ARCHIVES
OF THERMODYNAMICS

Experimental study of the potential of concentrated NaCl solutions for use in pressure-retarded osmosis process

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Abstract

Pressure retarded osmosis is a process that enables useful work generation from the salinity difference of solutions. The literature most often considers using pressure retarded osmosis with natural sodium chloride (NaCl) solutions, such as seawater, dedicated for open systems. To explore the full potential of this process, however, optimized, highly concentrated solutions of various compounds can be used. The presented research is focused on evaluating the impact of increasing draw solution temperature and concentration on the permeate flow in the osmotic process. The permeate flow is directly related to achievable work in this process, therefore, it is important to find feed and draw solution parameters that maximize it. An experimental setup developed in this study provides full control over the process parameters. Furthermore, the performance characteristics of the membrane over process time were investigated, as it became evident during preliminary experiments that the membrane impact is significant. The studies were conducted without back-pressure, in a configuration typical of the forward osmosis process, with solution circulation on both sides of the membrane. The obtained results show a clear positive impact of both the temperature and concentration increase on the potential output of a pressure retarded osmosis system. The membrane behaviour study allowed for correct interpretation of the results, by establishing the dynamics of the membrane degradation process.

Keywords: Pressure retarded osmosis; Renewable energy; Experimental investigation; High-concentrated solution; Increased temperature effect

1. Introduction

World energy use is still largely based on fossil fuels, which are closely linked to emissions of pollutants and carbon dioxide, affecting the state of the ecosystem. As the world's energy demand continues to grow, energy conversion methods previously not used on a large scale are gaining interest. One of these methods is extracting useful power from the difference of concentrations in solutions based on thermodynamic potential (i.e., Gibbs free energy). There are two most promising basic ways of utilization of this potential, i.e., pressure-retarded osmosis (PRO) and reverse electrodialysis (RED), which can be used to generate useful power from salinity difference.

The RED uses membranes for the ions, but not water, transport. Two types of ion-selective cation-exchange membrane (CEM) and anion-exchange membrane (AEM) are used [1]. A number of these membranes are arranged in a variable

pattern between a cathode and an anode. The channels between the membranes are alternately filled with highly concentrated salt solution and diluted salt solution, which flow along the membranes. The difference in electrochemical potential resulting from the difference in salinity causes the transport of ions through the membrane from the concentrated solution to the diluted solution. In a sodium chloride solution, sodium ions permeate through CEMs towards the cathode, and chloride ions permeate through AEMs toward the anode. Electro-neutrality of the solution in the anode cell is maintained by oxidation on the surface of the anode. Electro-neutrality of the solution in the cathode cell is maintained by reduction on the cathode surface. The electric potential difference builds up between two neighboring electrodes. This potential difference allows an electron transfer from the anode to the cathode via an external electrical circuit when the external load is connected to the circuit. The main advantage of using the RED system for obtaining useful energy is that electricity is generated directly from salinity differences between neighboring channel streams. Each pair of membranes generates a voltage of around 0.1–0.2 V from typical river and sea water [1]. It was found that in order to overcome energy losses at the electrodes, at least 20 pairs of membranes are needed. Substantial advances are being made to increase RED power density and energy efficiencies by improving membrane materials, spacing and architecture. The highest power density that was attained for the RED stack with 50 cell pairs was 0.93 W/m^2 at about 3 V, neglecting electrode and pumping losses (based on each type of ion-exchange membrane having a 0.5 m^2 total surface area, or 93 W/m^2 based on the planar cross-sectional area between the electrodes [1]). The main challenge for the commercialization of RED is the cost of ion-exchange membranes, but a global increase in demand leads to a price decrease. Over the past two decades, new materials, improved fabrication methods and increased production of membranes have resulted in a decrease in their costs. But they are still very high, and the RED technology is quite expensive.

The PRO process enables the generation of useful work from the concentration difference of solutions separated by a semipermeable membrane. In the osmosis process, the system tends to equilibrate by equalizing the concentrations on both sides of the membrane. The only way to reach this goal is for the solvent to flow toward the highly concentrated solution, resulting in its dilution. The governing variable of this process is osmotic pressure. It is defined as the minimum pressure needed to stop the osmotic process completely. By applying pressure lower than the osmotic pressure to the concentrated side, one may partially retard the flow of solvent, but in effect, the permeating solvent now has the potential to perform useful work [2], i.e., when passing through the membrane, it increases its pressure to that applied on the concentrated side. There are two other well-known osmotic processes that have elements in common with PRO, i.e., forward osmosis (FO) for wastewater treatment [3] and reverse osmosis (RO) for water purification [4], but these processes are implemented differently and have different objectives than PRO.

The literature most often considers using PRO with natural sodium chloride (NaCl) solutions, such as seawater and river water, or industrial solutions, e.g., seawater desalination concentrate [5]. With the average salinity of seawater in the range of 35 g/kg and the assumption of negligible salinity of clean water, such a system can provide approximately 0.6 kWh of energy per cubic meter of freshwater used. The osmotic pressure of standard seawater is at the level of 0.27 MPa [6]. Moreover, in the vast majority of the papers, only open systems are analyzed depending on the available source of streams feeding the system. The schematic of such a system is shown in Fig. 1a. In these systems, the full potential of PRO as a power generation method cannot be explored due to existing parameter limitations of natural and industrial NaCl solutions.

The second approach, which is less common in scientific publications, is a closed PRO system, shown in Fig. 1b, that allows more extensive manipulation of the parameters of the feed and draw streams, as well as the use of optimized synthetic solutions [7].
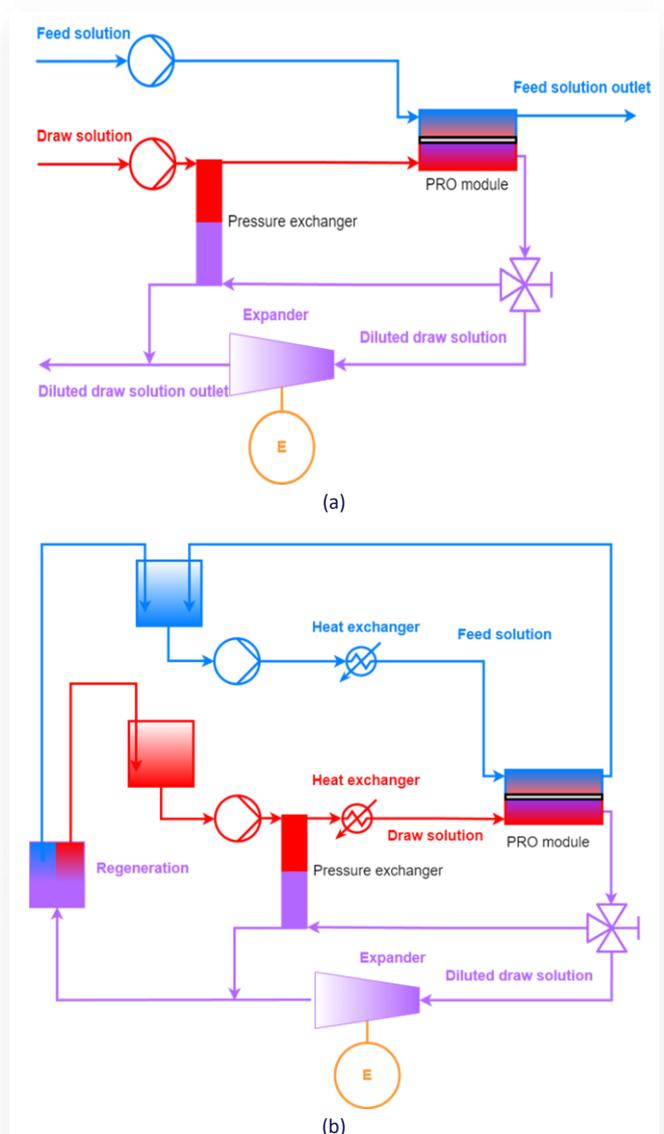

Fig. 1. Schematic of open (a) and closed (b) PRO system.

Alternative engineered solutions have more advantageous properties, such as high solubility and high osmotic pressure, allowing for greater flow of the solvent through the membrane, i.e., producing much higher power densities [8]. To maximize the osmotic pressure, the molecular mass of the solvent should be as low as possible. The selected solutions should also minimize separation energy requirements during the regeneration process, i.e., they should have a high recovery rate using low-grade heat. The considered solutions are, among others, inorganic and organic salts, aqueous solutions of very soluble ammonia salts, diluted ammonia-carbon dioxide solution in water, and aqueous solutions of ammonia hydrocarbons [8–10]. Therefore, the application of a closed PRO system allows optimization of the process depending on the intended applications. Moreover, such systems operate without any inflow of freshwater and require the draw solution regeneration. Methods of regeneration may be divided into thermal and mechanical. Thermal methods use external heat sources to separate components of the solution. A few such methods of solution regeneration have been proposed, e.g., distillation, separation using critical temperatures (over which some liquid vapours become miscible or immiscible), and separation using differences in solubility of dissolved species in a solvent for different temperatures. In essence, the external heat input, required for solution regeneration, can be converted to mechanical energy, resulting in an osmotic heat engine (OHE) [10]. Such an engine may utilize low-grade renewable energy heat sources, such as solar [11] and geothermal energy [12] or waste heat, for solution regeneration and heating of solutions in the PRO cycle, which may also increase the process performance [9]. Additionally, a closed PRO system does not require access to saltwater and freshwater in close proximity, and therefore, its environmental impact and constraints are limited.

The main problem with the PRO and its commercial applications is the low power density per square meter of the membrane when seawater or low-concentrated solutions are used. One of the ways to overcome this problem may be the application of high-concentrated or saturated solutions as working media. Several studies in which hypersaline agents were applied, including the pilot-scale facilities, were described in the work of Bajraktari et al. [13]. Very high power densities per square meter of the membrane are obtained for such working media due to the significant rise in the osmotic pressure and water flux through the membrane with increasing salt concentration. The power density was reported to increase at a higher rate than the draw solution concentration, e.g., by doubling its salinity, power density rises with factors between 2.5 and 3.75 [13]. However, most working media that came from natural resources (i.e., hypersaline lakes, salt domes, hypersaline geothermal water, etc.) and technological processes (industrial brines, e.g., from destination process, brine wastewater, etc.) were considered in the open PRO systems. These media have a NaCl concentration of up to 3 mol/dm³, which is far below the NaCl saturation point in the water (c.a. 5.36 mol/dm³ at 20°C) [12].

So far, the studies encountered in the literature are mostly focused on investigating solution concentrations corresponding to the standard seawater concentration in open PRO systems

[14]. While some laboratory- and pilot-scale studies were conducted on open PRO systems [15], the studies presented to date regarding closed-loop PRO systems are purely theoretical [16].

To fill the apparent gap in the scope of studies published to date, the studies presented in this paper have been extended to include highly concentrated solutions (i.e., at salt concentrations much higher than those used in the literature), allowing the maximum theoretical potential of the reagent to be determined for future applications in closed PRO systems. The experimental setup designed for the purpose of this study mimics the layout and behaviour of a closed-loop PRO system on a laboratory scale. This enables an investigation of the process in conditions similar to those encountered in future real systems. In addition, determining the effect of solution temperature on process dynamics will allow better prediction of the behavior of such systems under varying operating conditions in order to investigate the possibilities of using low-grade heat to improve the efficiency of PRO systems. The experimental study also makes it possible to detect potential technical problems resulting from increasing the concentration and temperature of working solutions, which are difficult to predict in theoretical analyses. The overall novelty of this study is that it undertakes the development of a methodology for the investigation of closed-loop PRO systems, operating on highly concentrated solutions at elevated temperatures, taking advantage of available heat sources to increase the power density of the setup to the highest possible level.

The research described in this paper focuses on evaluating the effects of the concentration and temperature of NaCl solutions on the flux of the solvent (water) flowing through the semipermeable membrane, which is directly related to the possible useful work generated in the PRO process. The study was conducted without back pressure (i.e., a configuration typical of the FO process), forcing only the flow of the media on both sides of the membrane.

Additionally, the preliminary results of experiments suggested that there is a need to investigate the behavior of the osmotic membrane. A significant decrease in the permeate flow between consecutive measurements led to the conclusion that membrane degradation may play a crucial role in evaluating the PRO process performance. Therefore, the study was extended to incorporate a long-term measurement performed in the reference conditions, designed specifically to investigate the membrane performance itself.

2. Description of the method and materials used

2.1. Test solution

Analytical grade NaCl and deionized water were used to prepare the draw solution. The salt was previously dried in a vacuum dryer. A solution with a concentration of 35 g NaCl/kg of the solution, which corresponds to the standard concentration of seawater, was chosen as the test baseline (reference solution). A concentration twice as high was chosen as the next measurement point, since the increase in osmotic pressure of the solution with its concentration is linear over the assumed measurement range. To determine the maximum potential of the solution,

a concentration of 211 g/kg of solution was investigated, corresponding to 80% of the maximum solubility of NaCl salt in water at 20°C. The saturated solution was not used since crystallization of the salt in the system at points of reduced temperature could lead to the introduction of the solid fraction to the pump and osmotic module, potentially leading to damage to these components. The solution was prepared in batches of 10 kg, and the resulting concentrations were verified by density measurements (METTLER TOLEDO, Densito, accuracy ±0.001 g/cm^3) and compared with literature values.

2.2. Test stand

The bench was mostly made of AISI 316 stainless steel to minimize the risk of component degradation when using highly concentrated salt solutions. The main component of the system was a specially designed and manufactured module consisting of two parts, which, when divided by a semipermeable membrane (Dupont, FilmTec XUS1203) and assembled, form two channels allowing the flow of feed and draw solutions. The parallel flow configuration of the module was used. The working area of the module was 100 × 250 mm^2, and the height of the channels was 2.2 mm. Special support inserts (i.e., spacers) printed using the Fused Deposition Modeling (FDM) method were installed on both sides to avoid membrane deformation and flow restriction. The module with the support insert installed is presented in Fig. 2.
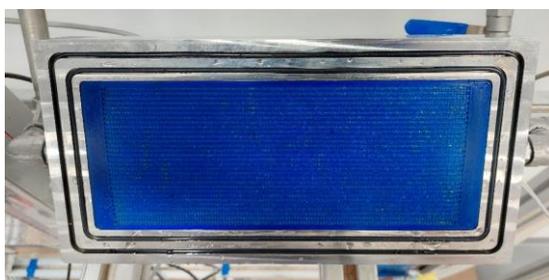

Fig. 2. Half of the membrane module with membrane support insert.

Fluid flow in the system was induced using two FOTTON FTL-222 diaphragm pumps with a maximum flow rate of 4 l/min each, powered by a 24-volt DC power supply and controlled using the method of Pulse Width Modulation (PWM). Keller PAA-33X pressure transmitters with a measurement range of 0.8-1.2 bar and an accuracy of 0.1% of the range were used to measure pressure on both sides of the module, communicating with the system via Modbus RTU RS485 protocol.

The control system, based on pressure measurement at the module outlets, ensures a zero pressure differential on both sides of the membrane (FO configuration operation) by adjusting the flow rate on the draw solution pump. Temperature control in the system was carried out by two complementary elements, i.e., a spiral heat exchanger made of a silicone hose immersed in a water bath connected to a chiller and an electric heater to facilitate the basic control and stabilization of medium temperature, and electric heaters of 300 W each placed in the flow of both

media and allowing precise control of the temperature of the solutions using the PWM method. Temperature measurements were carried out using PT100 resistance sensors connected to MAX31865 transmitters communicating with the system using the SPI protocol. The solution temperature was maintained within the range of ± 0.5°C from the set value for the experiment duration.

For medium storage, two 2 l polypropylene vessels were used. Mass changes in the tanks were measured using two RAD-WAG 3000.X2 precision balances (accuracy ± 7.5 mg, calculated as the sum of max repeatability and linearity errors), communicating over the VISA protocol. The proprietary control and data acquisition system was integrated using a Raspberry Pi 4 8GB single-board computer with a dedicated application written in Python. A diagram of the test stand is shown in Fig. 3.
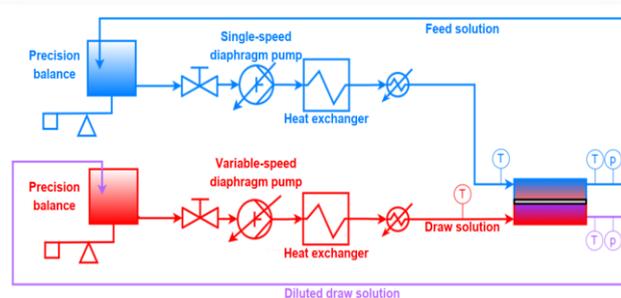

Fig. 3. Test stand schematic.

2.3. Experiment methodology

Nine cases were analyzed during the study, considering different draw solution concentrations and variable temperatures in the PRO system, as shown in Table 1. During testing, the temperature was kept equal for draw and feed solutions. The result obtained for a 35 g/kg NaCl solution at 20°C was used as the reference.

Table 1. Investigated cases.

Solution concentration (g/kg)	35			70			211		
Temperature (°C)	20	40	60	20	40	60	20	40	60

All measurements were carried out in the same configuration, using equal volumes of solutions. Before the experiment was performed, the system temperature was stabilized, and the concentration of the circulated solutions was verified by measuring their densities. Once the temperatures and pressures on both sides of the membrane were stabilized, the measurement was started and stopped automatically after a preset time of 80 minutes, identical for every case. At the end of the experiment, for a given point, the density of the feed solution was measured – to determine any salt backflow – and that of the draw solution – to determine the dilution during the process. Then, by analyzing the data, the mass fluxes of the solutions were determined. To reduce the effect of evaporation and hygroscopicity

of the draw solution, the result was calculated as the average of these fluxes. After taking into account the density of water at the measurement temperature and the membrane active area, the mass flux was converted to unit volume flux.

3. Analysis of preliminary results

Figure 4 shows the obtained data as a function of solution temperature and concentration. The permeate stream gains, however, did not match the theoretical expectations. Despite the fact that doubling the solution concentration doubles the osmotic pressure in a low-concentration range, the flow increases marginally. Strong temperature dependency is evident and reasonably consistent, which would follow in line with the expectation, i.e., the solution temperature variation affected the solution properties, such as the solution viscosity and solute diffusivity, which resulted in the rise in permeate flux with the rising system temperature. The impact of the solution concentration is not as pronounced.
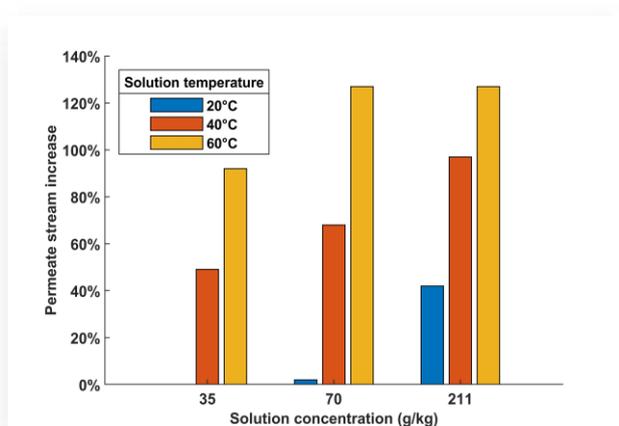

Fig. 4. Obtained flux increments as a function of draw solution concentration and process temperature with respect to the reference case (35 g/kg and 20°C).

The series of measurements was completed with an additional measurement under reference conditions. This measurement showed a significant decrease in the permeate flux compared to the first reference case. Since all the measurements were performed using the same membrane coupon, gradual degradation of the membrane in the process was suspected.

For the preliminary result correction, a linear degradation characteristic was assumed – no data collected at the time suggested a different behavior. The linear character of the degradation and its dependency on process time were checked for a solution concentrated to 35 g/kg at 20°C in a separate series of seven measurements.

The measurements in question were conducted using a single, new membrane coupon. The experiment duration was set to 80 minutes, to mimic the conditions during the actual measurement series – fixed-length runs separated by breaks for solution replenishment. Average hourly permeate flows (i.e., solvent streams through the membrane) were calculated for each experiment and the results in a normalized form (i.e., in relation to the first measurement) are presented in Fig. 5. The measured stream

of solvent passing through the membrane was converted to a volumetric rate and divided by the membrane active area, resulting in mass flow rate in liters per square meter of membrane per hour (LMH), which is the standard permeate flow measurement unit in the membrane processes.
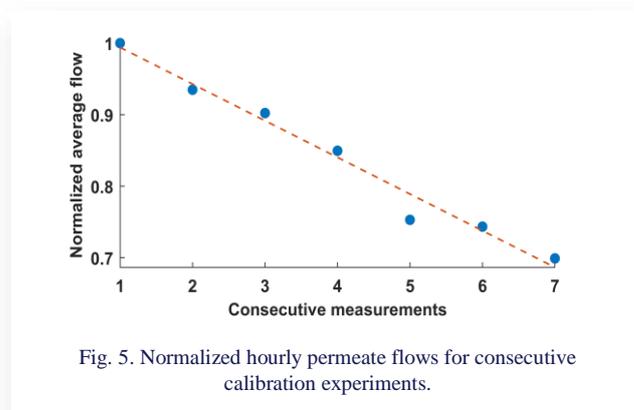

Fig. 5. Normalized hourly permeate flows for consecutive calibration experiments.

The results for each experiment run were then normalized to the flow obtained in the first measurement. It is clear that the mass flow rate is consistently reduced, and its characteristic over time is approximately linear. The average permeate flow reduction was calculated to be in the range of 5% per measurement, and this value was used as a correction factor in the post-processing of preliminary results for other solution concentrations and temperatures, further described in section 5. This reduction rate is consistent with the results of the first and last measurements of the main experiment series, conducted in reference conditions. Thus, an assumption can be made that the rate of flow reduction is constant in the examined case, and the impact of temperature and solution concentration is negligible.

4. Membrane behaviour study

A membrane behaviour study was undertaken to complement the previous step reference measurements and better understand the degradation process. The principal idea was to perform a long-term measurement on a single membrane sample and observe the permeate flux over time. 100 hours was chosen as a reasonable time scale for such an experiment. The solution used was kept at reference conditions – a concentration of 35 g/kg and a temperature of 20°C. Similarly to the first experiment, the draw solution concentration and deionized water purity were assessed at the beginning and end of each experiment step by density measurements. Temperature and pressure stabilizations were achieved before each step.

Naturally, the draw solution dilution is an important factor that is changing the permeate flux. To minimize its impact, the draw solution reservoir was expanded, so that the total mass of the draw solution in the system was kept over 15 kg at any time in a 20 l polypropylene vessel. The mass change was measured only at the feed solution side. Additionally, the measurement was performed in three distinct steps, between which the solution was regenerated by salt addition – bringing its concentration back to the reference values. With these precautions, the dilution did not exceed 7% at the end of each experiment step.

Results of the membrane behaviour study are shown in Fig. 6. The divisions between the steps of measurements are clearly visible. The flow increase at the beginning of each step is be-
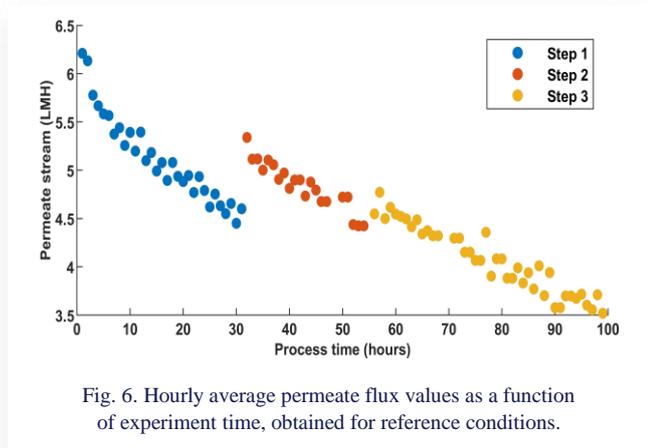

Fig. 6. Hourly average permeate flux values as a function of experiment time, obtained for reference conditions.

lieved to be caused by the membrane settling after the test rig was turned off and on again. A diminishing flow trend is evident.

In every measurement, after the initial settling, the trend becomes linear. Despite increasing the solution concentration between steps, the general trend is also negative. Significantly longer measurements would have to be carried out to confirm or deny the existence of any plateau, i.e., whether the flow decreases consistently to a complete blockage or settles at an ultimate, stable level. However, in the proposed time scale, one should assume that the characteristic is macroscopically linear.

5. Final results

The linear degradation assumption proposed in section 4 was applied to the preliminary results shown in section 3. The corrected unit volume fluxes flowing through the membrane were compared with the flux obtained under reference conditions. The obtained increments are shown in Fig. 7.

A strong correlation between the solution temperature and the process dynamics is evident and consistent for individual concentrations. The impact of concentration on the obtained flux is less pronounced, but it still seems reasonable to use solutions
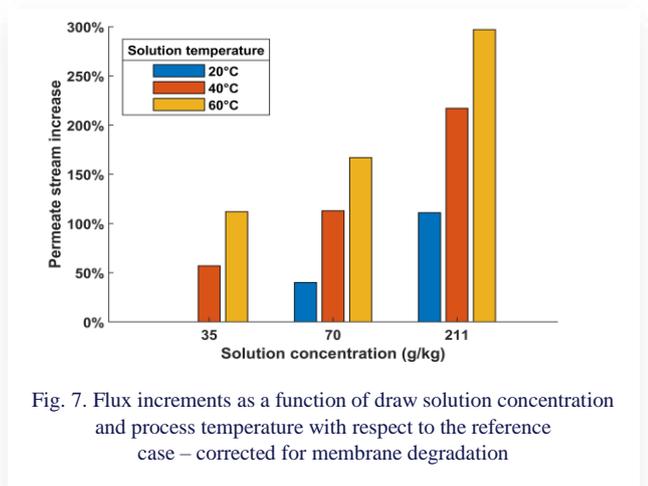

Fig. 7. Flux increments as a function of draw solution concentration and process temperature with respect to the reference case – corrected for membrane degradation

with the highest practically applicable concentration. The performance characteristics obtained also allow for a conclusion that increasing the temperatures of the working solutions provides measurable performance benefits in osmotic systems, while reducing the risk of crystallization occurring at high concentrations of the draw solution. In order to verify these assumptions, further studies should confirm the independence of membrane degradation characteristics from temperature and solution concentration. In conclusion, the study showed that the low-grade heat source can be used not only for solution regeneration in the closed PRO systems but also for increasing the temperatures of solutions to improve the overall efficiency of the process.

6. Conclusions

The experiments, conducted in a lab-scale osmotic system, allowed for establishing the characteristics of permeate flow as a function of solution concentration and temperature. A highly concentrated NaCl solution was used without any unforeseen problems, providing a noticeable increase in the system performance. Doubling of the permeate stream was achieved with the increased draw solution concentration of 211 g/kg, without increasing its temperature. Thus, a viable potential of highly concentrated solutions in real conditions was confirmed. The rise in temperatures of solutions also positively impacted the membrane water flux, showing the potential for using low-grade heat sources to heat the solutions. The temperature impact is especially prominent, as in the case of the highest solution concentration, the temperature increase by 20°C provided a flow increase of over 200% compared to the reference case, and a further 20°C increase allowed an increase of nearly 300%. Solution heating may be a viable, low-tech and high-impact way of increasing the power output of PRO systems even without increasing the concentrations, provided that suitable waste or low-grade heat sources are available on site. Overall, the results show that the power density of the PRO process can be improved with the introduction of closed-loop systems utilizing highly concentrated solutions. Furthermore, available heat sources can be used to increase the power density further by increasing the solution temperature. This is a promising sign for the development of such systems, as the power density is not dependent only on the available membrane surface and can be maximized within the existing space restrictions. However, the membrane degradation was observed during the measurements and investigated in separate experiments. The results helped estimate the degradation dynamics and thus evaluate data from the first experiment set. The membrane behavior warrants further study to establish the process dynamics in a broader temperature and concentration range and to investigate the possible causes of this phenomenon. So far, the existence and basic characteristics of the effect have been established. Further studies will be conducted to evaluate the osmotic potential of other prospective solutions, i.e., organic salt solutions, using the methodology presented in this work.

Acknowledgements

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