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Ahmad, M., Oatley-Radcliffe, D. & Williams, P. (2017). Can a hybrid RO-Freeze process lead to sustainable water supplies?. *Desalination*

http://dx.doi.org/10.1016/j.desal.2017.10.031

Can a hybrid RO-Freeze process lead to sustainable water supplies?

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Abstract

In this paper we investigate the potential for using suspension crystallisation for the production of clean drinking water from a seawater source. The experimental results show that the produced water from the suspension crystallisation plant not only meets water quality standards but is comparable in ionic composition to premier bottled water from around the globe. The experimental results obtained from a pilot scale suspension crystallisation unit showed that the achievable water recovery was around 41% and the salt rejection ratio reached over 99%, which is comparable with most desalination technologies. Moreover, a hybrid RO-Freeze plant has been proposed that is capable of significantly increasing the potable product water that could be achieved by RO alone (~400% increase), while simultaneously concentrating the RO brine (used as feed water) producing a super brine of ~13wt%. While there is a cost to this additional process in terms of capital and energy that must be quantified, the obvious increase in water harvest and reduction in residual brine quantity lead to a very attractive desalination process. If the energy demands are acceptable, then this technology could lead to a more sustainable water future.

Keywords: desalination, freeze, brine, reverse osmosis, membranes, suspension crystallisation

Highlights:

- Suspension crystallisation has been successfully demonstrated as a desalination technology.
- The product water generated is of drinking water standards.
- A hybrid RO-Freeze process has been proposed that increases water recovery by 400%.
- Desalination brines can easily be concentrated to the order of 13w%.

1.0 Introduction

Along with energy, shelter and food, access to a source of fresh clean drinking water is essential to all life on earth. Today, the over-exploitation of existing fresh water supplies along with the increasing demand of water for drinking, agriculture and industry is generating a critical availability issue and according to the United Nations (2012), 783 million people, or 11% of the global population, remain without access to safe drinking water and almost 2.5 billion do not have access to adequate sanitation. The World Water Council estimates that the planet will be around 17% short of the fresh water supply needed to sustain the world population by 2020 (Charcosset, 2009). The world's oceans contain around 97% of all water on earth and many view this reservoir as an unlimited water resource that should be exploited for future needs. Unfortunately, as most people are aware, seawater is contaminated with high levels of salt (~35,000 ppm) and other biological, physical and chemical agents and refining is necessary to form a clean water product that is safe for drinking. Based on the technology employed, desalination plants are usually characterised into two main types; thermal processes (including multi-stage flash (MSF), multi-effect distillation (MED) and vapour compression distillation (VC) and membrane filtration processes (reverse osmosis (RO), nanofiltration (NF), forward osmosis (FO), electro-dialaysis (ED)), although there are other technologies such as ion exchange and hybrid processes may also be used. Details and reviews of these technologies and methods are available elsewhere (Clayton, 2011; Khawaji et al., 2008; Miller, 2003; Greenlee et al., 2009; Ghaffour et al., 2015; Ng et al, 2015). Currently, the annual worldwide contracted capacity of RO is 37 million m^3 per day which represents ~74% of the global total installed desalination capacity (Oatley-Radcliffe, 2017).

Although desalination technology has progressed rapidly, the technology itself is still imperfect. Despite best efforts desalination is costly and largely inefficient. The desalination process requires a huge amount of pressure or heat to separate the water from the salt and other impurities, which in turn requires energy and therefore money. Large quantities of concentrated brine are produced and need to be disposed of (Ahmad and Williams, 2011) and the desalination process is also considered to be detrimental in terms of environmental impact and cost (Karagiannis & Soldatos, 2008; Ghaffour et al., 2013). Therefore, selection of the most appropriate process to be used and optimisation is vital for successful desalination operations.

In this paper a hybrid RO-Freeze technology process will be considered for the purposes of increased sustainability desalination. The fact that Freeze technology can purify and concentrate liquids has been known for many years. The simplest natural example is that sea-ice has a much lower salt content than seawater, a phenomenon used by the inhabitants of the Polar Regions as a source of drinking water. The basic Freeze process involves three simple stages;

1. The water sample (seawater) is partially frozen.
2. The ice crystals generated are separated from the remaining liquids.
3. The ice crystals are melted to yield clean water and the residue liquid (brine) is disposed.

The Freeze process and Freeze technologies have been extensively reviewed in two recent papers (Williams et al., 2015; Randall and Nathoo, 2015) and from an industrial separations viewpoint, Freeze technology has a number of important advantages:

- A very high separation factor,
- High energy efficiency since the latent heat of freezing is low compared to the latent heat of evaporation (333.5 kJ/kg and 2256.7 kJ/kg, respectively (Rogers and Mayhew, 1994)), which leads to a lower energy requirement in comparison to other processes,
- Insensitive to biological fouling, scaling and corrosion problems because of the low operating temperature, which means less use of chemicals and thus lower operating costs.

- Absence of chemical pre-treatment means no discharge of toxic chemicals to the environment.
- Inexpensive materials of construction can be utilized at low temperature, which results in lower capital cost.

Despite these important advantages for Freeze technology this technique has only been used to a very limited extent industrially for desalination and one of the major prohibiting factors is a lack of appropriate test data. In previous work we demonstrated the Freeze process for the recovery of clean water from synthetic saline solutions and Arabian Gulf seawater using a commercially available ice-maker at bench scale (Williams et al., 2013). The work progressed to demonstrate a hybrid RO-Freeze process to improve desalination water recovery using a commercially available falling film unit at pilot scale (Williams et al, 2015). In this work, we further demonstrate the capability of Freeze technology for desalination by considering the use of a suspension crystalliser in a hybrid RO-Freeze process. In contrast to the falling film unit used previously which generated both a low concentration and super concentrated brine, the suspension crystalliser has the capability to generate clean water from the discarded RO brine. Thus, potentially increasing the clean water recovery from the process.

2.0 Description of suspension crystallisation and basic operation

Suspension-based melt crystallisation is a highly selective, low energy consuming and solvent-free separation process used for the purification of organic chemicals (Ulrich and Glade, 2003; Tähti, 2004). This technology is capable of achieving high purity products and ecological production methods (Ulrich and Glade, 2003). Suspension-based melt crystallisation has previously been used for purifying various chemicals and concentrating waste water. Some typical applications are; acetic acid, acetonitrile, adipic acid, benzene, caprolactam, durene, ethyl lactate, hexamethylendiamine (HMD), ionic liquids, lactic acid, methylene diphenyl isocyanate (MDI), methacrylic acid, o-phenylphenol, p-Diisopropylbenzene, p-Dichlorobenzene, p-Chlorotoluene, p-Nitrochlorobenzene, p-Xylene, phenol, trioxane, and waste water [Ulrich and Glade, 2003; Sulzer, n.d.]. According to Ulrich and Glade (2003), the important advantages of the suspension-based melt crystallisation approach are:

- superior purification can be produced from a single crystallisation stage,

- higher crystal production rate per unit volume of equipment,
- suspension crystallisation process uses less energy to attain the same separation as solid layer crystallisation,
- the suspension crystallisation process is often carried out in a continuous mode, whereas solid layer crystallisation is usually carried out in batch mode.

According to Sulzer (n.d.), suspension crystallisation plants consist of two loops, i.e. crystallisation and separation loops, as shown in Figure 1. The main equipment in the crystallisation loop included a crystalliser, a stirred growth vessel, and a circulation pump. The separation loop contains a piston type wash column, circulation pump, scraper, tube heat exchanger, and product and residue valves. The crystalliser is classified as a tubular and scraped surface heat exchanger. This means that the crystalliser includes scraper blades, which are installed on a rotating shaft driven by a motor. This crystalliser is a single insulated thermostated double wall reaction vessel, typically made of stainless steel, which consists of a vertical tube in which the crystal layer grows as a cylindrical shell (during the crystallisation operation). However, these crystals are removed from the crystalliser's surface to the bulk liquid by means of the scraper blades. By continuously circulating the cooling medium around the crystalliser, the ice crystals are nucleated and progressively crystallised on the refrigerated surfaces of the crystalliser. These ice crystals are then rapidly removed from the crystalliser's surface by the scraper and then transported to the stirred growth vessel as a suspension via the circulation pump. The crystals gradually grow in size within the bulk liquid inside the stirred growth vessel. The stirred growth vessel is made of an insulated vessel and an overhead stirrer assembly. When the crystals reach the desired size and mass ratio (between the crystals and the bulk) in the crystallisation loop, the crystal slurry then proceeds to the separation loop, where the crystals are washed in a wash column and then recovered as product via a heater. The wash column is classified as a piston type wash column. The wash column consists of a piston, cylinder, and scraper. The piston head is covered with a filter screen, which is attached to the reciprocating shaft driven via a pneumatic piston drive or a hydraulic cylinder to achieve a reciprocating motion. The scraper is attached to a rotating shaft driven by a motor. For food applications, the wash column is typically made of glass, because the washing process is controlled with light cells (Ulrich and Glade, 2003). According to Sulzer (n.d.), the piston type wash column works on a four-stroke cycle; (i) filling, (ii) compressing, (iii) washing, and (iv) scraping.

3.0 Materials and Methods

3.1 Preparation of feed water samples

Two different sources of saline water have been used and tested individually as feed-samples in this experimental study. The feed materials are aqueous solutions of sodium chloride and process brines. Barduhn (1965) stated that prepared synthetic water using aqueous solutions of sodium chloride (NaCl) gives results very similar to process brines. Therefore, synthetic water using aqueous solutions of NaCl were prepared, used, and examined as feed, in order to validate the potential capability of the suspension crystallisation process. As far as the water sample preparation is concerned, the feed samples using synthetic water were prepared by dissolving a predetermined mass of NaCl salt (Sodium Chloride PA-ACS-ISO, Order Number: 131659.1211, Panreac, Spain) into a known mass of deionised water (produced by UV water purification system, Direct-Q3, Trade Name: Direct-Q). In addition to this, two different sources of process brine were used and tested individually as feed-samples. The examined process brines were: Arabian Gulf (AG) seawater (4.9 wt% by weight of dissolved salt) and reject brine (6 wt% by weight of dissolved salt), produced from a Reverse Osmosis (RO) membrane desalination plant. The AG seawater and RO Brine were collected from the feed stream and reject brine discharge of the Kadhmah Bottled Water (KBW) plant. The KBW plant represents one of the main research projects of the Water Technologies Department's of the Water Resources Division at the Kuwait Institute for Scientific Research (KISR), located at the Doha Research Plant in Kuwait. The KBW plant consists of two series of RO membrane units (using the series product staging method), as well as a blow moulding machine, a rinsing-filling-capacity machine, a labelling machine, and packaging units including their auxiliary equipment. The feed of the first stage of the RO membrane units is AG seawater. Although the salinity of the reject brine of first stage of RO membrane units are much more than that of the reject brine of the second stage, both reject brines of the two stages are dumped into open AG seawater. The feed samples from the feed stream of the first stage of RO membrane units (i.e. AG seawater) and the dumped RO brines (i.e. the mixed RO brines of two stages) were collected individually from the KBW plant, and tested as feeds in this investigation.

3.2 Analytical methods

Following preparation of feed samples and after completion of the experiments, physiochemical analysis was carried out as appropriate for the given material. The physiochemical analysis of water samples included measurements of the temperature, Total Dissolved Solids (TDS), electrical conductivity, pH, volume, and weight. The salt concentration of the feed sample was measured via a conductivity meter (Cond 3110, Tetra Cond 325) and a conductivity probe (Conductivity probe model: TetraCon 325, Wissenschaftlich-Technische Werkstätten GmbH) to ensure that the correct concentration was achieved. The measurements of TDS were experimentally determined, using the gravimetric method, by evaporating a known weight of the water sample to dryness, and weighing the solid residue. The equipment involved in obtaining gravimetric measurements are an oven (Heraeus instruments, Type: UT 12P, D-63450 Hanau, Kendro Laboratory Products), Petri dish, and laboratory balance (Mettler PM 460, Delta Range, CH-8606). The accuracy of salt concentration results was assured through a simple mass balance. The volume and weight measurements were determined by a laboratory beaker, scaled borosilicate glass cylinder (Barcode: CYL-350-020J, Fisher Scientific) and a laboratory balance (Mettler Toledo, Model: PM30-K). The running time for the crystallization process was measured by a stopwatch timer (HS-10W Stopwatch, Casio). In addition, when the source of the feed sample was either AG seawater or RO brine, full water chemistry analysis was conducted for all samples of each experiment. The purpose of conducting full water chemistry analysis is to detect the major components of ionic composition found in the feed, product, and residue. A DR 5000 Spectrophotometer (Hach, DR 5000) along with powders pillows reagent were used to detect the major components (ionic composition) of the feed water sample using seawater and RO retentate. The reliability of full chemical analysis of the water samples was ensured by using a charge balance error. When appropriate, the physicochemical analysis of water samples was performed using the measuring instruments, auxiliary equipment, tools, and laboratory facilities of Sulzer Chemtech Ltd (Buchs SG, Switzerland). For further investigation, several sets of water samples were sent to both the Doha Desalination Research Plant laboratory and the Central Analytical Laboratories (CAL) at KISR (Kuwait) for performing similar chemical analysis and also carrying out full chemical analyses on water samples to detect the major ionic composition. In addition, the measurements of running time, residue temperature, HTM operating temperature (i.e. crystallisation temperature), washing temperature, stroke, time adjustment, running time of cycle action and adjustment, etc., were monitored and recorded. At the same time, some of these variables were controlled through a software package installed in a computer-controlled system connected to the pilot equipment.

3.3 Experimental set-up

Figure 2 shows the skid mounted pilot plant unit (Freeze Tec, Rig No.: 0-02-503-000, Type: FTC-1C Chem., Sulzer Chemtech Ltd.) used by Sulzer for investigating new fields of application. This pilot plant was used in this experimental study. The description of the pilot plant and basic operation is available in Section 6.2. This pilot plant, however, does not include a stirred growth vessel, so during the crystallisation operation, the crystals gradually grow and mature in size within the bulk liquid inside the crystalliser, instead of using a stirred growth vessel. Although Sulzer Chemtech Ltd commercialises complete test units that are equipped with all necessary equipment, including the cooling compressors, this skid mounted pilot plant does not include a complete built-in refrigeration unit. The pilot plant is provided with a heat exchanger, which is connected to a heat transfer unit (LAUDA, ITH Series, Type: W350/25kW) via tubes for low temperature applications, as shown in Figure 2b (note only the tubes are showing, the LAUDA unit is off camera). The heat transfer unit utilises synthetic thermal oil as the HTM. Apart from the heat transfer unit, the skid is completely equipped with all necessary measuring instruments and control devices. The crystalliser and feed tank are made of stainless steel, which is thermodynamically insulated from the surrounding environment. The pilot plant also contains a wash column with a visible wash front.

Figure 3 shows the crystallisation loop (1), and the separation loop (2) within the pilot plant. The crystallisation loop consists of a feed tank (3), a melt circulation pump (M-102) (4), a crystalliser (5), an ice scraper motor (M-101) (6), a refrigerant circulation pump (M-104) (7), a refrigerant heat exchanger (8), a heat transfer unit (9), and a slurry supply valve (YV-201) (10). The separation loop consists of a piston type wash column (11), an ice scraper motor (M-200) (12), a melting loop (13), a filtrate discharge valve (YV-202) (14), a filtrate recycle valve (YSV-205) (15), a filtrate reject valve (YV-203) (16), and a product output valve (YV-204) (17). The melting loop (13) contains a product circulation pump (known by Sulzer as a Meltloop pump)

(M-220) (18), meltloop pressure valve (YSV-206) (19), product heat exchanger (20), and heating system (known by Sulzer as a Melter) (H-200) (21). The pilot plant is also provided with a cabinet light (22), and air heating system (23). The purpose of the cabinet light is simply for illumination purposes. The air heating system comprises of a heater and fan. The air heating system is used to avoid ice encasing the main components and pipelines of the unit. The pilot plant also contains other equipment, such as a feed pump (YSV-400) and an external wash pressure system (P-225); however, these items of equipment were not used in these experiments. For identification purposes, tag numbers were given to equipment items in order to identify them in the software package (e.g. the tag number for the melt circulation pump is M-102). All valves involved in the pilot plant are classified as automatic valves, except for the five valves that deal with the melt, which are hand-operated ball valves. The feed valve is used to allow feed fluid to flow from the feed tank to the crystallisation loop by gravity. The product and residue valves are used for the purpose of sampling. Two bleed valves have been used to remove air pockets present in the crystallisation and separation loops, before starting the experiment. The pilot plant is connected to a laptop computer, which runs a software package specifically designed for controlling the operation of the pilot plant. Moreover, the software package records and compiles the operational data of the pilot plant. The installed software package is known as "Plot-Chem", and was published by Sulzer Chemtech Company. The pilot plant has an insulated feed tank installed above the crystalliser. The feed tank is covered with a lid, which is used to avoid thermal energy losses and protect the feedstock from the entry of dust, suspended matter, particles, etc. The feed tank has a resistance wire (i.e. heating element), wrapped around it, to control the feed temperature inside the tank during pilot plant operation. The resistance wire heating is controlled and driven by the laptop. Unfortunately, the separation process of the wash column was controlled by means of light cells (this is due to the pilot plant being designed for food applications), and since the RO retentate is colourless, a colour indicator (Chromotrope FB, MKBD4111, Sigma-Aldrich) was added before the experiments were carried out. Before starting the experiment, the final feed sample was made by dissolving a weighed amount of a colour indicator in a known weight of RO retentate. Upon completion of the feed preparation, the feed tank was then covered with the lid.

4.0 Results and discussion

4.1 Suspension crystallisation trials

The trial tests were limited in run number and time to only achieve the objectives of this study and a summary of the experimental data is presented in Table 1. For clarification, performance optimisation of the pilot plant was performed based on the maximum salt rejection ratio. Sub-runs a-1, b-1, and c-1 represent tests to optimise performance of the pilot plant, determining the operating limits and the optimal process conditions. These tests correspond to multistage operation, in order to increase the water recovery ratio of the suspension pilot plant whilst carrying out the experiments. For industrial applications, the operation of the suspension crystallisation process would be carried out in a single-stage process. For the optimised pilot plant operation, sub-runs a-2 and b-2 examined the performance of pilot plant to minimise the RO retentate as much as possible and to determine the quality of the product water. T1 represents the overall experimental data of sub-runs a-1, b-1, and c-1, whereas T2 represents the overall experimental data of sub-runs a-2 and b-2. This means that T1 and T2 represent a continuous operation test corresponding to single-stage operation.

Table 1 shows that the pilot plant was tested with different salt concentrations of RO retentate ranging from 61,139 up to 99,886 ppm. Table 1 also shows that sub-runs a-1 and a-2 utilised the RO retentate from the Kadhmah (RO) desalination plant as a feed material. The TDS value of the feed water used in sub-run a-1 is slightly higher than that in sub-run a-2. This is due to the different mass ratio between the colour indicator and RO retentate for sub-run a-1 compared to sub-run a-2. Before starting sub-run a-1, the feed sample was prepared by dissolving a mass of 10.53 g of colour indicator into a mass of 116.959 kg of RO retentate. For sub-run a-2, the feed sample was prepared by dissolving a mass of 3.09 g of colour indicator into a mass of 86.829 kg of RO retentate. The concentration of colour indicator was slightly reduced in order to reduce the colour concentration of feed, and improve the separation performance and wash column performance, which is controlled in the crystallisation unit by light cells activated by the colour of the ice crystal bed. Upon completion of each sub-run, the final product was collected for chemical analysis, whereas the final residual liquids were collected at the end of the final sub-runs c-1 and b-2 for chemical analysis. Figure 4 shows the water samples of product and residue collected and taken for physiochemical analysis.

Although sub-runs a-1, b-1, and c-1 represent initial trial parametric testing used for optimising the key operating parameters, nevertheless, preliminary experimental results show that the suspension pilot plant was potentially capable of recovering high purity product from RO retentate (waste brine). The overall salt rejection and permeate water recovery ratios were 96.67% and 42.99% respectively for run 1. The suspension crystallisation pilot plant was able to reduce the TDS value from 61,200 ppm down to 2,036 ppm overall, whereas the individual run TDS was 61,200 and 1,200 ppm for feed and product respectively for sub-run a-1 and were 76,292 and 200 ppm respectively for sub-run b-1. In sub-run a-1, the pilot plant produced a final product at a TDS value of 1,200 ppm, which is slightly higher than that in drinking water standards (1,000 and 500 ppm according to the World Health Organization (WHO) and Food and Drug Administration (FDA) respectively (Semerjian, 2011)). When the process key-parameters of the pilot plant were set to within the optimum operating limits in the subsequent freezing stage (i.e. sub-run b-1), the pilot plant was potentially capable of producing a final product water ready for immediate use from a feed salt concentration of 76,292 ppm. This was mainly due to the optimum operating conditions not being known and applied in the initial test i.e. run a-1. When the operating temperature of the residual liquid reached -8.2°C, deterioration in the separation performance of the pilot plant was observed and resulted in the product water quality becoming relatively poor, sub-run c-1. However, even with the highest feed concentration (99,886 ppm) and negative operating conditions, the pilot produced saline water of near brackish water quality. With regard to deterioration in the performance of the pilot plant, further investigation was carried out in sub-run b-2 to check whether this limitation occurred due to process optimisation or other factors, this will be discussed later. In general, the total results from run 1 for the final product were very encouraging with the pilot plant producing a final product of near drinking water standards in terms of TDS.

For run 2, the performance of the pilot plant was monitored over an operational period of 4.77 hours. Figure 5 shows the operating and performance parameters for the pilot plant versus running time. Figure 5a shows that the start-point temperature of the crystallisation and operating residue were -11.3°C and -4.6°C, respectively; while the end-point temperature of the crystallisation and operating residue were -19.4°C and -8.2°C respectively. Figure 5b shows that the cycle time adjustment was kept at a constant value (35 seconds) during the course of operation, while the cycle time action averaged about 29 seconds up to 2.77 hours of operation and then rapidly declined to an average time of 21.5 seconds. The reduction in the cycle time action indicated a decrease in the operational period of the separation process, leading to an increase in the total product water output. The maximum and minimum values of cycle time action were 31 and 20 seconds respectively. Figure 5c shows that the bed-height averaged at about 31 mm and the bed-height was maintained at constant value of 30 mm from 2.93 hours until the end of the test. The average product flowrate was 7.41 kg/h as shown in Table 1. During the course of operation, final product water samples were periodically collected from the melting loop for chemical analysis. Figure 5d shows that the maximum and minimum TDS value of product water sample was 174 and 77 ppm respectively. This means that the TDS values of product water measured below 175 ppm at all times, which gives a clear indication that the pilot plant was capable of producing a final product of drinking water quality. During the collection of product water samples, the pilot plant was able to produce product water of near transparent solution, similar to drinking water as shown in Figure 4 (bottom). Visual observations confirmed the physiochemical analyses. The colour indicator in these product water samples disappeared entirely in comparison with feed sample, which indicates that the dissolved salts have been significantly reduced. However, a slight amount of colour indicator was visually observed in the other samples of product water and therefore the final product water samples from runs a-2 and b-2 contain a slight amount of colour indicator as shown in Figure 4 (middle). Nonetheless, although slightly coloured, these samples were far less coloured than the feed sample. For industrial applications, the appearance of colour indicator, i.e. indicating some residual salts remain in the product water, this will not represent an obstacle that could negatively affect the commercial application as the levels observed in Table 1 meet drinking water specification. In addition, the separation process in the wash-column could easily be controlled via electrical conductivity instead of light cells and thus removing the need to use colouration in the first place. In general, the overall results from run 2 proved that suspension crystallisation was able to lower the salt concentration from 61,139 ppm to 111 ppm at considerable yield. Table 1 shows that the overall product water recovery and salt rejection ratios were 40.67 and 99.82% respectively. The concentration of RO retentate achieved was over 59% at a TDS value of 103,004 ppm.

4.2 Comparison to RO desalination and other produced waters

A summary of physiochemical analysis of the water samples for run 2 in comparison with the Kadhmah (RO) desalination plant is presented in Table 2. The Kadhmah (RO) desalination plant consists of two RO units using a series product staging method. Table 2 shows that the average TDS value of seawater fed to the RO plant is 49,074 ppm, while the TDS value of RO retentate fed to the suspension pilot plant was 61,130 ppm. Nevertheless, the TDS value of product water from the suspension plant is slightly lower than that obtained by the RO plant, at 111 ppm and 160 ppm respectively. As expected, the salt rejection ratio of the suspension pilot plant is slightly higher than that of the RO plant at 99.82% and 99.67% respectively. The water recovery ratio of the suspension pilot plant is much higher than that of the RO plant at 40.67% and 14.28% respectively. Note that the RO plant operates at a relatively low recovery rate when compared to other commercial plants, most RO plants operate at around a 40-50% recovery ratio. However, the freeze process will easily cope with the change in brine concentration expected at higher recovery ratios. Table 2 also shows that the average ion concentration values of Ca^{2+}, Mg^{2+}, and $(SO4)^{2-}$ for RO retentate fed to the suspension crystallisation pilot plant were 1,476 ppm, 1,463 ppm, and 4,800 ppm respectively. The suspension crystallisation pilot plant was capable of reducing the hardness ions down to 5.7 ppm, 10.24 ppm and 3.69 ppm respectively. In addition, there was also a level of enrichment of Na^+ and Cl^- ions at concentrations of 20,881 ppm and 32,200 ppm respectively and the suspension pilot plant was able to reduce these concentrations down to 141ppm and 100.27 ppm respectively.

Table 3 compares the major ionic composition of product water from the suspension crystallisation pilot plant to market leading bottled water brands that are commercially available in many countries around the world. This gives a clear indication that the product water of the suspension pilot plant emulates international and regional premier bottled waters. Although the product water from the suspension pilot plant was enriched with Na^+ and Cl^- ions in comparison with RO product (see Table 2), these concentrations are still within accepted limits and are comparable with international standards for drinking and bottled waters as shown in Table 4. Table 4 provides clear evidence that the product water from the suspension crystallisation pilot plant was produced within the allowable international limits; in terms of TDS, pH, and major ionic composition and that the product water from the suspension crystallisation pilot plant contains acceptable levels of Na^+ and Cl^- ions, while maintaining low concentrations in hardness ions (such as; Ca^{2+}, Mg^{2+}, and $(SO_4)^{2-}$ and NO_3^-).

In general, experimental data has proven that the suspension crystallisation pilot plant is capable of providing a product ready for immediate use, i.e. for human consumption, and simultaneously able to concentrate the waste stream from a RO plant to a considerable degree. The performance results obtained provided clear evidence that the proposed treatment system would be ideal for concentrating the RO retentate and, more specifically, the RO retentate with a TDS value of less than 80,000 ppm.

4.3 Further discussion

In order to increase the permeate water recovery ratio, the operating temperature of the residue can be reduced down to -15°C, so long as the eutectic point is still far away from the residue temperature. When the operating residue temperature was reduced below -8.2°C in sub-run b-2, a gradual deterioration in the separation performance of the pilot plant was observed. The TDS value of the product water increased rapidly up to 1,500 ppm and then fluctuations occurred with a gradual increase until this value reached 15,000 ppm. At this stage, a number of attempts were made to improve the quality of product water by optimising the key operating parameters. Unfortunately, all these attempts failed to improve the product quality. In fact, this phenomenon was previously detected in run 1, more specifically in sub-run c-1, and similar behaviour was also noticed during operation at an operating residue temperature of -8.2°C. This confirms that the allowable operating temperature of the residue should not be less than -8.2°C. The reason for this performance deterioration may be explained by one of the following; (i) the optimal operating conditions were not reached at low operating temperature, and this was due to the experimental investigation being carried out over a limited number of tests and key operating parameters; (ii) the solubility limit of one or some salts in the residual liquid may have been reached, although precipitation of solid salts was not visually observed from the experiments. Table 2 indicates signs that precipitation of salts may have occurred as there are losses in concentration, specifically Ca^{2+}, Mg^{2+}, and Na^+. (iii) precipitation of solid colour indicator. However, based on visual observation, there was no clear sign of salts precipitation composed of colour indicator in the remaining residual liquid or in the pilot plant equipment throughout the experiments; and (iv) precipitation of solid sodium sulphate (i.e. $Na_2SO_4.10H_2O$) in the residual liquid. According to Pounder (1965), when the temperature of seawater is reduced below -8.2°C, some of the sodium sulphate might be precipitated. This means that the seawater will exist in three different phases; solid H_2O, solid sodium sulphate ($Na_2SO_4.10H_2O$), and remaining residual liquid. This critical temperature (i.e. -8.2°C) does not represent the eutectic temperature for seawater. However, at this temperature, some solid salt phase begins to appear in the remaining residual liquid. Precipitation of solid salts was not visually observed from the experiments. However, there is a sign of precipitation of salts composed of $(SO4)^{2-}$ and Na^+ since the percentage losses in their ionic concentration are 9.09 and 33.11% respectively (see Table 2).

Table 1, specifically sub-run c-1, shows that the pilot plant was able to reduce the TDS value of the feed from 99,886 ppm down to 15,000 ppm. The experimental results were not encouraging when the operating temperature of the residue reached -8.2°C or less, where the pilot plant did not produce a final product of near potable quality. However, the pilot plant produced final water of near brackish water quality that can be easily treated by brackish water RO membranes to reach a final product water of drinking water quality. This gives a clear indication that the suspension crystallisation system can be used for desalting RO retentate in a rectification stage and also for concentrating the residue from a previous stage, i.e. a stripping stage.

This experimental investigation deals with two different types of residual liquids, namely a final residue and the operating residue. The water sample of the operating residue was not considered for chemical analysis in this study, as the remaining ice crystals could not be separated from the operating residue. However, the operating residue will be considered as the final residue for industrial applications, because commercial plant will be equipped with a filtration system which enables recovery of the remaining ice crystals from the crystalliser. According to Sulzer, two possible scenarios for recovering the remaining ice crystals from the crystalliser can be applied. The first scenario is to remove the ice crystals and operating residue from the crystalliser and separate by filtration. The ice crystals can then be purified and recovered via a wash column unit. The second scenario is to remove the operating residue from the crystalliser leaving the ice crystals behind using a filtration system. This means that the remaining ice crystals will be mixed with the incoming feed water in the crystalliser before starting the subsequent freezing stage. This step helps by either diluting the incoming feed water and simultaneously decreasing the incoming feed temperature (when the incoming feed temperature is higher than its freezing point), which will result in an increase in the permeate water recovery ratio compared to the previous stage as the feed concentration will be reduced, or will help maintain the crystal slurry ratio at 30 wt% inside the crystallization loop when the temperature of the incoming feed reaches its freezing point. The first scenario might be more feasible for desalting and concentrating RO retentate (brines) due to the increased total permeate water recovery ratio and simultaneous reduction in the waste stream (as far as possible). However, the economic aspects (i.e. capital cost and energy consumption), process simplicity and reliability must be taken into consideration to determine which scenario is the most suitable for desalination applications.

The product water recovery ratios for the pilot plant were theoretically computed at different temperatures of operating residue. These analytical calculations were performed on different saline waters, such as ocean seawater, Arabian Gulf seawater, and Reverse Osmosis (RO) brine. For simplification, several assumptions were considered in these analytical calculations, namely; (i) the TDS value of product water was assumed to be 111 ppm as found in the experimental run 2 (see Table 1); (ii) The mass of feed was assumed to be 86.832 kg, i.e. the same as that in experimental run 2 (see Table 1); (iii) The TDS value of feed is constant, which is dependent on the type of saline water being treated (i.e. ocean seawater (35,000 ppm), Arabian Gulf seawater (49,000 ppm), and RO brine (61,000 ppm)); (iv) The TDS values of the residue were obtained from a phase diagram. By rearranging salt and mass balance formulas, the remaining unknowns, which are the masses of product and residue, can be theoretically obtained, leading to the permeate water recovery ratio being determined; (v) The pilot plant can produce a final product water of near drinking water standards until the residue temperature reached -15°C; (vi) The operating residue was assumed to be the final residue (i.e. all ice crystals in the pilot plant were recovered and purified).

Figure 6 shows the influence of feed concentration and residue temperature upon the water recovery ratio. As expected, the water recovery ratio is not proportional to both the feed salt concentration and the residue temperature. The lower residue temperature leads to an increase in the water recovery ratio. This is in agreement with the experimental data obtained from the suspension crystallisation pilot plant. Moreover, the lower initial salt concentration of feed leads to an increase in the water recovery ratio. When the pilot plant is individually fed with ocean seawater, Arabian Gulf seawater and RO brine, then at residue temperature of -6°C, the estimated permeate water recovery ratios are 66.09%, 52.48%, and 40.72%, respectively. When the residue temperature is further decreased to -8.2°C, then the permeate water recovery ratios will be increased to 72.83%, 61.92%, and 52.49%, for the same saline waters respectively. By reducing the residue temperature down to -15°C, then the estimated permeate water recovery ratio reached 82.28%, 75.17%, and 69.02% respectively.

4.4 Scale-up of the hybrid RO-Freeze process

The scale-up for this application is very straightforward because commercial plant can be established simply by adding the appropriate number of crystallisation and separation loops to achieve the required capacity. This experimental investigation was carried out with the RO retentate of the Kadhmah (RO) desalination plant and this plant is now used as an example test case for scaling-up the suspension crystallisation process. This example will give an estimation of the mass and salt concentration of the three liquid streams for large-scale application using continuous mode. These values are also used for estimating the water treatment costs. The proposed treatment system was scaled-up according to the experimental results obtained from run 2, taking into consideration that the temperature of the operating residue was -8.2°C. Hence, a water recovery ratio of 53% was obtained from the experimental graph as shown in Figure 6. The TDS value of the product water was 111 ppm (i.e. mass percent of 0.01%) according to run 2 results (see Table 1), whereas the volume flow-rate of product was determined by the water recovery ratio. The salt concentration of the residue was determined from the phase diagram corresponding to -8.2°C, whereas the volume flow-rate of the residue was determined by mass balance. A novel treatment option configuration, which represents the hybrid integration of a RO plant and suspension crystallisation plant is shown in Figure 7 and contains the estimated values for feed salt concentration, product water, and residue (concentrated brine) are 6.1%, 0.01%, and 13% by weight of salt respectively. The estimated volume flowrates are 21.9 $m^3 h^{-1}$, 11.61 $m^3 h^{-1}$, and 10.29 $m^3 h^{-1}$ for the same water streams respectively. According to Sulzer, this commercial plant can be established without any limitation; however, such an application might be hindered by the economic aspect, e.g., capital and operational costs. Table 5 shows the estimated annual rates of drinking water production, RO retentate, and residual liquid of Kadhmah RO plant, the suspension crystallisation commercial plant, and a hybrid desalination plant. The estimated annual rate of RO retentate is significantly reduced from 191.84 to 90.14 tonnes year^{-1} by treating the RO retentate through a large-scale application of the suspension crystallisation process. Furthermore, the production of drinking water can be substantially increased from 34.17 to 135.87 tonnes year^{-1} by incorporating the commercial scale suspension crystallisation process into Kadhmah RO plant.

5.0 Conclusions

This study assessed and validated the viability of using a suspension crystallisation process as a water desalination system for producing drinking water. Although the pilot trials were slightly limited in number and the equipment used was not designed for desalination applications, the results were highly encouraging and demonstrated that a substantial amount of potable product water could be produced while simultaneously concentrating the RO brine used as feed water. The analysis results showed that the product water quality was comparable to international drinking and bottled water standards. The experimental results showed that the water recovery was around 41%, while the salt rejection ratio reached over 99%. Therefore, the suspension crystallisation process might be competitive with other desalination processes in RO brine applications. Based on the experimental results, the suspension crystallisation process was scaled-up and combined with a commercial plant using RO membrane technology to hypothesize a hybrid RO-Freeze technology. The production rate for the combined plant was estimated and compared to the RO plant only. These theoretical results demonstrate that the combined plant is able to produce substantially higher quantities of drinking water (~400%, 100/34.17x135.87) than in the basic RO plant. This was achieved by concentrating the waste steam to a considerable extent producing a super brine at ~ 13wt%. While there is a cost to this additional process in terms of capital and energy that must be quantified, the obvious increase in water harvest and reduction in residual brine quantity lead to a very attractive desalination process. If the energy demands are acceptable, then this technology could lead to a more sustainable water future.

Acknowledgement

The authors would like to thank Sulzer Chemtech Ltd for kindly supplying equipment and for their assistance during the pilot trials, particularly S. Dette, M. Stepanski, F. Lippuner and H. Engstler.

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Captions for Figures and Tables

Figure 1: A process flow diagram of a typical suspension crystallisation unit (taken from Sulzer, (n.d.))

Figure 2: Sulzer Chemtech Ltd pilot suspension crystallisation unit used in this study, a) left hand view and b) right hand view.

Figure 3: Process flow diagram for the suspension crystallisation pilot plant used in this study (adapted from a screen shot of the software control system).

Figure 4: Samples collected from the suspension crystallisation pilot trials. Top – samples from run 1, Middle – samples from run 2 and Bottom - product water sample from sub-run (a- 2) in comparison with feed sample.

Figure 5: Suspension crystallisation data from run 2 over the run time. a) operating temperature, b) cycle time, c) bed height and d) product water concentration.

Figure 6: Experimental and predicted water recovery ratio at different residue temperatures for different types of saline water.

Figure 7: Example of the potential industrialised hybrid Reverse Osmosis (RO) – Freeze desalination plant, where; Q is the volume flow-rate (m^3 h^{-1}), and C is the salt concentration, where subscript f = feed water, p = product water, b = brine, and 1 and 2 refer to the stage number.

Table 1: Summary of the performance data for the suspension crystallisation pilot plant used for treating different salt concentrations of RO brine, where; WR is the permeate water recovery ratio, SR is the salt rejection ratio, RT is the running time, and APFR is the average product flow-rate.

* Water recovery ratio is computed as mass ratio of product water to the feed. Water recovery ratio = (product / feed) x 100.

** Salt rejection ratio describes the quantity of salt which is removed by the suspension pilot-plant computed as a percentage. Salt rejection ratio = ((feed concentration - product concentration) / feed concentration) x 100.

Table 2: Comparison of major component analysis and operational parameters between the Kadhmah (RO) desalination plant and the suspension crystallisation trials (run 2).

* the RO stage 1 produces 8 m^3 h^{-1} product water and 6.5 m^3 h^{-1} is sent to the second stage and 1.5 m^3 h^{-1} is sent to drain.

Table 3: Chemical composition of the product water from the suspension crystallisation pilot plant compared bottled water brands.

Table 4: Chemical composition of product water from the suspension pilot plant in comparison with international standards related to the quality of drinking water and bottled water (adapted from Semerjian, 2011), where; WHO: The World Health Organization, IBWA: International Bottled Water Association, CAC: Codex Alimentarius Commission, LIBNOR: Lebanese Standards Institution, FDA: Food and Drug Administration, and EU: European Union standards.

Table 5: Estimation of the annual rates of all water streams for the Kadhmah RO desalination plant, the proposed suspension crystallisation plant, and the combined hybrid plants, where (t/y) represents tonnes per year.

* the RO stage 1 produces 8 m^3 h^{-1} product water and 6.5 m^3 h^{-1} is sent to the second stage and 1.5 m^3 h^{-1} is sent to drain

Figure 1

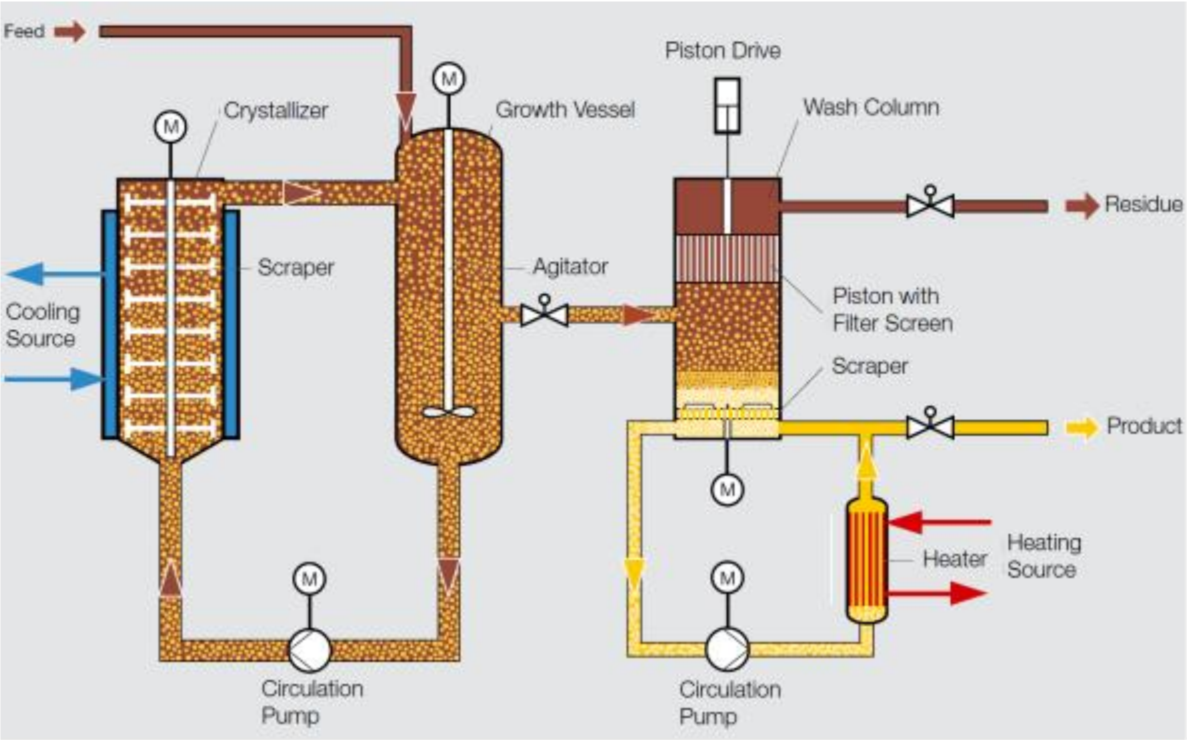

Figure 2

Figure 3

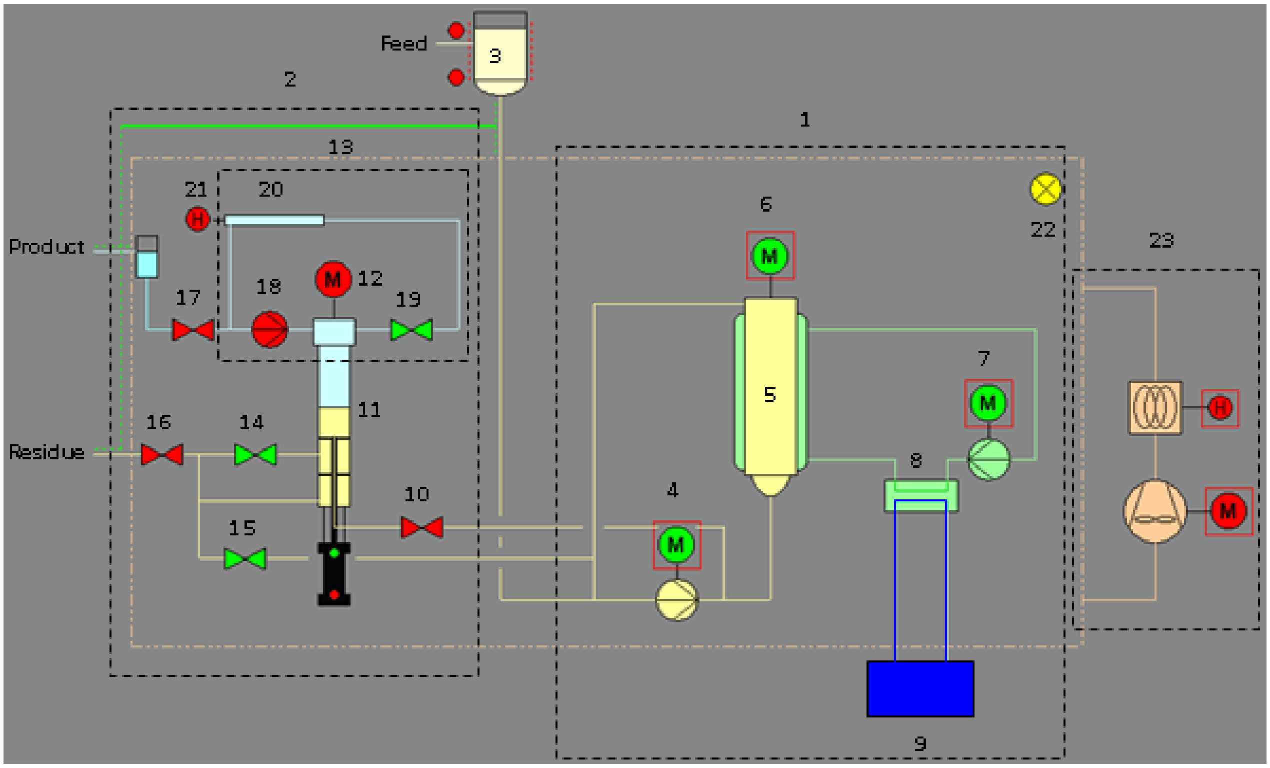
Feed
3
2
1
13
21
H
20
6
M
22
23
Product
18
M
12
19
17
7
M
H
5
11
16
14
Residue
8
M
4
M
10
15
9

Figure 4

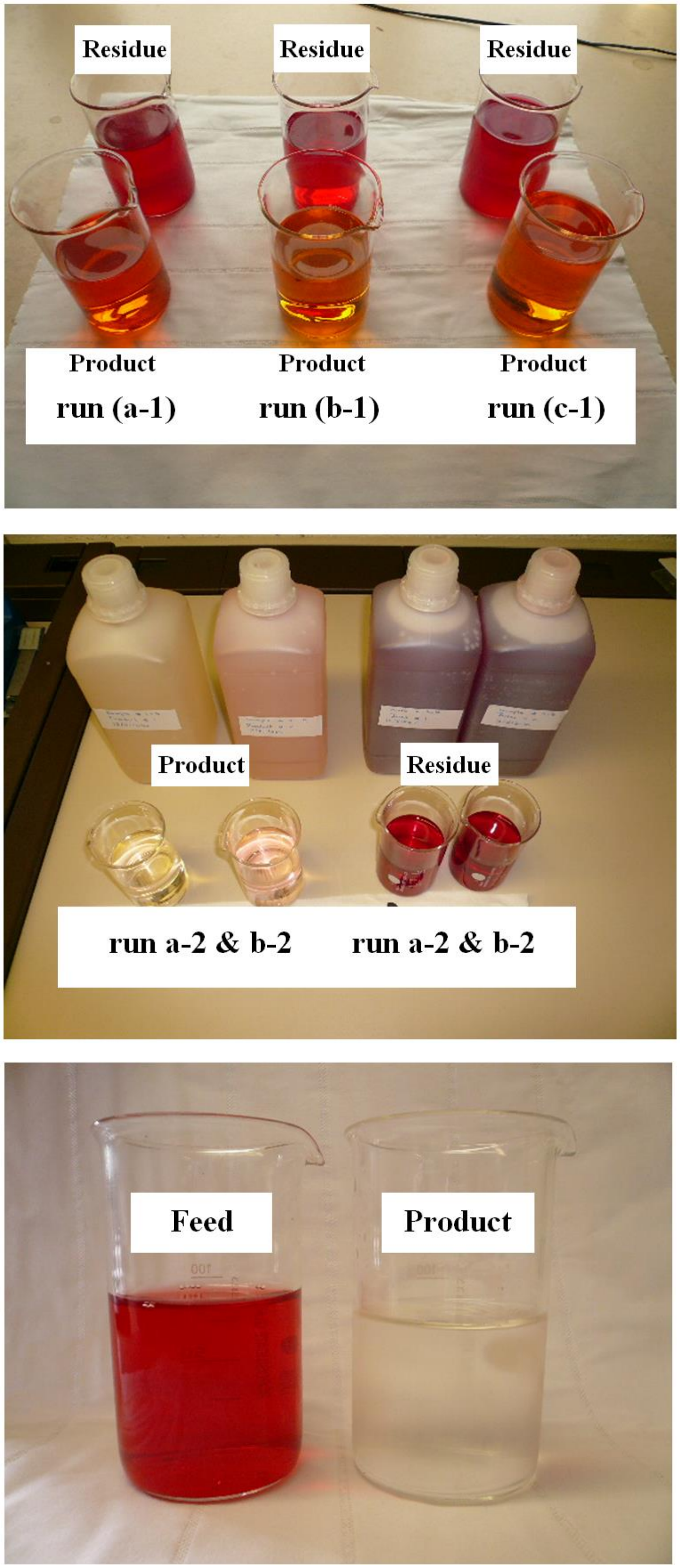
Residue
Residue
Residue
Product
run (a-1)
Product
run (b-1)
Product
run (c-1)
Product
Residue
run a-2 & b-2
run a-2 & b-2
Feed
Product

Figure 6

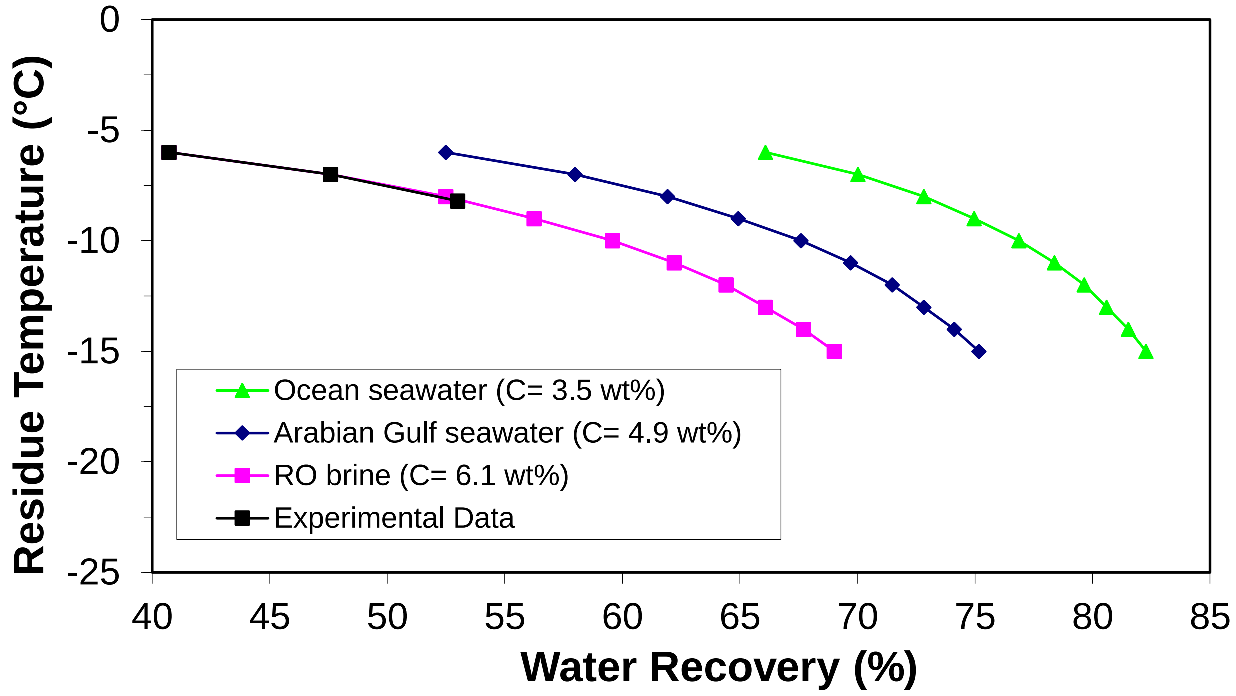
0
-5
-10
-15
-20
-25
Residue Temperature (°C)
40
45
50
55
60
65
70
75
80
85
Water Recovery (%)
Ocean seawater (C= 3.5 wt%)
Arabian Gulf seawater (C= 4.9 wt%)
RO brine (C= 6.1 wt%)
Experimental Data

Figure 7

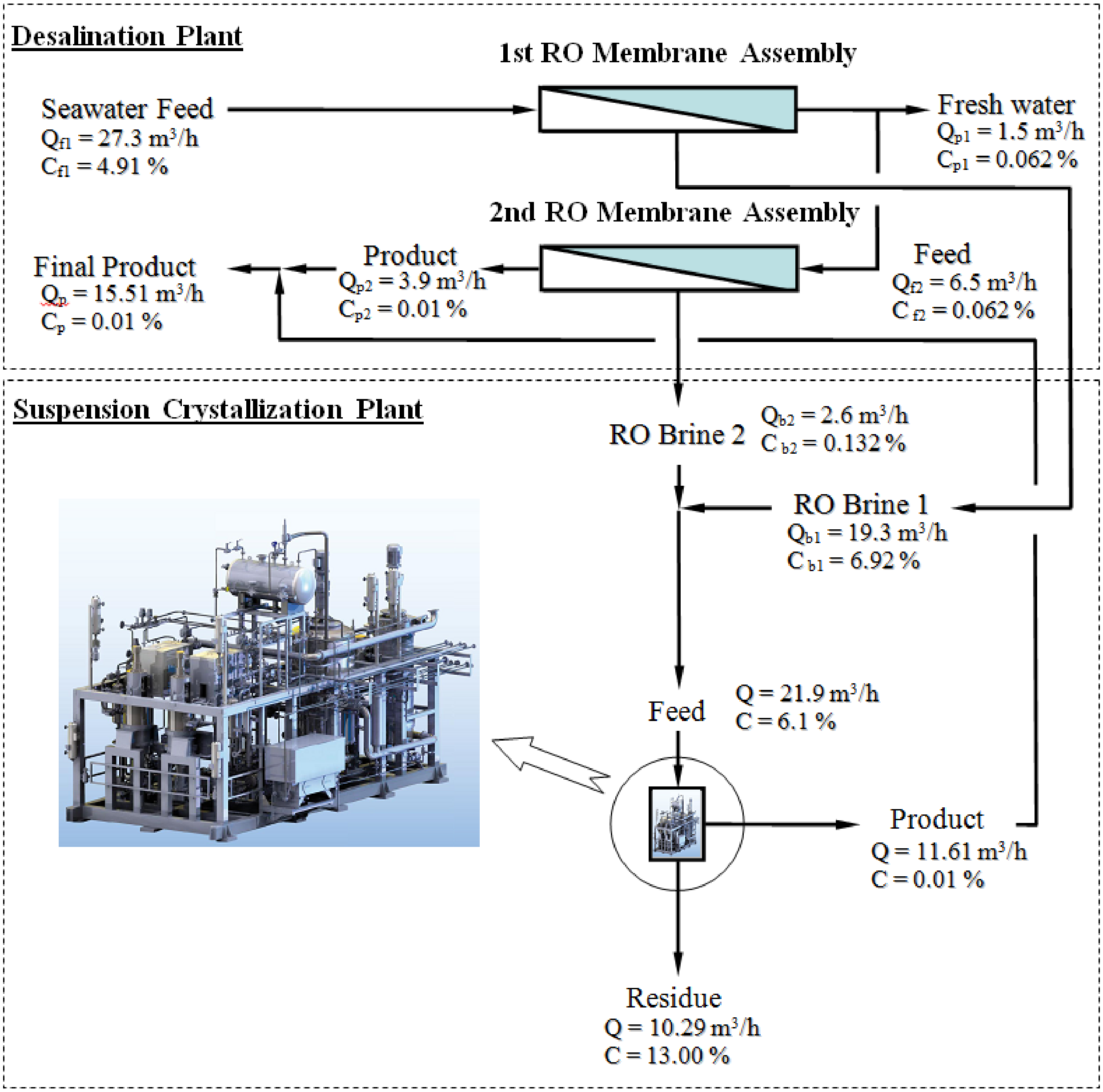
Desalination Plant
1st RO Membrane Assembly
Seawater Feed
$Q_{f1} = 27.3\ m^3/h$
$C_{f1} = 4.91\ \%$
Fresh water
$Q_{p1} = 1.5\ m^3/h$
$C_{p1} = 0.062\ \%$
2nd RO Membrane Assembly
Final Product
$Q_p = 15.51\ m^3/h$
$C_p = 0.01\ \%$
Product
$Q_{p2} = 3.9\ m^3/h$
$C_{p2} = 0.01\ \%$
Feed
$Q_{f2} = 6.5\ m^3/h$
$C_{f2} = 0.062\ \%$
Suspension Crystallization Plant
RO Brine 2
$Q_{b2} = 2.6\ m^3/h$
$C_{b2} = 0.132\ \%$
RO Brine 1
$Q_{b1} = 19.3\ m^3/h$
$C_{b1} = 6.92\ \%$
Feed
$Q = 21.9\ m^3/h$
$C = 6.1\ \%$
Product
$Q = 11.61\ m^3/h$
$C = 0.01\ \%$
Residue
$Q = 10.29\ m^3/h$
$C = 13.00\ \%$

Figure 5

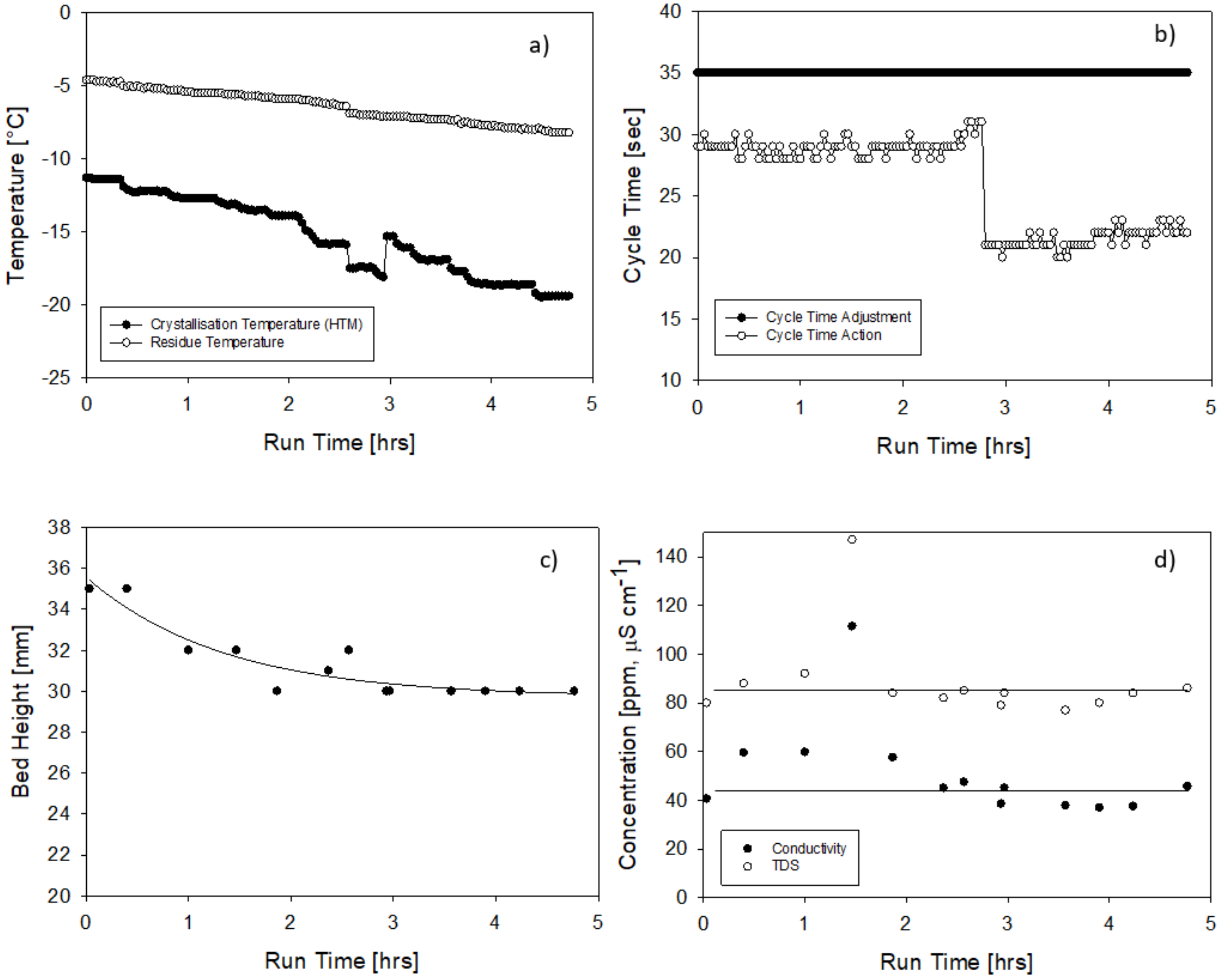
a)
Temperature [°C]
0
-5
-10
-15
-20
-25
Run Time [hrs]
0
1
2
3
4
5
Crystallisation Temperature (HTM)
Residue Temperature
b)
Cycle Time [sec]
40
35
30
25
20
15
10
Run Time [hrs]
0
1
2
3
4
5
Cycle Time Adjustment
Cycle Time Action
c)
Bed Height [mm]
38
36
34
32
30
28
26
24
22
20
Run Time [hrs]
0
1
2
3
4
5
d)
Concentration [ppm, µS cm^{-1}]
140
120
100
80
60
40
20
0
Run Time [hrs]
0
1
2
3
4
5
Conductivity
TDS

Table 1

Run	Sub - Run	Feed			Product			Residue			WR*	SI
		Mass	TDS	Freezing Point	Mass	TDS	Freezing Point	Mass	TDS	Freezing Point		
		(kg)	(ppm)	(°C)	(kg)	(ppm)	(°C)	(kg)	(ppm)	(°C)	(%)	(
1	a-1	116.97	61,200	-3.10	23.51	1,200	-0.07	93.46	76,292	-4.23	20.10	98
	b-1	93.46	76,292	-4.23	22.12	200	-0.03	71.34	99,886	-5.80	23.67	99
	c-1	71.34	99,886	-6.07	4.65	15,000	-0.52	66.69	105,805	-6.20	6.52	84
T1		116.97	61,200	-3.10	50.28	2,036	-0.11	66.69	105,805	-6.20	42.99	96
2	a-2	86.832	61,139	-3.10	21.713	80	-0.03	65.119	81,498	-4.50	25.01	99
	b-2	65.119	81,498	-4.50	13.599	160	-0.04	51.52	103,004	-5.90	20.88	99
T2		86.832	61,139	-3.10	35.312	111	-0.03	51.52	103,004	-5.90	40.67	99

Table 2

Parameter	Unit	Kadhmah (RO) Desalination Plant			Sulzer Suspension Pi	
		Feed	Product	Brine	Feed	Produ
Mass	kg	-	-	-	86.832	35.31
Flow Rate	(m^3/h)	27.30	3.9*	21.9	-	0.007
pH	-	7.13	7.6	7.42	7.42	7.20
TDS	(mg/L)	49,074	160	61,139	61,130	111
Conductivity	(mS/cm)	63.6	0.43	76	76	0.35
Freezing Point	(°C)	-2.76	-0.13	-3.1	-3.1	-0.11
Ca^{2+}	(mg/L)	1,080	50	1,476	1,476	5.7
Mg^{2+}	(mg/L)	1,387	2	1,463	1,463	10.24
Na^+	(mg/L)	16,523	12	20,881	20,881	141
Cl^-	(mg/L)	25,480	18	32,200	32,200	100.2
$(HCO_3)^-$	(mg/L) as $CaCO_3$	175.6	135	241.2	241.2	1.8
$(SO_4)^{2-}$	(mg/L)	3,900	3	4,800	4,800	3.69
NO_3^-	(mg/L)	2.7	1.7	2	2	< 0.0

Table 3

Parameter	Unit	Pilot Plant	Abraj	Al-Rawdatain	Gulfa	Buxton	Highland Sprin
		Under Research	Kuwait	Kuwait	U.A.E.	UK	UK
pH	-	7.2	7.8	7.8	8.1	7.4	7.8
TDS	(mg/L)	111	112	168	120	280	170
Ca^{2+}	(mg/L)	6	25	45	19	55	40.5
Mg^{2+}	(mg/L)	10	5	7	29	19	10.1
Na^{+}	(mg/L)	141	7	9	21	24	5.6
Cl^{-}	(mg/L)	100	55	9	56	37	6.1
$(HCO_3)^{-}$	(mg/L) as $Ca\,CO_3$	1.8	50	155	155	248	150
$(SO_4)^{2-}$	(mg/L)	4	20	22	44	13	5.3
NO_3^{-}	(mg/L)	< 0.05	1	8	5.9	< 0.1	3.1

Table 4

Parameter	Unit	Pilot Plant	WHO	IBWA	CAC	LIBNOR
		Under Research	Drinking Water	Bottled Water	Bottled Water	Bottled Water
pH	-	7.2	6.5-9.5	6.5-8.5	-	6.5-8.5
Conductivity	(µS/cm)	347	-	-	-	-
TDS	(mg/L)	111	1,000	500	-	500
Ca^{2+}	(mg/L)	6	-	-	-	81
Mg^{2+}	(mg/L)	10	-	-	-	50
Na^{+}	(mg/L)	141	-	-	-	150
Cl^{-}	(mg/L)	100	250	250	-	200
$(SO_4)^{2-}$	(mg/L)	4	-	250	-	250
NO_3^{-}	(mg/L)	< 0.05	50	44	50	45

Table 5

Kadhmah RO Plant			**Suspension Plant**			**Hybrid Plant**		
Feed	Product*	Brine	Feed	Product	Residue	Feed	Product*	Residue
(t/y)	**(t/y)**	**(t/y)**	**(t/y)**	**(t/y)**	**(t/y)**	**(t/y)**	**(t/y)**	**(t/y)**
239.15	34.17	191.84	191.84	101.70	90.14	239.15	135.87	90.14