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Studies on membrane scaling during water desalination by direct contact membrane distillation

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ABSTRACT

The formation of deposits on the membrane surface during membrane distillation (MD) of tap and ground saline water was studied. The effect of feed composition on the intensity of membrane scaling was discussed. Scanning electron microscopy observations indicated that a deposit, which precipitated during brine desalination, covered the entire surface of membranes, resulting in a decline of the membrane permeability. The impact of membrane morphology on the pores wetting was investigated in detail. It was found that hydrophobic membranes with small pores and thicker walls were recommended for commercial MD applications. They were found to be more resistant for wetting, especially if the scaling phenomenon occurred. A feasibility of freshwater production from brines was evaluated. During desalination of saturated NaCl solutions, almost 99% salt rejection was achieved.

Keywords: Membrane distillation; Desalination; Scaling; Hydrophobic membrane

1. Introduction

Membrane distillation (MD) has been extensively studied for many years, and a significant increase in the number of papers can be observed in recent years. Most of achievements were described in review papers [1–8]. Moreover, additional information for both novice and advanced MD readers was provided in the book [9]. The MD process enables the development of environmentally friendly desalination technology, and three research projects on the topic, that is, MEDINA, MEDESOL and MEDIRAS, have been funded by the European Commission within the sixth and seventh framework programs [6,10–12]. One of the objectives of these projects was to implement MD desalination technology into the market.

Major barriers for commercial acceptance of MD process and its implementation to industry are associated with a membrane pore wetting, low permeate rate and a flux decline. In the case of freshwater production by MD, the process must be competitive against desalination technologies based on the reverse osmosis (RO), which requires to achieve both high yields and good thermal efficiency in the MD process. The fulfilment of these conditions will need the application of membranes specially designed for MD process [2–9,12–18].

A high mass transport rate can be ensured by making a MD membrane as thin as possible, while a low heat loss requires to fabricate a membrane as thick as possible. Therefore, a choice of membrane thickness is a compromise between a high permeate flux and a low heat transfer by conduction [9]. The modelling of the process indicated that the optimum thickness of the hydrophobic layer would be about or even below 10 μm [19]. A hydrophobic layer of this thickness must be supported by a porous hydrophilic layer [6,9]. One of the advantages of the multi-layered membrane is that an increase of the overall membrane thickness enables a low heat transfer. The membrane morphology, such as

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thickness, pore size distribution and porosity, has also been theoretically studied by Laganà et al. [20]. They concluded that the optimum membrane thickness was within the range of 30–60 μm. However, if scaling phenomenon occurred, the significantly thicker membranes, for example, 400 μm, were proposed for industrial applications [21].

The osmotic pressure does not affect significantly the driving force in MD process; thus, this process can be used for production of freshwater from brines and is considered as a complementary process to RO. It actually enables a further concentration of RO brines, which enhance the global water recovery of the desalination process [9,14,22–25]. Since many such feed waters contain sparingly soluble salts, the ability to use MD for desalination of these waters is restricted when salt solutions become supersaturated [3,21]. The adsorption of dissolved organic compounds on membrane surface or scaling induces membrane wetting, and a decrease of the permeate quality is observed. Therefore, both membrane fouling and scaling have been identified as a major challenge currently hindering the realization of full-scale MD installation for desalination purposes [6,23–28]. The formation of deposit layer on the membrane surface induces also new thermal and mass transfer resistances, which reduce the permeate flux; moreover, scaling would cause a physical damage in membrane pores structure [27–30].

Different methods of scaling intensity limitation in MD process were proposed [26–28]. The best solution is the application of additional processes, which eliminates saturation states in the feed before it inflows into the MD module [31]. An accelerated precipitation softening, which was integrated with direct contact MD (DCMD) to establish a desalination process for high-recovery desalting of RO concentrate, created such a possibility [29]. Another method is a cyclic removal of deposit from the membrane surface by module rinsing. Nghiem and Cath [26] reported that Na_2 EDTA solutions were used for membrane cleaning covered by the silica and calcium sulphate precipitated during MD of RO brines. The application of polytetrafluoroethylene (PTFE) and polypropylene (PP) membranes was compared regarding a fouling susceptibility and resistance to chemical cleaning. Only the performance of polypropylene membranes was comparable before and after cleaning. It implies that a scale deposition on the PP membranes is less adhered to the membrane. In another study, PP membranes were coated by fluorosilicone to prevent pore-clogging and to avoid the surface nucleation and particle attachment [32]. However, crystals of sparingly soluble salts were observed on the membrane surface after MD experiments. These crystals caused a partial membrane wetting, and as a result, a continuous increase of distillate conductivity was observed during MD process [32].

In this work, an intensity of scaling formed on the surface of polypropylene membranes was investigated.

2. Experimental

The studies on the water desalination were carried out using a DCMD. The experimental setup is presented in Fig. 1. The feed flowed inside membrane capillaries (lumen side), whereas the distillate flowed through the inter-capillaries space (shell side). Peristaltic pumps were used, and the volumetric flow rate of feed and distillate streams was equal to 6 ± 0.2 mL s^{-1}. The feed temperature was fixed at 353 ± 1 K by Nűga temperature regulator (Germany). The distillate stream was cooled by tap water, and its inlet temperature was maintained at 290–293 K. Process temperatures were measured using thermometers with ±0.2 K accuracy. At the beginning of each experimental series, the distillate loop was refilled by 0.5 L of distilled water (5–7 μS cm^{-1}). The obtained permeate flux was calculated on the basis of distillate volume increase within studied period of time.

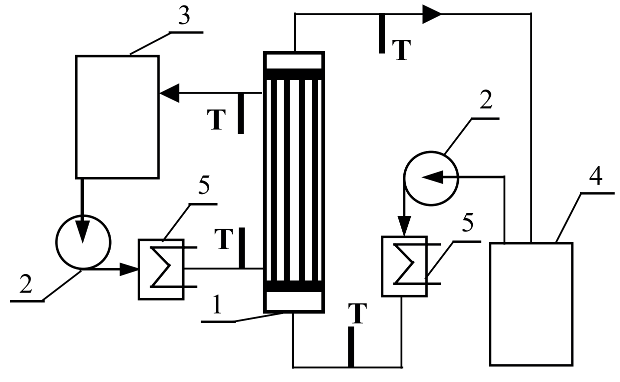


Fig. 1. MD experimental setup.
Notes: 1 – MD capillary module, 2 – peristaltic pump, 3 – distillate tank, 4 – feed tank, 5 – heat exchanger, and T – thermometer.

Capillary, polypropylene membranes Accurel PP manufactured by Membrana GmbH (Germany) were assembled inside MD modules. Length of MD modules was equal to 23 cm, whereas external and internal diameters of the used membranes capillaries were 2.6 and 1.8 mm (Accurel PP S6/2 – MD1, MD3, MD4), or 8.6 and 5.5 mm (Accurel PP V8/2 HF – MD2), respectively. Membranes characterized with pore sizes of nominal and maximum diameter equal to 0.2 and 0.6 μm, respectively, and with porosity of 73% (manufacture's data). The internal membrane area (lumen side) was equal to 0.012 m^2 for used modules.

A tap water and ground saline water from Dębowiec Mine and Saltworks (Poland) were applied as a feed in presented MD study. Long-term DCMD studies were performed using a cyclic (Mode I) and a continuous mode (Mode II) of installation operation. Studies were carried out at constant feed parameters (temperature and flow rate), in order to evaluate the rate of wettability of applied membranes as a function of elapsed time for DCMD process.

Both, morphology and composition of deposits formed on membranes surface were studied using scanning electron microscopy (SEM) coupled with energy dispersion spectrometry (EDS). Samples for cross-sectional observations were prepared by fracture of capillary membranes in liquid nitrogen. All samples were next sputter coated with gold and palladium.

Anions and cations concentrations were measured using ion chromatography method with conductivity detector (850 Professional IC, Herisau Metrohm, Switzerland). The separation of anions was performed on 1.7 × 3.5 mm Metrosep RP guard column in series with a 250 × 4.0 mm Metrohm A Supp5-250 analytical column. A solution comprising 3.2 mM L^{-1} Na_2CO_3 + 1.0 mM L^{-1} $NaHCO_3$ (flow rate 0.7 ml min^{-1}) was used as eluent. A C2 guard column in series with a 150 × 4.0 mm Metrosep C2-150 analytical column was used for cations separation. In this case, a mixture of tartaric acid (4 mM L^{-1}) with 0.75 mM L^{-1} 2-picoline acid acted as an eluent. Additionally, a Jobin Yvon Ultrace 238 JY atomic emission spectrometer with inductively coupled plasma (ICP–AES) was used to determine cations content.

Ground saline water, beside 75 g NaCl L^{-1}, also contained (mg L^{-1}): Br^- – 292, J^- – 226, SO_4^{2-} – 194.4, NO_3^- – 3.4, Si^{4+} – 15.2, K^+ – 260, Ca^{2+} – 1,638, Mg^{2+} – 840, Fe^{2+} – 2, NH_4^+ – 31.6, and Sr^{2+} – 77.2. The tap water (produced from surface water) had the following average ionic composition (mg L^{-1}): K^+ – 7, Na^+ – 29, Mg^{2+} – 18, Ca^{2+} – 65, HCO_3^- – 160, Br^- – 0.15, SO_4^{2-} – 88, NO_3^- – 1.3, Cl^- – 55.

3. Results and discussion

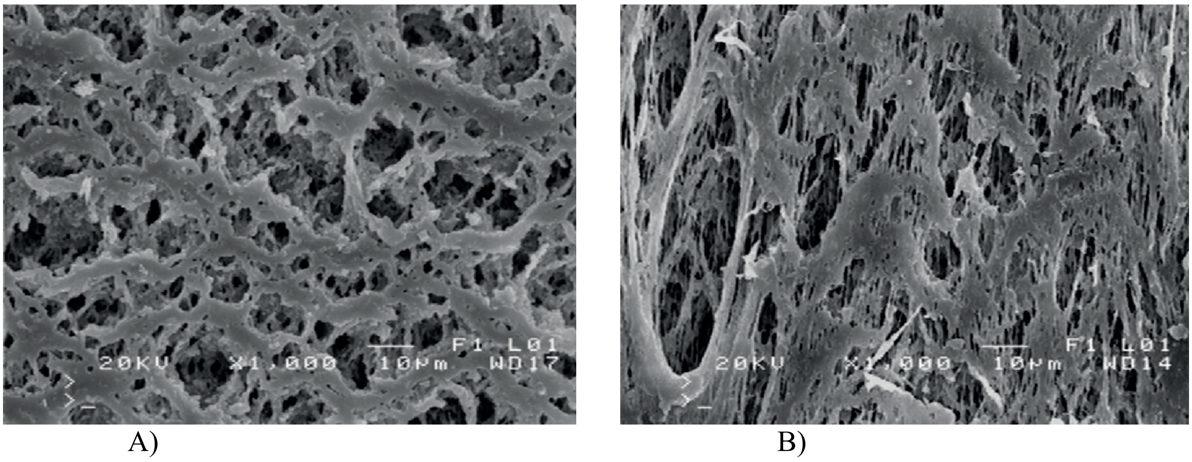
3.1. Membranes morphology

The microporous structure of applied capillary membranes was analysed based on SEM studies results. Observations confirmed that membranes exhibited significant differences in their structures (Figs. 2–4). All membranes had sponge-like structures. The size of pores located on the membrane surface inside capillaries (lumen side) was significantly smaller than that on the external capillary surface (Fig. 2). The largest differences in the surface pore size were observed for Accurel PP S6/2 membranes. In this case, several pores observed on the external surface were larger than 10 μm (Fig. 3). Such large surface pores are quickly wetted during MD process, what can initiate membrane scaling [22,27,33]. The surface wettability of Accurel PP membranes was observed after about 50 h of MD process duration [34].

Pore sizes inside the wall of applied capillary membranes were found to be significantly smaller, more uniform and similar to those presented in Fig. 4. Such small pores are resistant to wetting; therefore, Accurel PP membranes can be used as MD membranes.

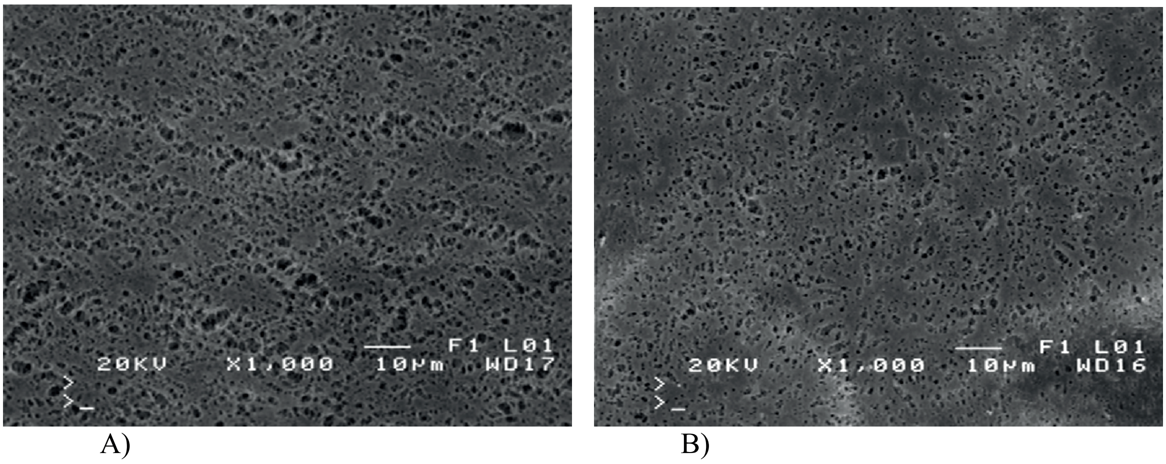
3.2. Brine separation by MD process

The concentration of readily soluble salt does not cause problems in MD process. It was confirmed by the results presented in Fig. 5, in which capacity and efficiency of module MD1 supplied by diluted NaCl solutions is shown. It was found that changes in the permeate flux were small at the beginning of DCMD process. The flux decreased from 678 to 615 L m^{-2} 24 h after 35 h, while after 100 h of process duration



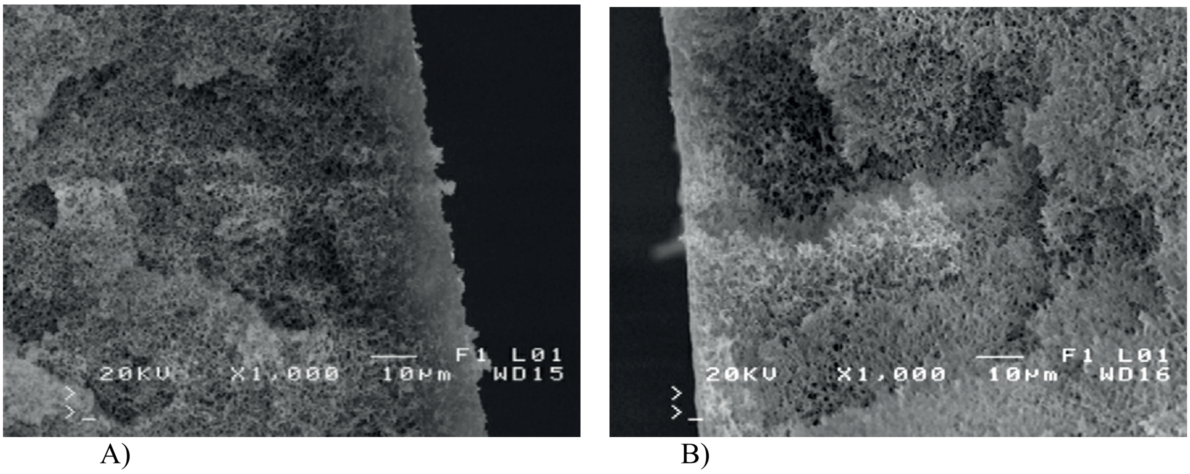
A) B)

Fig. 2. SEM images of the external surface of polypropylene membranes: (A) Accurel PP S6/2 and (B) Accurel PP V8/2 HF.



A) B)

Fig. 3. SEM images of the internal surface of polypropylene membranes: (A) Accurel PP S6/2 and (B) Accurel PP V8/2 HF.



A)

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Fig. 4. SEM images of the cross-section of polypropylene membranes: (A) Accurel PP S6/2 and (B) Accurel PP V8/2 HF.

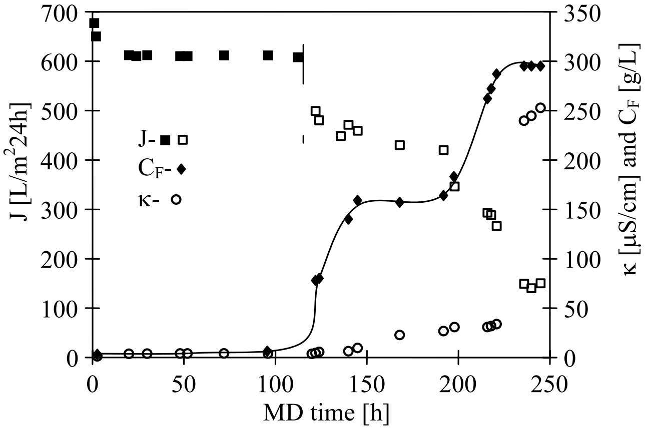


Fig. 5. Changes of permeate flux and distillate electrical conductivity during the process run. Feed: 1 g NaCl/L (0–120 h) solution and ground saline water (120–250 h). Module MD1.

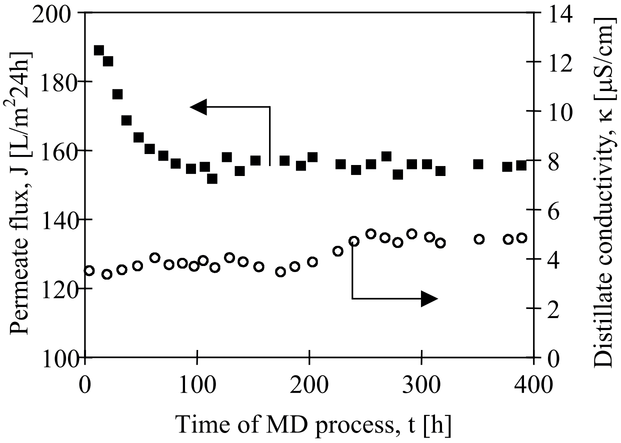


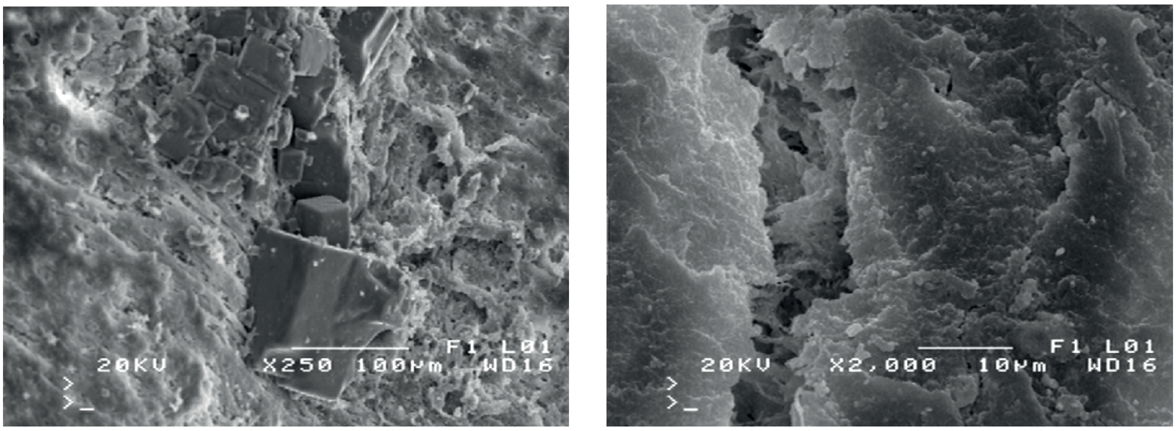
Fig. 6. Changes in permeate flux and electrical conductivity of distillate during long-term ground saline water desalination. Module MD2. Constant salts concentration in the feed (140–150 g NaCl L^{-1}) starting at 100 h.

the permeate flux was equal to 611 L m^{-2} 24 h. MD process performance for 120 h resulted in salt concentration increase in feed from 1 to 5 g NaCl L^{-1}, and as result, the electrical conductivity of the distillate was increased from 4 to 10 μS cm^{-1}. Such an increase of the electrical conductivity value indicated that same pores in the Accurel PP S6/2 membranes were wetted, what was also the main reason of observed slight decline of MD1 module yield. Most probably, at the initial period of module exploitation, the largest pores with the dimensions exceeding 10 μm (Fig. 2(A)) were wetted.

Ground saline water was used as a feed in subsequent experiments, after MD process of NaCl solution. At such feed exchange, permeate flux decreased to 500 L m^{-2} 24 h. Pure water was transferred through pores of the membrane from the brine; thus, twofold increase of salts concentration in feed was observed. Subsequent MD process was carried out within 150–200 h at a constant salt concentration in the feed. The permeate flux was stabilized at 440 L m^{-2} 24 h. The results confirmed that a decrease of efficiency of the desalination process from 500 to 440 L m^{-2} 24 h was mainly caused by an increase of salt concentration in the feed. At the last step of desalination experiments (Fig. 5), the amount of salt in the feed was concentrated to 300 g L^{-1}, and the permeate flux decreased to 165 L m^{-2} 24 h. At such high concentration of salt in the feed, the electrical conductivity of permeate was equal to 250 μS cm^{-1}.

The results of ground saline water desalination with Accurel PP V8/2 HF are presented in Fig. 6. The amount of salt in the feed was concentrated twofold during the first 100 h, and the permeate flux decreased from 200 to 160 L m^{-2} 24 h. In this case, the obtained permeate flux was smaller than the one obtained for module with Accurel PP S6/2 membranes, membrane walls of which were over four times thinner. Although an increase in the membrane thickness reduces heat loss due to the thermal conductivity, the resistance of vapour diffusion through a membrane increases with an increase in the wall thickness. Therefore, the yield of MD process with MD2 module was poorer.

During desalination of brine, an increase in the electrical conductivity from 3.8 to 5 μS cm^{-1} was observed between start point (100 h) and 250 h of MD2 module operation (Fig. 6), and a value of 5 μS cm^{-1} was maintained during the next 150 h. Such low value of distillate conductivity suggested that the membranes wetting was limited. It was confirmed by the fact



A) B)

Fig. 7. SEM images of deposit formed on the membrane surface during ground saline water desalination: (A) module MD1 – Accurel PP S6/2 and (B) module MD2 – Accurel PP V8/2 HF.

that during desalination of brine the permeate flux decreased from 200 to 160 L m^{-12} 24 h, and this value was kept almost constant during the last 150 h of MD process operation.

3.3. Membrane scaling

SEM observations confirmed that a layer of deposit was formed on the membranes surface during exploitation of MD modules (Fig. 7). Observations indicated that a deposit covered the entire surface of membranes, what resulted in a decline of the membrane permeability. Thus, the observed decrease of permeate flux (Figs. 5 and 6) resulted not only of the salt concentration in the feed but also of scaling and, most probably, of the wettability of certain pores located in the surface coating layer of the membrane wall (surface wetting). Similar results were observed during other long-term investigations [7,11,30,33].

The studies of chemical composition of deposit formed in MD modules, carried out by SEM-EDS method, demonstrated the presence of the following elements: Si, Na, Ca, Fe, Cl and S. The deposit was mainly formed by silicon compounds. Moreover, small amounts of calcium ions present in the deposit indicated that $CaSO_4$ was also probably formed. After studies of brine separation by MD, the used modules were rinsed with distilled water; thus, residues of feed and solutes (e.g., NaCl) could be removed from the membrane module. However, SEM-EDS point analysis confirmed that cubic NaCl crystals were still present, as it is shown in the centre of Fig. 7(A). Most probably, NaCl solution filled the scaling layer and wetted pores, and NaCl crystals were formed during the natural drying of the membrane samples performed before SEM studies.

If scaling layer is also formed inside the membrane wall, then the thickness of used membrane should be significantly larger than that of the deposit layer, what allows maintaining the air gap inside the membrane wall. In this case, enhancement of the wall thickness allows increasing the resistivity of membranes for leakages, even in the case of very intensive scaling caused by $CaSO_4$ crystallization [27]. Regarding such the dependence, Accurel PP V8/2 HF membranes, wall thickness of which was more than three times larger in a comparison to Accurel PP S6/2 membranes, seemed to be more promising for water desalination [21].

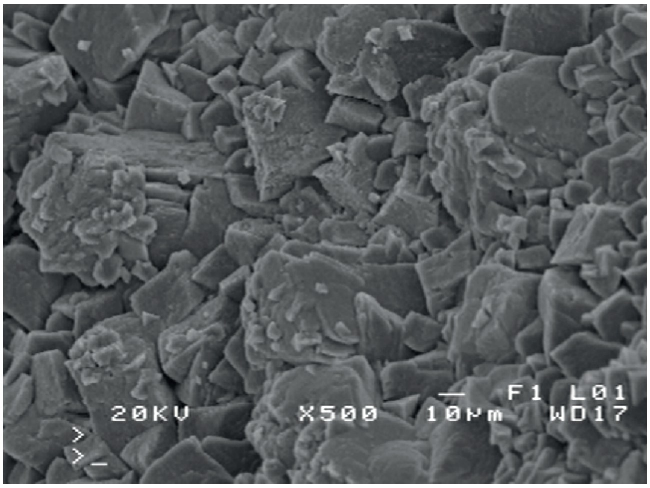


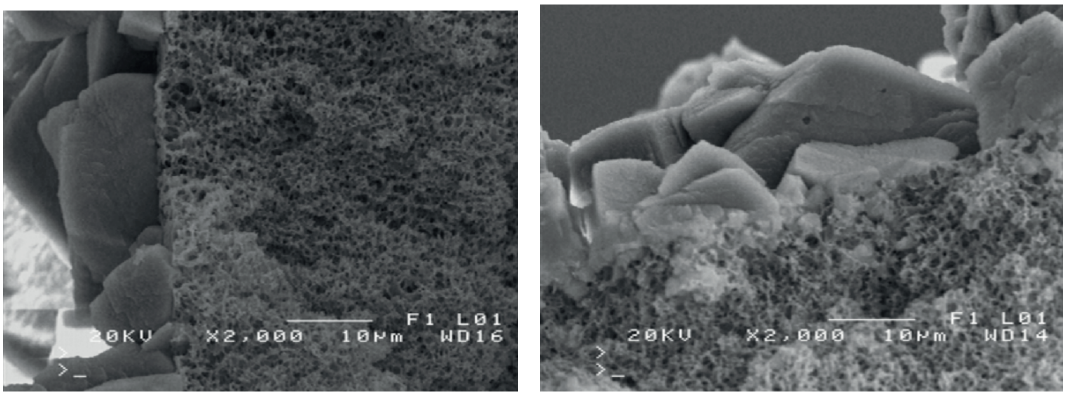
Fig. 8. SEM image of $CaCO_3$ deposit on the Accurel PP S6/2 membrane. Module MD3.

The rate of liquid penetration inside pores can be reduced by an increase of hydrophobicity of membrane material, or by a decrease of pores diameter [3,9,33]. Therefore, Accurel PP V8/2 HF membrane, which had slightly smaller pores, should be more resistant to pores wettability.

Natural water contains significant amounts of bicarbonates. Heating of water causes their decomposition into carbonates and CO_2. At typical MD conditions, bicarbonates decomposition plays the dominant role in calcium carbonate precipitation [28,31]. Precipitation of calcium carbonate in heated natural water is governed by the following reactions:

$$2HCO_3^- \leftrightarrow H_2O + CO_2\uparrow + CO_3^{2-}$$
$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3\downarrow$$
$$2HCO_3^- + Ca^{2+} \rightarrow H_2O + CO_2\uparrow + CaCO_3\downarrow$$

MD investigations with tap water as a feed confirmed that a layer of deposit was formed on the membrane surface (Fig. 8). In the performed experiments, the point at which concentration of salts in tap water was two times increased,



A) B)

Fig. 9. SEM image of membrane cross-section with $CaCO_3$ deposit: (A) after 40 h of MD process – module MD3 and (B) after 130 h of MD (process with cyclic module rinsing with 3% HCl) – module MD4.

it was replaced by a new portion of tap water (Mode I). It was found that after 40 h of experiment, the thickness of deposit layer reached about 10 μm (Fig. 9(A)).

The presence of deposit layer on the membrane surface significantly reduced the process yield (module MD3) from 600 to 290 L m^{-2} 24 h. The deposit of carbonates could be however removed by rinsing the membrane module with 3 wt.% HCl solution, what enabled almost complete recovery of the membrane initial efficiency. However, after third performance of the cleaning, the electrical conductivity of distillate increased from 4 to 8.9 μS cm^{-1}, whereas the maximum permeate flux decreased to 520 L m^{-2} 24 h. Such results pointed out on wetting of some pores of the hydrophobic membrane. It was found that deposits precipitated on the membrane surface facilitated the wetting of pores, which were in the direct contact with the deposit [28,33]. The pores wetting was probably a reason of deposits formation, not only on the membrane surface (Fig. 9(A)) but also inside pores (Fig. 9(B)). During the subsequent rinsing of module using HCl solution, deposits were dissolved, although pores were filled by HCl solution, what could accelerate wetting of the hydrophobic membranes.

4. Conclusions

The performed studies on MD process demonstrated that 99% salt rejection was achieved for saturated NaCl solutions, which confirmed that the technique could be used for brine desalination.

A decrease of the membrane wall thickness significantly improved the obtained permeate flux. However, it was found that the liquid systematically wetted the consecutives pores during MD process; hence, the thickness of the air-layer inside the membrane wall was reduced. It allows to conclude, thin walls membranes will be wetted in a relatively short time. Therefore, hydrophobic membranes of thicker walls are recommended for commercial DCMD applications, especially when the scaling phenomenon occurs.

Acknowledgement

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