



## Design and test of a reverse osmosis pressure cell for in-situ small-angle neutron scattering studies



Dietmar Schwahn<sup>a,\*</sup>, Herbert Feilbach<sup>b</sup>, Thomas Starc<sup>b</sup>, Vitaliy Pipich<sup>c</sup>, Roni Kasher<sup>d</sup>, Yoram Oren<sup>d,\*</sup>

<sup>a</sup> Technische Universität München, Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), D-85748 Garching, Germany

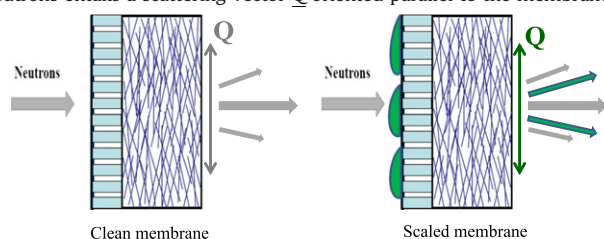
<sup>b</sup> Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science JCNS and Institute for Complex Systems ICS, D52425 Jülich, Germany

<sup>c</sup> Jülich Centre for Neutron Science JCNS-FRM II, Outstation at FRM II, D-85747 Garching, Germany

<sup>d</sup> Zuckerberg Institute for Water Research, Jacob Blaustein Institutes for Desert Research, Ben-Gurion University of the Negev, Sede Boqer Campus, 84990, Israel

### GRAPHICAL ABSTRACT

Neutron scattering patterns for a clean and scaled RO membrane. Scale is shown as green domains resulting in enhanced scattering (green arrows). The pathway of neutrons entails a scattering vector  $\underline{Q}$  oriented parallel to the membrane surface.



Neutron scattering patterns for a clean and scaled RO membrane. Scale is shown as green domains resulting in enhanced scattering (green arrows). The pathway of neutrons entails a scattering vector  $\underline{Q}$  oriented parallel to the membrane surface.

### ARTICLE INFO

#### Article history:

Received 31 May 2016

Received in revised form 19 November 2016

Accepted 29 November 2016

Available online xxxx

#### Keywords:

Reverse osmosis (RO) wastewater desalination

In-situ RO studies using Small-Angle Neutron Scattering (SANS)

SANS characterization of RO membrane

### ABSTRACT

We introduce a new method for real-time studies of membrane scaling and biofouling on thin film composite membranes (TFC) in reverse osmosis and nanofiltration water treatment using in-situ small-angle neutron scattering (SANS). SANS delivers information on nano and microscopic structures that support the interpretation of relevant engineering parameters such as membrane permeability and water flux. A flow cell high pressure SANS is described, followed by SANS characterization of TFC membranes finding  $\sim 0.5 \mu\text{m}$  large cavities and  $\sim 300 \text{ \AA}$  diameter large rod-like cavities inside the non-woven polyester and micro-porous polysulfone layer, respectively. In-situ desalination experiments in cross-flow mode at an applied pressure of 6 bars and feed flow velocity of 0.2 cm/s are followed. The scattering cross-section times sample thickness ( $\mu_t = \Sigma_t \times D_s$ ) derived from the transmission coefficient shows an overall enhancement due to newly formed scattering centers which is accompanied by a reduced membrane permeability measured simultaneously. This observation is supported by enhanced

**Abbreviations:** SANS, small-angle neutron scattering; T, transmission coefficient of neutrons;  $\mu_t$ , sum of linear coefficients of neutron absorption, incoherent and SANS scattering; A, scattering amplitude;  $d\Sigma/d\Omega$ , differential macroscopic cross-section;  $\Sigma_t$ , total macroscopic cross-section;  $\Sigma_{\text{abs}}$ , total absorbing macroscopic cross-section;  $\Sigma_{\text{SANS}}$ , total macroscopic cross-section of SANS;  $Q^2$ , second moment with meaning of the invariant of scattering; b, coherent scattering length;  $\rho$ , coherent scattering length density;  $\Delta\rho^2$ , scattering contrast;  $\Omega_M$ , volume of molecule;  $\delta$ , scattering angle;  $\lambda$ , wavelength of neutron;  $\underline{Q}$ , momentum transfer;  $k_z$ , wavenumber;  $R_g$ , radius of gyration;  $P_d$ , Porod constant;  $\alpha$ , exponent of power law of  $d\Sigma/d\Omega$  at  $Q > 1/R_g$ ;  $V_p$ ,  $S_p$ ,  $N_p$ ,  $\Phi_p$ , volume, surface, number density, and volume fraction of domains exposed to scattering;  $l_c$ , coherence length of domains;  $D_s$ , thickness of sample; HAP, hydroxyapatite; PMP, protein mineral particle; RO, reverse osmosis; NF, nano-filtration; TFC, thin-film composite; TMP, trans membrane pressure; SSE, simulated secondary effluent.

\* Corresponding authors.

E-mail addresses: [d.schwahn@fz-juelich.de](mailto:d.schwahn@fz-juelich.de) (D. Schwahn), [yoram@bgu.ac.il](mailto:yoram@bgu.ac.il) (Y. Oren).

## 1. Introduction

The strong demand for potable water in arid areas around the globe is leading to an increasing number of sea, brackish and wastewater desalination plants using reverse osmosis (RO) and nanofiltration (NF) technologies. Despite this vast activity, fresh water supply in many countries, especially those that are remote from the sea, is still a serious problem. In these cases, recycling of impaired water such as municipal wastewater to a level of unlimited application is a reasonable solution. In fact, this is done today in several locations around the globe such as in Singapore, Windhoek (Namibia), Orange County (CA, US) and Kuwait and is considered by many other countries. Another, not less important aspect of recycling is protection of the environment. Membrane-based technologies are extensively used today in these respects.

A serious problem in RO/NF desalination of municipal wastewater is biofouling and scaling of the membranes, limiting membrane efficiency and lifetime [1–3]. Ning et al. [4] reported that calcium phosphate and organic matter accumulated on the RO-membrane, suggesting that nanoparticles pass through microfiltration and ultrafiltration membranes, ending up on RO-membranes as a cake-layer fouling. In particular scaling by calcium phosphate in municipal wastewater treatment systems is a severe problem as no efficient antiscalting agents are currently available [5].

Calcium-phosphate mineral formation during RO wastewater desalination was studied using surface pressure–area isotherms and spectroscopic analyses of a solution simulating the ionic profile of domestic secondary-treated wastewater effluents (termed simulated secondary effluent - SSE); it was found that calcium phosphate mineralization was accelerated by organic chemical groups that are present on biofouled membranes [6]. In this respect the issue of biofouling-induced scaling is, to a large extent, strongly linked to bio-mineralization [7]. In addition, recent small-angle neutron scattering (SANS) experiments by the authors showed strong influence of organic molecules present in biofilms on mineralization in a model synthetic salt solution [8,9]. Proteins such as BSA, lysozyme and the polysaccharide alginate (AA) added to the solution, induced within a few seconds strong precipitation of nearly  $\mu\text{m}$  large composite particles composed of organic components as well as calcium phosphate and carbonate. In another study gold-nanoparticles were used as templates for studying surface mineralization by SANS in model solutions for wastewater desalination [9, 10]; for example it was found that BSA-gold nanoparticles induced fast precipitates of  $0.2 \mu\text{m}$  that were composed of 50%–80% minerals, identified as calcium phosphate.

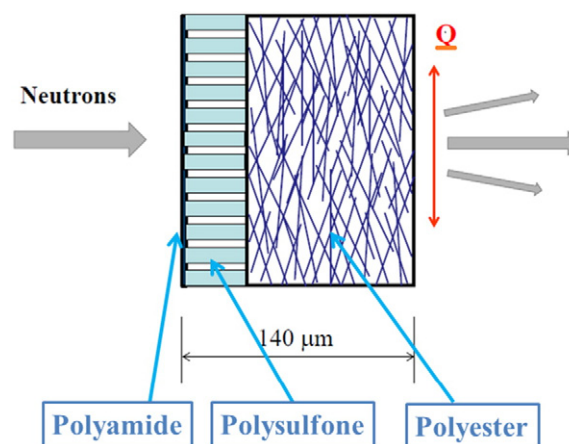
In this study we report on developing of a new cell for in-situ SANS studies of fouling and scaling at thin film composite (TFC) RO/NF membranes. Application of SANS to polymeric TFC membranes appears rational, as static and kinetic properties of polymers are studied with neutron scattering methods [11,12]. Similar in-situ SANS experiments on ceramic ultrafiltration membranes are reported in [13]. Typically, as schematically shown in Fig. 1 TFC RO-membranes are composed of an aromatic polyamide film of 100 to 250 nm thickness on top of a  $\sim 40 \mu\text{m}$  thick porous polysulfone layer. These two layers are supported by a third layer of a  $\sim 100 \mu\text{m}$  thick non-woven fabric made of polypropylene or polyester. The aromatic polyamide skin layer is highly cross-linked whereas the fraction of the cross-linked polyamide repeating units from total polyamide units is about 65% [14–16]. Filtration of aqueous BSA solutions of different pH values was explored and compared with the permeate flux [17]. Another in-situ SANS measurements studied the formation of a cake layer at the surface of an ultrafiltration

membrane which was exposed to a 0.48% aqueous Laponite solution at a transmembrane pressure of 0.5 bars [18].

Currently, several non-invasive real-time methods are presented in literature and demonstrated their application in membrane processes. Greenberg and coworkers developed the ultrasonic time-domain reflectometry (UTDR), where the membrane surface is monitored by sensing the reflection of ultrasonic radiation from the membrane [19]. The UTDR was proved efficient in studying membrane characteristics, as well as monitoring membrane fouling and biofouling [20,21]. However the detection of biofilm and scaling on membrane surfaces is still a significant challenge for UTDR due to the small difference in acoustic properties at the fouling/membrane and fouling/feed solution interfaces [20]. Electrical impedance spectroscopy (EIS) was also demonstrated as a membrane monitoring and characterization technique, as was applied for studying membrane fouling [22–26]. Nevertheless, unlike UTDR or SANS it is in essence an invasive technique that requires special arrangements to provide the proper electrical connections. SANS might be outstanding with respect to other methods mentioned above as it enables basic studies of membrane phenomena and distinguishing between inorganic and organic fouling via contrast variation and their evolution during desalination on a level of microscopic length scale under most realistic real-time conditions. It is the main goal of this effort to elucidating the influence of fouling on scaling using in-situ SANS monitoring at conditions that are close to those prevailing in actual membrane processes.

## 2. Theoretical background of SANS

Scattering of photons and, as in our case, neutrons occurs in momentum ( $\underline{P}$ -) space in contrast to optical, electron (TEM) and atomic force (AFM) microscopies which are reproducing their results in real ( $\underline{R}$ -) space. This implies that scattering methods do not provide pictures of our real world but rather of the so-called reciprocal or the momentum space [27]. The coordinates of the reciprocal space are formed by the components of the scattering vector  $\underline{Q}$  which corresponds to the momentum transfer  $\Delta\underline{P}$  (the symbols  $\underline{Q}$  and  $\Delta\underline{P}$  are representing vectors) of the neutrons and are interrelated via the well-known de Broglie relationship  $\Delta\underline{P} = \hbar \underline{Q}$  ( $\hbar$ , Planck constant  $h$  divided by  $2\pi$ ). The absolute



**Fig. 1.** Schematic design of a thin film composite RO membrane as related to neutron scattering. The three layers are of aromatic polyamide (100–200 nm thick), porous polysulfone (about  $40 \mu\text{m}$  thick), and polyester (about  $100 \mu\text{m}$  thick). The overall thickness of the present membrane is  $\sim 140 \mu\text{m}$ .

Download English Version:

<https://daneshyari.com/en/article/4987905>

Download Persian Version:

<https://daneshyari.com/article/4987905>

[Daneshyari.com](https://daneshyari.com)