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Carbon nanotube/polysulfone nano composite membranes for the filtration of crude oil/water mixture

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Executive summary

Membrane technology is widely used for water treatment purposes in a wide array of industries. This research focused on the use of this technology in the oil and gas industry. This is primarily because of its low cost and low energy requirements, its environmental friendliness and its ability to treat large quantities of waste water. The ability to scale membranes makes them very attractive for use on offshore platforms where space is limited. This research focused on the use of polymeric membranes in the treatment of produced water primarily from enhanced oil recovery (EOR) processes. This is when the energy of the oil or gas reservoir is boosted using chemicals, heat, biological or technical techniques. Water is a highly used resource in this process and this contributes to the large quantities of produced water waste from this industry.

Carbon Nanotubes (CNTs) were first produced and characterized. CNTs are widely studied nano-materials due to their good qualities and vast applications. Numerous techniques have been developed for the synthesis of this material with chemical vapor deposition method (CVD) being most common. This involves the growth of CNTs, usually in the presence of a catalyst at a high temperature. The effect of the catalyst support (zeolite and $CaCO_3$) on the synthesis of CNTs was first studied. It was observed that CNTs that were grown from zeolite supported catalysts (CNT-z) had smaller outer diameters ranging from 10nm to 50nm, while CNTs grown from $CaCO_3$ supported catalysts (CNT-c) had larger outer diameter of 20nm to 80nm. This disparity in the outer wall diameter was still evident after functionalization with HNO_3. Another difference between the two supports was the CNT purity achieved after synthesis. Functionalized CNT-z (fCNT-z) had more catalytic impurities than functionalized CNT-c (fCNT-z), which was almost clean of any residual catalyst. The impurities in the fCNT-zs were found to be SiO_2 . This was removed by HF treatment to give very pristine CNT-zs. The CNTs were characterized with Raman, Fourier transform infrared radiation (FTIR), thermogravimetric/derivate weight loss analysis (TGA/DTG), scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). The fCNT-cs were used in the membrane preparation because of their larger size, which meant better interaction with the membrane structure.

Two techniques were used in the production of the membranes: the solution casting method (SC method), which produced non-porous, dense and isotropic membranes, and the phase inversion method (PI method), which produced micro-porous anisotropic membranes. The membranes were modified using fCNTs and a polyester non-woven fabric (NWF). The membranes were characterized using the SEM, atomic force microscopy (AFM), contact angles, FTIR, TGA/DTG and tensile

strength analysis. The surface morphologies clearly revealed the surface pores while the cross sections showed voids. The pore sizes ranged from 1.598μm for the membranes with the NWF, to 0.191mm for the CNT imbedded membranes without the NWF. The surface pores were not as evident in the solution cast membranes. The dense nature of these membranes meant they had significantly higher tensile strengths and Young's moduli. Rougher membranes were produced with the PI method and this confirmed the presence of pores with lower contact angles. TGA/DTG showed that SC membranes were more thermally stable at higher temperatures than the membranes produced *via* PI.

Functionalized CNTs interacted with the hydrophobic membranes to enhance its physical, chemical and mechanical properties. CNTs reduced the contact angles, increased the surface roughness, pore and void sizes and the tensile strengths for both kinds of membranes. It however reduced the thermal stability and rigidity of the membranes. The addition of the NWF support also decreased the pore sizes of the membranes by PI and introduced voids into the membranes by SC.

The PI membranes were tested for performance using a synthetically made produced water. It was shown that increasing pressure increased permeate flux. Functionalized CNTs also increased permeate flux while controlling fouling via pore blockage. The addition of NWF also resulted in flux decline by providing additional resistance to the flow of permeate through the membrane. Water and oil permeates were then collected and tested for oil concentrations. The results showed oil rejections ranging between 78% and 90% with the mixed matrix PI membranes supported on a polyester NWF having the highest oil rejections.