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**Procedia
Engineering**www.elsevier.com/locate/procedia**Euromembrane Conference 2012****[P2.099]****Real time, in-situ monitoring of surface and structural properties of thin film polymeric membranes using electrical impedance spectroscopy**A. Antony¹, T. Chilcott², H. Coster², G. Leslie*¹¹University of New South Wales, Australia, ²University of Sydney, Australia

The only practical method for monitoring the performance of thin film composite (TFC) aromatic polyamide membranes in real time relies on the normalisation of bulk observations of pressure, flow and conductivity. These measurements lack the sensitivity to detect the subtle changes occurring across the membrane that set the trend for either improvement or decline in system performance[4]. Direct observation at the nano-scale on how the various layers of the composite membrane respond to the movement of water or ions during operation is not practical in industrial settings. What is needed is an analytical technique that can be used *in-situ* and *in real* time to identify changes that occur across the thin film (from the feed side to the permeate side) and a model that can relate such changes to the functionality of the membrane (i.e. reduced fouling). The present article aims at achieving this using Electrical Impedance Spectroscopy (EIS).

EIS utilizes an alternating current over a wide range of frequencies to probe materials or devices to depth scales ranging from the atomic to macroscopic dimensions and studying the polarization phenomena associated with the electrical response. EIS works by injecting a sinusoidal alternating current of known frequencies into a system and measuring the voltage (potential difference) across the system including the phase difference between the current and voltage. From this the impedance, phase angle, conductance and capacitance can be determined. The variation of these properties with frequency commonly referred to as dispersion, can then be used to determine the number and properties of “layers” which compose the system of interest.

Conductance is the ability of the system to conduct electric charge while capacitance, is the capacity of the system to store the charge. For an electrically homogeneous system G and C are independent of frequency. However, in the electrically heterogeneous membrane system comprised of electrically distinct substructures G and C are strongly dependent on frequency.

The nascent stages of flux decline and scale formation during the processing of a 2000 ppm solution of calcium carbonate (initial LSI 0.3) were monitored *in situ* as process recovery increased from 0 to 87% using Electrical Impedance Spectroscopy (EIS). As expected, after filtration in batch mode for 18 hours, CaCO_3 precipitation presented as a decrease in the temperature normalized flux (J/J_0) from 1.0 to 0.73 and an increase in normalized permeate conductivity from $5 \text{ } \square \text{Scm}^{-1}$ to $35 \text{ } \square \text{Scm}^{-1}$ (Figure 1). The permeate conductivity increased in concert with the decline in flux and the ionic rejection efficiency decreased from 98 to 80% as the filtration progressed from 0 to 18h.

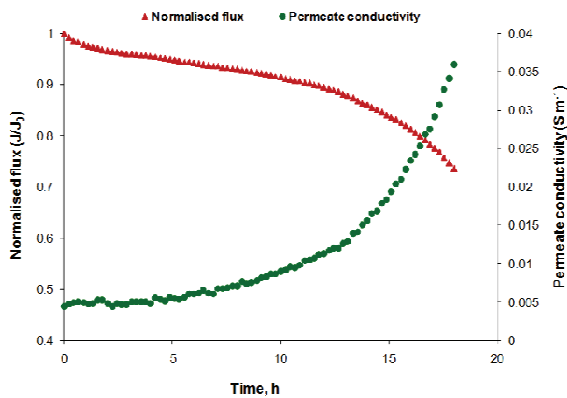


Figure 1 Normalized flux decline and permeate conductivity as a function of time.

During the fouling experiment, EIS spectra generated by alternating current at frequencies between 1 Hz and 10^6 showed a decrease in impedance with increasing recovery. The most significant spectra were generated at 5×10^5 Hz where a change in phase angle from -40° to $+5^\circ$ was observed as recovery increased from 0 to 84%, indicating that the system became less conductive and more resistive as the recovery increased. Spectral data was fitted to a Maxwell-Wagner dielectric software model to resolve the six electrically distinct layers (Figure 2) that occur in series from the feed side to the permeate side of the membrane.

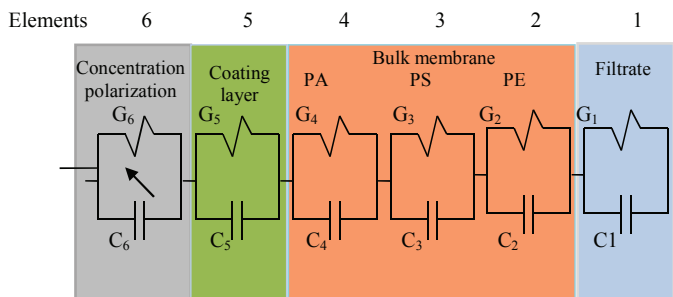


Figure 2 Equivalent circuit model for the in-situ RO system (PA-Polyamide, PES – Polyethersulfone and PE – Polyester).

The model was consistent with the observed change in electrical conductivity of permeate which increased from 0.004 to 0.02 Sm^{-1} that occurred as salts accumulated on the membrane surface with increasing recovery. Furthermore the fitted EIS measurements verified the orders of magnitude of literature values for the thickness of the polyamide layer (ca. 100 nm), polyethersulfone layer (ca. $50 \text{ }\mu\text{m}$) and polyester support ($120\text{-}150 \text{ }\mu\text{m}$) that constitute commercial reverse osmosis membranes. Temporal changes in conductance of these layers during filtration were greatest for a zone positioned immediately above the polyamide active layer which changed from 4.9 to 22 Sm^{-1} which can be attributed to the accumulation of salts on the surface of the membrane. The results indicate that EIS data when fitted to an appropriate model can be used to characterise the various structural elements of a reverse osmosis membrane and the changes that occur *in situ* at the membrane surface during filtration.

Keywords: Electrical Impedance spectroscopy, Realtime monitoring, Reverse Osmosis, Polyamide thin film composite

