (2500, 3000, 4000 and 5000 mg/l). The flux of the membrane is varied 200 to 500 lit/ m^2 .hr. The maximum removal of TDS at 84% was observed for an influent flow rate 200 ml/hr and hence the experimental results pertaining to 2500 mg/l of TDS and for the influent flux of 400 lit/m².hr. This performance was observed while the experiment runs on amino functionalized SWCNTs based polymer composite (PVDF/SWCNTs) nanofiltration membranes.

Keywords -- Desalination, Membrane filtration, Polyvinylidene fluoride, Single walled carbon nanotube, Sonication

I. INTRODUCTION

Desalination process is widely used throughout the world and there is a need of more efficient membranes that can separate salt from water. Several approaches have been made for desalination. Recently, CNT gained more attention in the field of water purification because of their variety of engineering and other applications. CNT membranes provide efficient transport of water molecules, because of their high aspect ratio and smooth hydrophobic walls. The main aim of this research was synthesis and evaluation of functionalized CNT based polymer composite nanofiltration membranes, study their performance for desalination using the dead - end filtration system, with various parameters like salt rejection, water flux and pressure.

II. MATERIALS AND METHODS

2.1 Materials

SWCNTs and MWCNTs were purchased from Nanoshell LLC, Wilmington DE. The hydrophilic PVDF membrane (pore size $0.22~\mu m$ and having a thickness of $120~\mu m$) was from Millipore. DMSO (HPLC grade), Ethanol and Sodium Chloride were from Sigma Aldrich.

2.2 Membrane Fabrication

Functionalized CNT based polymer composite nanofiltration membranes were synthesized by vacuum filtration method. Required amount of CNT were dispersed in DMSO solvent to a concentration of 0.5 mg/ml. After that the suspension was sonicated (30 minutes) in a water bath sonicator, the suspension was filtered through vacuum filtration. The well dispersed suspension was deposited on a PVDF membrane of 25mm diameter. Subsequently, Ethanol followed by De-ionized water was passed through the membrane for the excess removal of DMSO. CNT based polymer composite nanofiltration membranes using normal and various functionalized (-OH, -COOH, -NH₂) SWCNTs and MWCNTs were fabricated by vacuum filtration method for uniform CNT loading of 10mg/cm².

2.3 Experimental Set up

The experimental set up principally has the fabricated modular Nanofiltration filtration system with 25mm membrane filter holder assembly with the membrane effective surface area of 1.7 cm². The CNT based polymer composite nanofiltration membrane was mounted on the filter holder and the membrane was fixed in position with the use of clamp. The feed water was pumped through the membrane by means of a peristaltic pump for varied inflow rate of 100,150,200 and 250 lit/hr and the permeate water was collected. Pressure gauge was fitted in the pipe to measure the pressure variations. The schematic of the experimental set up is presented in Fig.2.1.

2.4 Experimental Methodology

The experimental results were used to evaluated the TDS removal efficiency of the fabricated membrane modules, which are normal and functionalized (4 composites in both SW and MW combinations) of CNT. The TDS is imparted by dissolving sodium chloride at required levels. The TDS removal efficiency of all the 8 composite membranes were observed under varied operating conditions viz., varying flow rate (100,150,200 and 250 ml/hr) and influent TDS (2500, 3000, 4000 and 5000 mg/l). The flux of the membrane is varied 200 to 500 lit/m².hr.

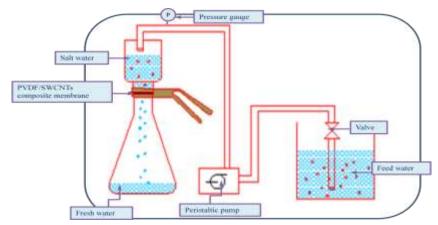


Figure 2.1: Schematic of the experimental setup

III. RESULTS AND DISCUSSION

3.1 Membrane characterization

Scanning Electron Microscopy (SEM) & Atomic Force Microscopy (AFM) were used to characterize the surface morphology of the membrane. **Fig.3.1.** Shows the SEM image of the functionalized CNT based polymer composite nanofiltrtion membrane. From the SEM images, it was observed that the brightest area represents the highest point of membrane surface and the dark regions indicate membrane pores. It seems that the surface porosity increased and the pore size of the membranes decreased due to the functionalization of CNT. The functionalization of the nanotubes helps in formation of physical bonds between the polymer and the functionalized nanotubes and so this influences the formation of the porous structure.

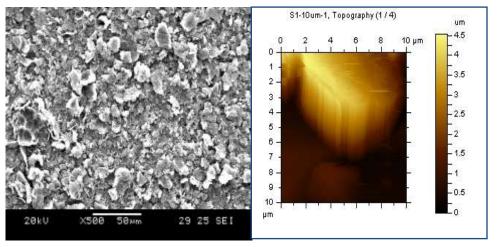


Figure 3.1: SEM image

Figure 3.2: AFM image

The surface roughness of the prepared membranes was determined by AFM. Fig. 3.2 shows the AFM image of the composite membrane. The surface roughness is an important membrane characteristic, which influences the adsorption capacity of the functionalized CNT based polymer composite nanofiltration membranes.

3.2 TDS Removal Efficiency of Functionalized CNT Based Polymer Composite Nanofiltration Membranes.

3.3 Experiments are conducted for all the 8 composite membranes and the results were observed under varied operating conditions.

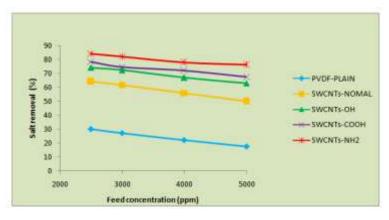


Figure 3.3: TDS removal efficiency of the PVDF/SWCNTs nanofiltration membranes.

Fig.3.3 shows the salt removal efficiency of the SWCNTs based polymer composite (PVDF/SWCNTs) nanofiltration membranes. It was noted that the rejection of salt for plain PVDF membrane was 31 %, and this was increased to 64% for PVDF/SWCNTs nanofiltration membrane for the feed concentration of 2500 mg/l for an influent flow rate 200 ml/hr. **Fig.3.4** shows the TDS removal efficiency of the MWCNTs based polymer composite (PVDF/MWCNTs) nanofiltration membranes. Composite membranes with functionalized CNT offered high potential merit for application in membrane adsorption process to remove TDS. Functionalization of the CNT makes the membranes more hydrophilic, imparts greater adsorptive nature, and reduced pore size. The reduced pore sizes and high adsorption help the membranes to be efficient enough for TDS removal.

The maximum removal of TDS at 84% was observed for an influent flow rate 200 ml/hr and hence the experimental results pertaining to 2500 mg/l of TDS and for the influent flux of 400 lit/m².hr. This performance was observed while the experiment runs on amino functionalized PVDF/SWCNTs nanofiltration membranes.

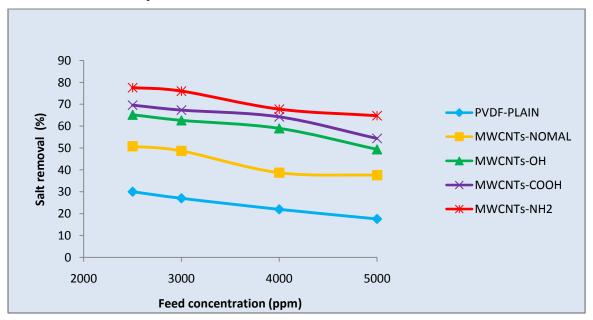


Figure 3.4: TDS removal efficiency of the PVDF/MWCNTS nanofiltration membranes.

IV. CONCLUSION

The synthesis and salt removal efficiency of functionalized CNT based polymer composite nanofiltration membranes were examined with various factors such as TDS removal, water flux and pressure. From the SEM and AFM images apparent pore size of the functionalized CNT based polymer composite nanofiltration membranes were smaller than the normal CNT based polymer composite nanofiltration membranes. Amino functionalized PVDF/SWCNTs nanofiltration membranes (84%) exhibited good TDS removal capacity than any other membranes for the feed concentration of 2500 mg/l for an influent flow rate 200 ml/hr at a low pressure of less than 1 bar. The results reveal that this membrane system can run without a high pressure pump, whereas reverse osmosis and ultra filtration require high pressure for operation.

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