

Design and results of a first generation pilot plant for supercritical water desalination (SCWD)

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ABSTRACT

A pilot plant of 5 kg/h based on the principle of supercritical water desalination (SCWD) has been designed, built and operated. The detailed design, operating procedures and performance of the plant is presented in this paper, along with the first results. Firstly, the plant has been tested for feed streams of 3.5 wt% NaCl to evaluate the stability and repeatability of the system, with the results indicating that mass balance closure is good and that reproducible results can be obtained. Furthermore, the results showed that 93% of the feed is recovered as fresh drinking water, which corresponds with expected results from phase equilibria simulations. The plant was further tested for higher feed concentrations of up to 16 wt% NaCl. For all feed concentrations, the NaCl concentration of the SCW was that of drinking water quality (< 600 ppm). Experimentally, using a single stage separator, a concentrated brine (38 wt% NaCl) was obtained and calculations showed that with a two-stage flash-evaporation scheme, zero liquid discharge (ZLD) can be obtained. Further modifications to the plant and tests with other salt mixtures are recommended in order to advance to industrial application.

1. Introduction

The demand for freshwater has risen in recent years due to the global population and industrialisation. The result is that many regions face a shortage of fresh drinking water and are not able to meet the current/growing demand from the existing freshwater resources [1,2]. For this reason, desalination technologies have been developed to convert saltwater to freshwater. In the past 20 years the global desalination capacity has increased with 58 million m³/d, with the capacity being 86.6 million m³/d in 2015 [3,4]. Desalination technologies are not only limited to seawater purification, but are also used to treat brackish water, river water, wastewater and brine. The purified water is used in numerous sectors including various industrial applications, power stations and tourism with municipal applications being the majority user. Conventional desalination technologies include reverse osmosis (RO), multi-stage flash distillation (MSF), multiple-effect evaporation (MEE) and electrodialysis (ED), with RO and MSF being the most used technologies [2]. Even though these technologies are well established, there are certain drawbacks such as low water recovery, $\pm 45\%$ for seawater RO (SWRO) [5,6] and $\pm 50\%$ for thermal processes such as MSF and MEE [6–9]. Parallel to the production of freshwater, these processes also generate brackish waste streams, which need to be managed. One of the major problems with the treatment of

the brine is the disposal into the oceans by coastal plants, which disrupts the ecosystems and threatens marine life. For plants located inland the brine streams are first treated before being discharged into bodies of water [1,2,10–12]. In recent years more stringent regulations have been put into effect for discharging the brines and for this reason zero liquid discharge (ZLD) technologies have been investigated and further developed [13,14]. ZLD technologies provide a manner to eliminate the production of brine streams, thereby reducing the impact of desalination on the environment. Supercritical water desalination (SCWD) is a technology, that presents a novel method of separating inorganic compounds from brackish water, with the potential of being ZLD technology [15,16].

At supercritical conditions (Pressure > 22.5 MPa, Temperature > 374 °C) the properties of water start to change significantly. The density of water decreases, which reduces the strength of the hydrogen bonds, causing the water to become non-polar. Consequently, the solubility of inorganic compounds reduces, which results in the formation of solid salt in supercritical water [16–18]. The removal of different inorganic salts from supercritical water has been extensively investigated in previous studies with various laboratory scale set-ups [19–26]. In our previous work [15], the potential of SCWD was investigated and presented. The phase equilibria of a NaCl–H₂O solution were measured on a laboratory scale set-up and the results

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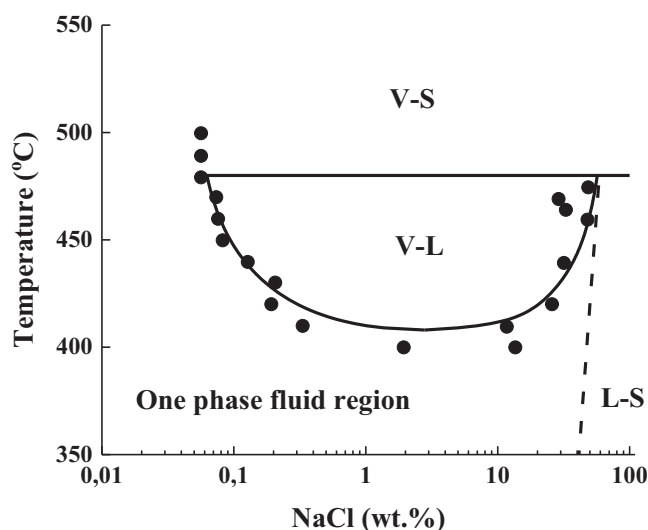


Fig. 1. Phase diagram of NaCl-H₂O system at 300 bar (- Anderko & Pitzer model [28]; - liquid-solid line [29]; ● experimental data [15]).

were used to illustrate the proof-of-principle of a two-stage separation process for SCWD. The first stage of separation is the continuous removal of supercritical water (SCW) from a liquid brine solution and occurs in the vapour-liquid (V-L) region at supercritical conditions (see Fig. 1). The next stage is the batch-wise separation of the liquid brine into salt and steam at subcritical conditions (100 °C and 1 bar). Additionally, heat integration, material selection and the controlled removal of salts at supercritical conditions was discussed. In a separate paper [27], the heat transfer mechanism of SCW flow at the mass fluxes and conditions expected in the SCWD pilot plant was investigated and used to design a heat exchanger for the pilot scale SCWD unit.

Recently, Ogden & Tremblay [30] published results of a prototype Joule-heating desalination system (feed rate 6.1–6.7 kg/h brine), in which they experimentally investigated the thermodynamic properties of multicomponent brines at operating temperatures of 387 to 406 °C and pressures of 230 to 280 bar. In their paper, they present a plant based on Joule-heating, in which the brine is directly heated within the reactor, instead of externally as with the pilot plant introduced in the present paper. Also, the liquid brine is used to heat the feed to the unit, instead of the SCW.

Based on the experimental data and findings of our previous two papers [15,27] a first generation SCWD pilot plant was designed and built. In this paper, the design and layout of the plant will be presented and the performance will be evaluated. First experimental results for a feed of ± 3.5 wt% NaCl will be presented. Furthermore, the findings for higher saline feed (7–16 wt%) experiments will be given and discussed. The aim is to show the viability of the first generation SCWD process for the desalination of brackish water and its applicability for different concentration brine streams encountered in convective desalination processes.

2. Pilot plant layout and key units

The pilot plant was designed and built at the high pressure laboratory of the University of Twente. The unit is located inside a concrete safety bunker and is fully automated so that it can be controlled from the outside during operation. The pilot plant has a maximum capacity of 8 kg/h of feed and was designed to operate at a maximum pressure of 380 bar and maximum temperature of 500 °C. The layout of the pilot plant is shown in Fig. 2.

From Fig. 2, it is seen that the pilot plant consists of several units, which can be divided into three sections namely the heat exchanger (HEX), gravity separator and brine recovery units (salt collector to

condensed vapour collector) (Sections 2.1 to 2.3). In addition to these units, there are two feed vessels and two product vessels. The first vessel contains demineralised water fed during the start-up and shut down of the system, while the other contains the saline feed. The first product vessel is for the collection of the drinking water coming from the gravity separator, while the second vessel is for the collection of the vapours produced during brine expansion. All vessels are placed on weighing balances to monitor the in- and out flow of the unit and to check the mass balance closure. A high-pressure LEWA diaphragm pump LDC1 (LEWA Herbert Ott GmbH & Co KG, Germany) is used to pressurise the saline feed. The pump has a maximum capacity of 25.0 L/h (with an uncertainty of 1% in mass flow measurements), a maximum operating pressure of 400 bar and is applicable to a wide variety of fluids including saline solutions. The feed pressure is set and controlled with a back pressure regulator, BPR-1 (TESCOM 26-1762-24A, Tescom Europe GmbH & Co. KG, Germany, $C_v = 0.14$).

2.1. Heat exchanger

Owing to the extreme operating conditions of SCW, the process is energy intensive and heat integration is required to make it commercially viable. For this unit, heat integration is achieved by utilising the SCW product to heat the saline feed in a heat exchanger that can operate from sub- to supercritical conditions. The feed will change from sub- to supercritical conditions, while the SCW will transition from super- to subcritical conditions. Prior to the design and construction of the heat exchanger, heat transfer characteristics of SCW was investigated through modelling in COMSOL Multiphysics and validated with experimental results [27].

From the findings, a double pipe counter-current heat exchanger was designed and constructed. The enthalpies for the design were determined from the correlations of Driesner [33] for the cold streams and the IAPWS formulation for the hot streams [49]. The inner and outer tubes of the heat exchanger are constructed from grade 2 titanium, with the dimensions listed in Table 1 (see Supporting information for detailed calculations). The double pipe heat exchanger is wound into a spiral coil (diameter of 40 cm and height of 40 cm) for stability and compactness. In the event of fouling due to salt deposition, the configuration of the heat exchanger is such that the saline feed is fed through the tube side, while the SCW product flows through the annular space (shell side) between the inner and outer tubes in a counter-current manner. Salt deposition from the feed can be easily cleaned, by running demineralised water through the system. To avoid contact between the inner surface and outer tube, a 0.5 mm titanium wire is wound around the outside of the inner tube before inserting it into the outer tube. The heat exchanger is also insulated with 20 mm thick glass fibre thermal insulation rope to reduce heat loss to the environment. In Fig. 3 the wound heat exchanger and inner tube with the 0.5 mm titanium wire is shown.

2.2. Gravity separator

In order to reach the desired feed temperature, additional heat is provided by an electrical heater located before the inlet to the gravity separator. The heater is comprised of a grade 2 titanium feed tube wound around a 2.5 kW aluminium block that provides the electrical heating.

The first stage of separation takes place in the gravity separator, where the SCW fluid phase is continuously separated from the concentrated liquid brine. The separator itself is constructed from Incoloy 825 with an inner diameter of 4 cm (wall thickness, $t = 2$ cm) and a length of 50 cm. The separator is placed in a 6 kW electric oven to ensure that isothermal conditions are maintained within the separator. The saline feed enters the separator through a dip-tube ($d_i = 3$ mm, $d_o = 5$ mm, $L = 20$ cm, grade 2 titanium), while the SCW fluid exits through the top, to the heat exchanger and liquid brine accumulates at

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