



Real-time monitoring of scale formation in reverse osmosis using electrical impedance spectroscopy

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ABSTRACT

Early detection of scale formation in reverse osmosis systems remains challenging since the bulk measurements of the operating data are not sufficiently sensitive to detect the subtle changes occurring across the membrane. Electrical impedance spectroscopy (EIS) was investigated as a tool for measuring the electrical properties in real time and to identify changes that occur during calcium sulfate scaling in a reverse osmosis system. EIS signals were obtained across a frequency range of 10^{-1} – 10^5 Hz during the filtration of calcium sulfate scale forming constituents in recirculation and batch modes. The measured capacitance and conductance of various electrically distinct layers were compared against the flux measurements, in order to assess their significance to indicate the scale formation. The rate of change in conductance was higher than the flux decline. Change in the conductance value around 38 Hz, corresponding to the coating layer on the active membrane surface was most suitable. From the suggested approach of monitoring the changes in conductance at 10–100 Hz, scale formation can be detected prior to observing any significant permeate flux decline.

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1. Introduction

Inorganic scale formation during reverse osmosis (RO) filtration is a significant contributor to the decline in hydraulic and removal performance, which eventually results in high operating and maintenance costs [1]. Optimization of system design and operating conditions, combined with appropriate addition of antiscalant agents is now commonly implemented to better control scale formation [2]. In water treatment industries, the only practical method for monitoring the performance of the RO system *in-situ* and real time relies on the normalization of bulk observations of pressure, flow and conductivity. Specifically, monitoring the performance of the tail end RO element is used as a tool to detect the scale formation. A ratio of the tail end specific flux to the overall system flux is relatively stable in absence of scaling; as scale develops in the tail element, the specific flux ratio declines, indicating the severity of the scaling [3]. Thus, continuous monitoring of the RO performance allows assessing the extent of fouling on the membrane and the need to conduct chemical cleaning. However, 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 [4,5]. Therefore, these monitoring parameters are

expected to inadequately detect the onset of material deposition on the membrane, and only deliver a significant signal, for well-established fouling formation [6,7]. This phenomenon is exacerbated in industrial membrane modules, in which significant fouling profiles are observed within the length of the membrane [6]. This lack of detailed and timely assessment of the fouling state can result in delay in applying the necessary cleaning strategies, negating their effectiveness for fully developed scaling layers. Therefore there is a great need to develop an advanced, *in-situ*, real-time monitoring technique, capable of early detection of the early stages of scale formation.

Among the several *in-situ*, real-time techniques reported [2,8], ultrasonic time-domain reflectometry (UTDR) and *ex-situ* scale observation detector (EXSOD) feature a potential for industrial implementation. UTDR relies on the use of ultrasonic sound waves to detect the thickness of the fouling layer [9], while EXSOD uses high resolution digital photography to assess the fouling characteristics [5]. Both methods are capable of detecting membrane scaling and indicating onset of fouling before significant change in flux decline can be measured. With the unique capacity of characterizing and analyzing the structures of micro-porous membranes, electrical impedance spectroscopy (EIS) has been proposed as another valuable approach to study inorganic fouling on RO membranes [10]. Previous studies from Coster and co-workers [10–13] have demonstrated that EIS offers potential for online monitoring of membrane fouling and optimization of operation systems. The recently developed EIS unit enables the

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continuous *in-situ*, real-time monitoring under high pressure conditions; recent studies demonstrated the application of EIS for the functional and structural characterization of RO systems during filtration by *in-situ* real time measurement at a sub-micron level [14,15]. These studies developed a method of functional characterization of membranes in real time during the filtration enabling to understand the changes that occur across the membrane and early detection of fouling. However no previous studies compared the significance of impedance signals against the conventional flux parameters.

In a previous study, dielectric structural modeling of EIS measurements could detect and characterize five electrically distinct layers and a concentration/diffusion polarization element throughout the filtration experiment [14]. These layers matched the known thicknesses of layers in the membrane system. The *in-situ* measurements provided a basis for characterizing the structural features of the RO system, each membrane sublayer responding to specific frequencies. The frequencies reported were taken as a rationale for the present study, monitoring the scale formation in real time using EIS. Calcium sulfate is a non-alkaline scale, presenting significant challenge for its removal from the membrane surface. The objective of this paper was to implement the EIS technique, to monitor the electrical property changes in RO membranes and compare the significance of impedance signals and flux decline in detecting the calcium sulfate scale formation. Based on both continuous and batch filtration modes, an improved protocol was developed to obtain more relevant information from the EIS data. Selection of relevant frequencies and impedance parameters was considered, to simplify the monitoring process.

2. Methods and materials

2.1. Experimental set-up

A filmtec BW30-FR RO membrane (Dow Filmtec) was used for performing experiments. All membrane samples were soaked in MilliQ water (Millipore, Australia) overnight prior to the experiment. The effective area of the BW30-FR membrane samples used in the experiment was 0.0186 m². A stainless steel filtration cross flow cell (INPHAZE, Australia) was used for the experiments. The high pressure cross flow filtration system used in this study is illustrated schematically in Fig. 1. The operating pressure was controlled by a GE Fanuc controller (GE automation Fanuc-VersaMax) and online

measurement of operating parameters was recorded using CIMPLICITY[®] HMI 8.0 software. An EIS device (INPHAZE, Australia) was connected to the system for the generation of alternative current (AC) signals and recording the impedance parameters nondestructively in real time.

2.2. Model solutions and experimental protocols

Recirculation mode experiments were performed with 10 L of feed solution containing 17 mmol/L of CaCl₂, 13.70 mmol/L of Na₂SO₄, 11.46 mmol/L of MgSO₄ and 0.0087 mmol/L of BaCl₂. In these experiments, both retentate and permeate were returned to the feed tank. Each experiment consisted of three stages: compaction, conditioning and filtration, and all of them performed at a feed with temperature of 26 °C and constant feed pressure of 1400 kPa. During compaction, the membrane was placed inside the stainless steel cell with permeate and retentate spacers, and MilliQ water was circulated through the system for 8 h in order to achieve a stable flux and to improve the impedance signal to the noise ratio. For the conditioning stage, MilliQ water was replaced by the feed solution without the scale-forming constituents including calcium chloride and barium chloride, and filtration performed for 4 h. After conditioning, the remaining salt solutions were added and mixed, and filtration was then performed for 36 h. During the entire filtration process, the permeate flux, conductivity and operational pressure were recorded by the control system. Simultaneously, impedance measurements were performed every 30 min.

Batch mode experiments were performed with 10 L of feed solution containing 8.89 mmol/L CaCl₂ and 7.16 mmol/L Na₂SO₄. Prior to commencing the experiment, the feed solution was stabilized for 3 h. The conductivity and pH of the feed solution was 0.31 S/m and 5.2 respectively, and the saturation index (SI) for calcium sulfate was 0.5, indicating the likelihood of calcium sulfate to precipitate. Each experiment consisted of two parts, compaction and filtration. For compaction, the membrane was placed inside the stainless steel cell with permeate and retentate spacers. MilliQ water was circulated through the system at 1000 kPa constantly for 10 h in order to achieve a stable operating performance, as well as a steady impedance signal. The MilliQ water was then replaced by feed solution and run in batch mode, where the retentate was returned to the feed tank, while permeate was collected separately. Feed and permeate samples were collected for every 10% recovery and the concentration of calcium ions measured through

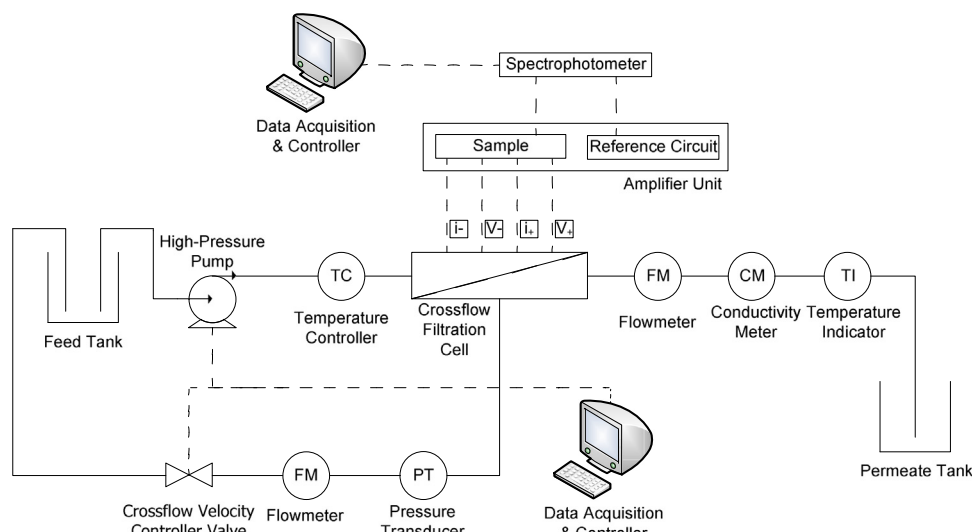


Fig. 1. Schematic representation of the RO cross flow filtration system for the *in-situ* impedance measurement.

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