Available online at http://ijcpe.uobaghdad.edu.iq and www.iasj.net
Iraqi Journal of Chemical and Petroleum Engineering
Vol.23 No.1 (March 2022) 15 – 22
EISSN: 2618-0707, PISSN: 1997-4884

Crystallization Process as a Final Part of Zero Liquid Discharge System for Treatment of East Baghdad Oilfield Produced Water

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Abstract

This study investigated the application of the crystallization process for oilfield produced water from the East Baghdad oilfield affiliated to the Midland Oil Company (Iraq). Zero liquid discharge system (ZLD) consists of several parts such as oil skimming, coagulation/flocculation, forward osmosis, and crystallization, the crystallization process is a final part of a zero liquid discharge system. The laboratory-scale simple evaporation system was used to evaluate the performance of the crystallization process. In this work, sodium chloride solution and East Baghdad oilfield produced water were used as a feed solution with a concentration of 177 and 220 g/l. The impact of temperature (70, 80, and 90 °C), mixing speed (300, 400, and 500 rpm), feed concentration (177 and 220 g/l), and time (0.5-9.5 h) on the crystallization performance for oilfield produced water treatment were investigated on evaporation rate and recovery. The recovery increased with increasing temperature and mixing speed while decreasing with an increase in feed concentration. Pure water and salts were recovered from the concentrated produced water, the recovery of pure water at 80 °C, 400 rpm, and 220 g/l feed concentration was 82.22 and 81.35% after 5.5 h for NaCl solution (i.e., simulated oilfield produced water) and oilfield produced water, respectively.

Keywords: Crystallization, evaporation, zero liquid discharge system, Iraqi oilfield produced water.

Received on 03/02/2022, Accepted on 22/02/2022, published on 30/03/2022

https://doi.org/10.31699/IJCPE.2022.1.3

1- Introduction

The public and industrial sectors consume a large amount of freshwater while producing a large amount of wastewater [1]. Various desalination technologies have been developed in recent decades to alleviate water scarcity [2]. The oil industry is a major source of pollutants that pollute the environment, having the ability to affect it at all levels: air, water, soil, and, as a result, all living beings on Earth [3, 4]. Iraq's oilfields are dispersed over vast areas. Water-to-oil ratios in oil fields, particularly in southern and northern Iraq, may reach 20%. In the near future, this ratio may reach the global average [5]. Iraq is one of the world's most important oil-producing countries, with the third largest proven oil reserves and the world's second largest oil exporter [6].

Produced water may contain various compounds including suspended solids [7], volatile organic compounds, dissolved solids [8], heavy metals, chemical additives like coagulants [6], scale inhibitors [9], free and dispersed oil and grease, microorganisms [10]. To produce high-quality treated water, a series of different technologies must be combined [11].

Hence, novel strategies for reusing produced water must be developed to address the problem of water scarcity [12]. Treated water has the potential to be a valuable product rather than a waste [10].

Zero liquid discharge (ZLD) is an ambitious wastewater management strategy that uses cost-effective methods to concentrate brine to near or complete dryness, with the majority of water recovered for reuse [13]. Zero liquid discharge desalination is regarded as a solution to the brine disposal problem because it produces only solid salts byproducts and clean water from the source water [14]. ZLD eliminates the risk of pollution associated with wastewater discharge and maximizes water usage efficiency, achieving a balance between freshwater resource exploitation and aquatic environment preservation [15]. Due to stringent discharge standards, ZLD is sometimes the only way to ensure regulatory compliance [16].

Mechanical vapor compression (MVC)-based brine concentrators and crystallizers are the most widely used technologies in ZLD. Brine concentrators based on membrane processes such as forward osmosis and membrane distillation have recently received a lot of attention due to their ability to use low-grade heat as an energy source [17]. A pretreatment step is required to improve membrane performance in ZLD.

Coagulation, chemical precipitation, adsorption, flotation, advanced oxidation, and electrocoagulation are the most common pre-treatment processes in the industry today [16]. Fig. **1** shows some processes in a zero liquid discharge system.

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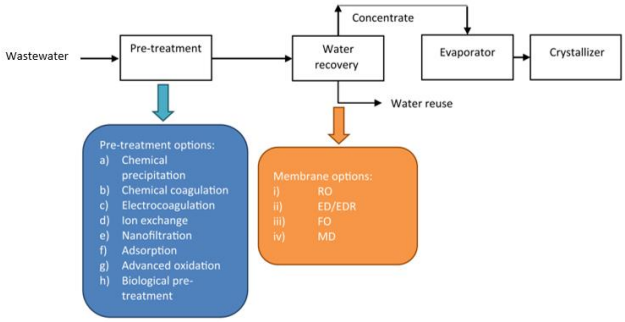

Fig. 1. The Processes of a ZLD System with Membrane and Thermal Treatment [16]

Crystallization is an important mass transfer operation that is frequently used in the manufacture of a pure product. During the process, a crystal usually is separated as a substance of definite composition from a solution of varying composition. A state of imbalance including a mass driving force, particularly a decrease in chemical potential (or concentration) between the bulk of the liquid solution and the crystal interface is required for the separation of a solid from a solution onto a crystal. As a result, the solution must be supersaturated. Although crystallization is commonly defined as the formation of a solid crystalline phase from a liquid phase via cooling, evaporation, or both [18].

Crystals have been produced throughout the history of the chemical industry using crystallization methods ranging from as simple as allowing vats of hot concentrated solution to cool to as complex as continuous, precisely controlled, multi-step processes that result in a crystal product with a specific size or size distribution, moisture content, shape, and purity [19, 20]. Crystallization can be done at high or low temperatures, and it generally requires less energy to separate pure materials than other commonly used purification methods [21].

Evaporation is a common process used in both water desalination and other feed stream treatment. It can be used to treat a wide range of feeds, including liquids, slurries, sludges, organic and inorganic streams, suspended or dissolved solids, and nonvolatile dissolved liquids [19].

Evaporation is used to concentrate a solution that contains a nonvolatile solute and a volatile solvent. The solvent in the vast majority of evaporations is water. Evaporation is accomplished by vaporizing a portion of the solvent, resulting in a concentrated solution of thick liquor. In a saturated mother liquor, evaporation can result in a slurry of crystals [22].

The primary goal of evaporation in some cases is concentrating the solution so that when it cools, salt crystals form and are separated. This type of evaporation is known as crystallization [20]. Evaporation is one of the simplest methods for crystallizing organic, inorganic, and organometallic small molecule compounds [23].

In an evaporative crystallizer, crystallization takes place by evaporating the solvent from feed, which can be either a weak unsaturated solution or a hot concentrated mixture.

When the temperature-solubility curve of a solute has a low slope, these crystallizers are used [19].

When the solution or mother liquor is saturated, equilibrium is reached in crystallization and this could be represented by a solubility curve. The temperature has the greatest influence on solubility. The effect of pressure on solubility is negligible [20, 24]. The solubilities of common salts in water as a function of temperature are depicted in Fig. 2. In most cases, the solubility of the salt increases with temperature. This means that when a hot concentrated solution from an evaporator is cooled to room temperature, crystallization may occur [20]. NaCl's solubility is characterized by a small temperature change.

This means that the NaCl solution was evaporated at a constant temperature until crystals formed [19, 20]. The most common brine treatment technologies in a ZLD system are brine concentrators and brine crystallizers [25, 26]. In our previous work Salih et al. [27] and Salih and Al-Alawy [28], the treatment of oilfield produced water from the East Baghdad oilfield with TDS = 86 g/l using three stages of the ZLD system namely oil skimming, coagulation/flocculation, and forward osmosis processes were studied. The best temperature and time for oil skimming were 40 °C and 2.5 h. which gave 95.8% removal for oil content. In the coagulation/flocculation process, the optimum PAC dosage and pH were 55 mg/l and 6.4 which gave 99.9% and 97.7% removal for oil content and TSS, respectively.

After the coagulation and flocculation process, the TDS of produced water reduced to 76 g/l. In the FO process, sodium chloride solution was used as a feed solution (FS) with a concentration of 76 g/l, while the draw solution (DS) was magnesium chloride. The produced water feed solution was concentrated to 220 g/l at DS concentration of 400 g/l $MgCl_2$ in batch mode with a constant DS concentration after 16.5 h at which the recovery was 65.67%.

This concentrated solution is considered waste and needs to be disposed of, which violates environmental regulations. If this concentrated solution is considered as waste, this does not achieve the zero liquid discharge system, which is known to end without waste. Therefore, to complete the process of the zero s liquid discharge system and recover all pure water and salts, we need to a final stage, which is crystallization.

This research aims to apply a crystallization process by simple evaporation to generate clean water and solid salts with no waste to attain zero liquid discharge system. The efficiency of the crystallization process will be estimated with different temperatures, mixing speeds, time, and concentrations of NaCl solution and oilfield produced water from the East Baghdad oilfield. The possibility of using an evaporation process to generate pure water and solid salts will be investigated with regard to evaporation rate and recovery.

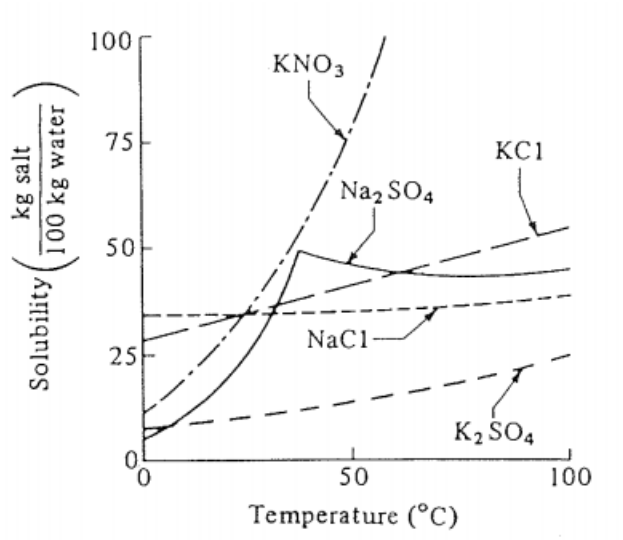

Fig. 2. Solubility Curves for Some Typical Salts in Water [20]

2- Experimental Work

Two types of feed were used: the first was NaCl solution (i.e., simulated oilfield produced water), and the second was oilfield produced water based on the concentrated feed from the forward osmosis process. NaCl (99%, India) was used to prepare a feed solution that has the same TDS as the East Baghdad oilfield produced water, The distilled water of conductivity 1.7 µS/cm was used to prepare the NaCl solution. The properties of oilfield produced water are listed in Table **1**.

Table 1. Properties of oilfield produced water from the East Baghdad oilfield after forwarding osmosis process

Characteristics	Value
TDS, mg/l	220000
Ca^{+2}, mg/l	11700
Mg^{+2}, mg/l	7475.045
SO_4^{-2}, mg/l	654.661
Cl^{-1}, mg/l	119464.21
Na^{+1}, mg/l	76500

The crystallization process was performed in a beaker of a capacity of 100 ml. Each beaker was filled with 100 ml of the produced water. All experiments were carried out under magnetic stirring at different mixing speeds (300, 400, and 500 rpm), different temperatures (70, 80, and 90 °C) measured by a thermometer, and time (0.5-9.5 h). Different concentrations (177 and 220 g/l) of the concentrated produced water from the FO process were examined. All experiments continue until most of the water evaporates leaving only salt as solid crystals and weighted by a balance.

The rate of evaporation can be calculated as follows [29]:

$$Rate\ of\ Evaporation = \frac{\Delta W}{\Delta t} \quad (1)$$

Where: ΔW is the water evaporated over time Δt.

The recovery of pure water measures how much of the feed water is recovered. Recovery was determined by using Equation 2 [30]:

$$\%Recovery = \left(\frac{V_P}{V_F}\right) * 100 \quad (2)$$

Where: V_P is the volume of water evaporated and V_F is the volume of water in the feed vessel.

Table **2** represents the range of the operating conditions and variables examined in the evaporation and crystallization processes.

Table 2. Operating Condition for Crystallization Process

Operating Conditions	Crystallization Tests
Feed solution, F_S	Simulated and real East Baghdad oilfield produced water
Mixing speed	300, 400, and 500 rpm
Temperature of feed solution, T	70, 80, and 90 °C
Feed Solution concentration, C_F	177 and 220 g/L
Time, t	0.5-9.5 h

3- Results and Discussions

The evaporation rate and recovery were measured in the crystallization process with three evaporation temperatures (70, 80, and 90 °C). As shown in Fig. **3**, the evaporation rate increased with temperature rise and decreased with time. For T=90 °C, the evaporation rate reached 16.56 g/h after 2.75 h of operation, for T= 80 °C the evaporation rate reached 12.64 g/h after 5.5 h of operation while reached to 8.2 g/h after 9.5 h of operation for T= 70 °C. Fig. **4** shows the recovery of pure water raised with temperature and time. The recovery reached 77.33% after 2.5 h of operation for T=90 °C, for the same time (t= 2.5 h) the recovery of T=80 and 70 °C were lower than that of 90 °C by 44.65%, and 69.48% respectively.

Heating the liquids causes more molecules of the liquid to gain enough kinetic energy to move faster and change to vapor. Because a molecule's kinetic energy is proportional to its temperature, evaporation occurs more quickly at higher temperatures. The higher the temperature, allowing molecules to escape from the surface of a liquid, this means the molecules near the surface have higher chemical potential (escape tendency) than other molecules in the liquid. When the liquid molecules collide, they transfer energy to each other based on how they collide. When a molecule near the surface absorbs enough energy to overcome the vapor pressure, it escapes and becomes vapor in the surrounding air. This increases the rate of evaporation and recovery. These findings are supported by those obtained by Misyura [24].

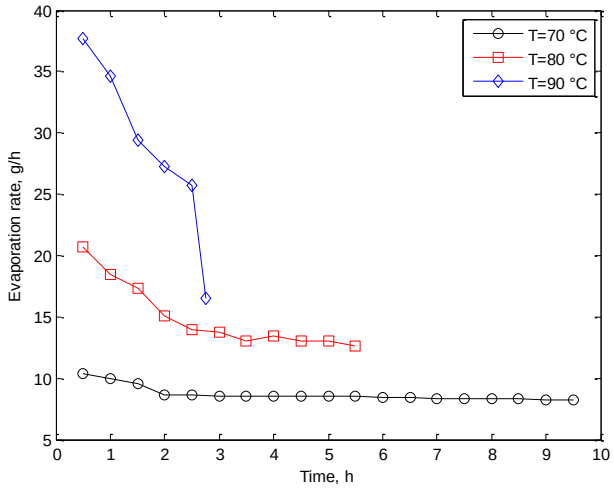

Fig. 3. Evaporation rate as a function of time for simulated PW as FS ($C_{FS,i}$=220 g/l, mixing speed=400 rpm)

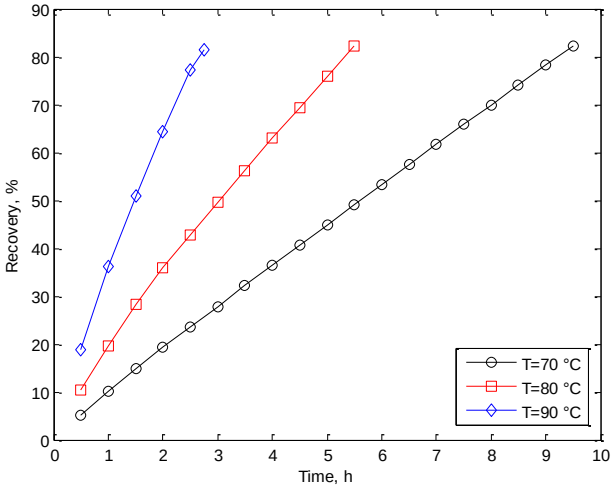

Fig. 4. Recovery of pure water as a function of time for simulated PW as FS ($C_{FS,i}$=220 g/l, mixing speed=400 rpm)

The evaporation rate and recovery were measured in the crystallization process with three mixing speeds (300, 400, and 500 rpm). As shown in Fig. **5**, the evaporation rate increased with rising mixing speed and decreased with time. For mixing speed=500 rpm, the evaporation rate reached 13.36 g/h after 5 h of operation, for mixing speed=400 rpm the evaporation rate reached 12.64 g/h after 5.5 h of operation while reached 12.52 g/h after 6 h of operation for mixing speed=300 rpm. Fig. 6 shows the recovery of pure water raised with mixing speed and time. The recovery of pure water reached 81.63% after 5 h of operation for mixing speed=500 rpm, for mixing speed=400 rpm, the recovery reached 82.22% after 5.5 h of operation, while it reached 80.79% after 6 h of operation for mixing speed=300 rpm.

The influence of mixing involves adding mechanical energy to a stable system. This mechanical energy is absorbed by the liquid being mixed as a result of billions of collisions between the stirrer and the liquid colliding with it, as well as other liquids.

This energy is equivalent to heat, so the entire system is heating up. The molecules will break free from the liquid's bonds and fly away as vapor, i.e., vaporize, the higher the mixing speed, the greater the particle breakage which prevents particles from aggregating. Therefore, an average mixing speed of 400 rpm was chosen. These findings are supported by those obtained by Choi [21].

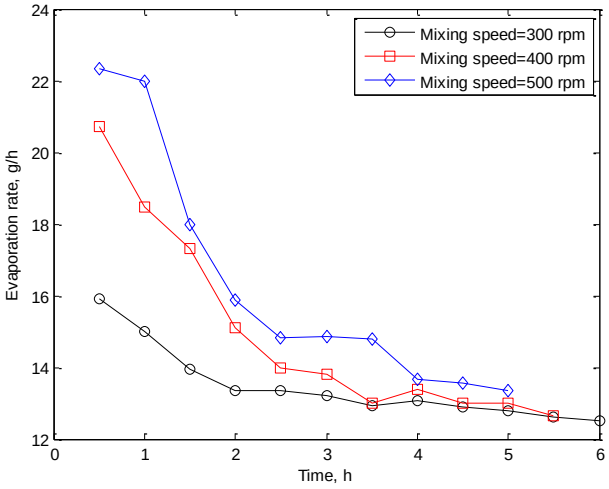

Fig. 5. Evaporation rate as a function of time for simulated PW as FS ($C_{FS,i}$=220 g/l, T=80 °C)

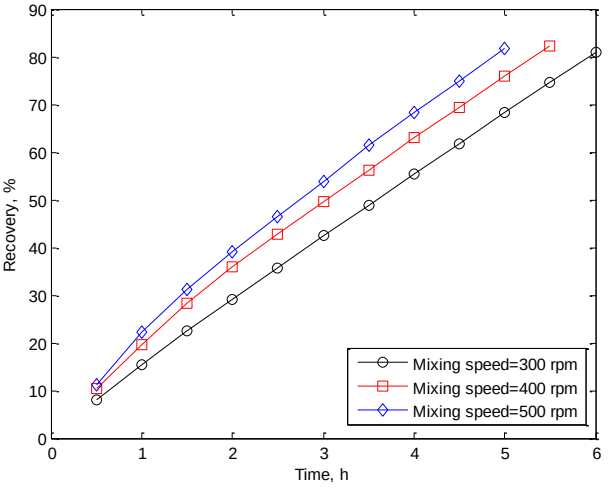

Fig. 6. Recovery of pure water as a function of time for simulated PW as FS ($C_{FS,i}$=220 g/l, T=80 °C)

The evaporation rate and recovery were measured in the crystallization process with two feed concentrations (177 and 220 g/l). As shown in Fig. **7**, the evaporation rate increased with decreasing feed concentration and time. For feed concentration=177 and 220 g/l, the evaporation rate reached 12.8 and 12.64 g/h, respectively after 5.5 h of operation. Fig. **8** shows the recovery of pure water raised with time and decreased with increasing feed concentration. For the same time of 5.5 h, the recovery of the feed concentration=220 g/l was lower than that of 177 g/l by 3.16%. Water activity decreases as the ion mass fraction (feed concentration) increases. This is due to a decrease in the chemical potential of the water. These findings are agreed with Naillon et. al. [31].

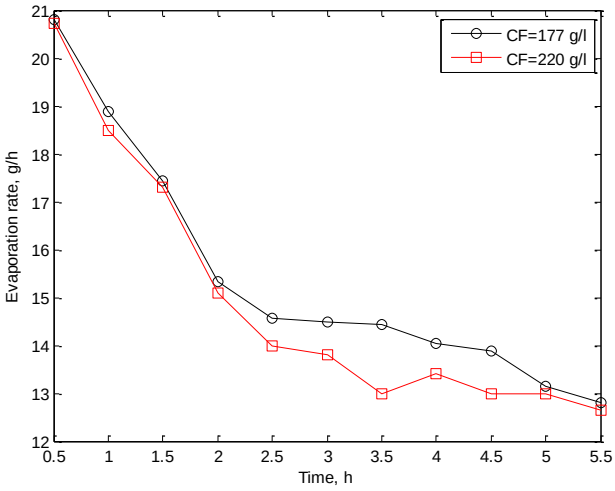

Fig. 7. Evaporation rate as a function of time for simulated PW as FS (T=80 °C, mixing speed=400 rpm)

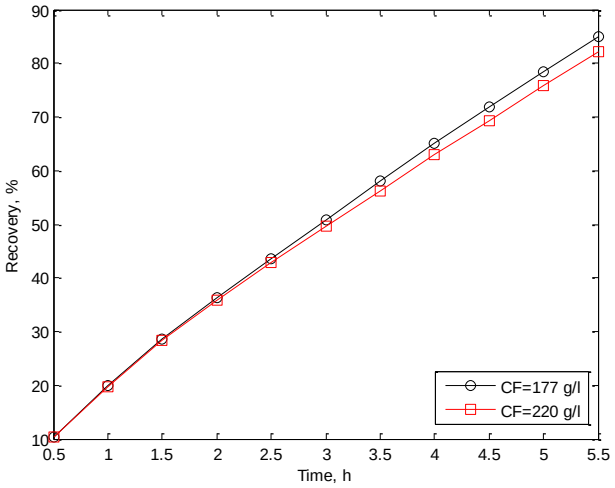

Fig. 8. Recovery of pure water as a function of time for simulated PW as FS (T=80 °C, mixing speed=400 rpm)

The evaporation rate and recovery were measured in the crystallization process with three evaporation temperatures (70, 80, and 90 °C) for East Baghdad oilfield produced water.

As shown in Fig. **9**, the evaporation rate increased with temperature rise and decreased with time.

The evaporation rate reached 16 g/h after 2.75 h of operation for T=90 °C, t, for T= 80 °C the evaporation rate reached 12.58 g/h after 5.5 h of operation while reached to 7.8 g/h after 9.5 h of operation for T= 70 °C.

Fig. **10** shows the recovery of pure water raised with temperature and time. For T=90 °C, the recovery reached 80.28% after 2.75 h of operation; for T= 80 °C, the recovery reached 80.95% after 5.5 h of operation, while it reached 80.49% after 9.5 h of operation for T= 70 °C.

The recovery reached 76.28% after 2.5 h of operation for T=90 °C, for the same time, the recovery of the T=80 and 70 °C were lower than that of 90 °C by 44.4%, and 69.59% respectively.

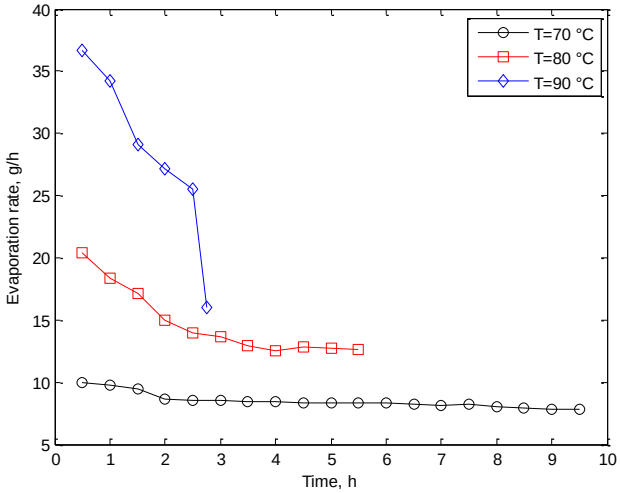

Fig. 9. Evaporation rate as a function of time for oilfield produced water as FS ($C_{FS,i}$=220 g/l, mixing speed=400 rpm)

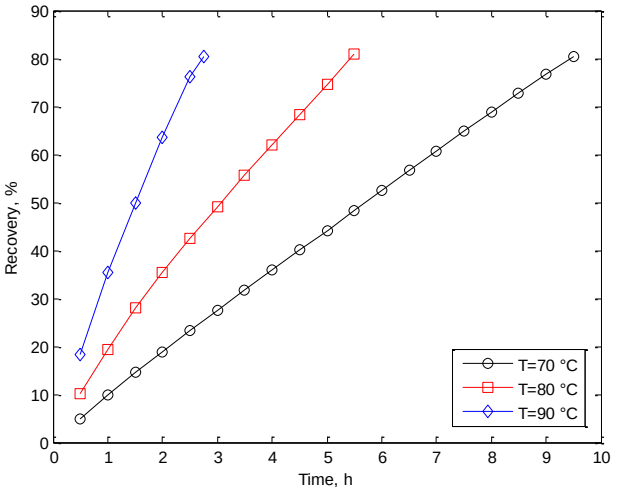

Fig. 10. Recovery of pure water as a function of time for oilfield produced water as FS ($C_{FS,i}$=220 g/l, mixing speed=400 rpm)

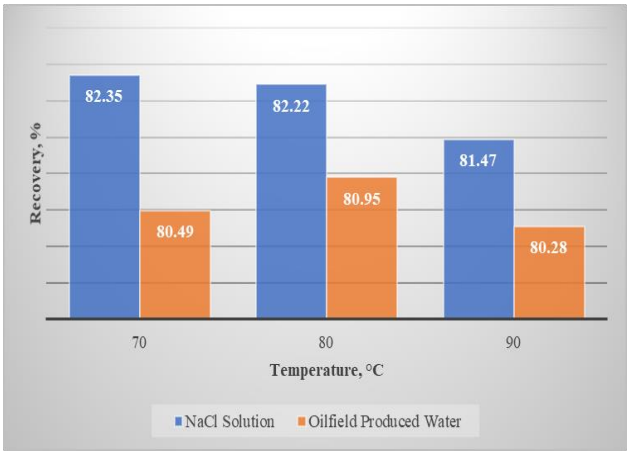

Fig. 11. Recovery of Pure Water for PW ($C_{F,i}$= 220 g/l, Mixing speed=400 rpm) for Crystallization process

Fig. **11** shows a comparison between the oilfields produced water and NaCl solution. At the end of each experiment the recovery at T=70, 80, and 90 °C for oilfield produced water is lower than that of NaCl solution by 2.26, 1.55, and 1.46%, respectively. This simple difference in results is because the oilfield produced water contains a mixture of salts, not only NaCl, but the largest proportion of the salts is NaCl.

4- Conclusion

This work shows that a crystallization process is an effective and viable option as a final part of a zero liquid discharge system for the treatment of high salinity oilfield produced water. The recovery for a temperature of 80 °C was 82.22 and 80.95% after 5.5 h for NaCl solution and oilfield produced water, respectively. While for 90 °C the recovery was 81.47 and 80.28% for NaCl solution and oilfield produced water after 2.75 h. The best mixing speed was moderate at 400 rpm. There is a small difference in the pure water recovery between NaCl solution and oilfield produced water.

Acknowledgment

The authors are grateful for the support of Thabit Abbas Ahmed and the Petroleum Research and Development Center staff during the experimental work.

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عملية البلورة كجزء نهائي من نظام التصريف السائل الصفري لمعالجة المياه المصاحبة لحقل شرق بغداد النفطي

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الخلاصة

تم في هذه الدراسة تطبيق عملية التبلور للمياه المصاحبة لحقول النفط من حقل شرق بغداد النفطي التابع لشركة نفط الوسط (العراق). يتكون نظام التصريف السائل الصفري (ZLD) من عدة أجزاء مثل قشط الزيت والتخثر / التلبد والتناضح الأمامي والتبلور ، وتعد عملية التبلور جزءًا أخيرًا من نظام التصريف السائل الصفري. تم استخدام نظام التبخر البسيط على نطاق المختبر لتقييم أداء عملية التبلور. في هذا العمل تم استخدام محلول كلوريد الصوديوم والمياه المصاحبة لحقول النفط من حقل شرق بغداد كمحلول داخل بتركيز 177 و 220 غم/ لتر. تم دراسة تأثير درجة الحرارة (70 و 80 و 90 درجة مئوية) وسرعة الخلط (300 و 400 و 500 دورة في الدقيقة) وتركيز الداخل (177 و 220 غم / لتر) والوقت (0.5–9.5 ساعة) على أداء التبلور بالنسبة لمعالجة المياه المصاحبة لحقول النفط، تم فحص معدل التبخر والاسترداد. يزداد الاسترداد مع زيادة درجة الحرارة وسرعة الخلط بينما يتناقص مع زيادة تركيز الداخل. تم استعادة المياه النقية والأملاح من الماء المصاحب المركز. كان استخلاص الماء النقي عند 80 درجة مئوية و 400 دورة في الدقيقة و 220 غم / لتر تركيز الداخل 82.22 و 81.35٪ بعد 5.5 ساعة لمحلول كلوريد الصوديوم (أي المياه الممصاحبة لحقل النفط المحاكاة) والمياه المصاحبة لحقل النفط ، على التوالي.

الكلمات الدالة: التبلور, التبخير, نظام التصريف السائل الصفري, المياه المصاحبة لحقول النفط العراقية.