

**EXPERIMENTAL ANALYSIS OF PRODUCED WATER
DESALINATION BY A HUMIDIFICATION-DEHUMIDIFICATION
PROCESS**

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ABSTRACT

Treatment and disposal of large amount of produced water is a considerable economic and environmental burden for oil and gas industry. On the other hand, for many remote areas with shorting of clean water supply and enriched with oil/gas production such as New Mexico, purified produced water could be a valuable water source for beneficial uses. The goal of this research is to purify produced water at wellhead, yielding water clean enough for beneficial uses like drilling, stimulating, or water flooding. A new concept of produced water purification by Humidification-dehumidification (HD) process was developed in which low-temperature energy sources, such as co-produced geothermal energy or solar energy, could be used to drive the water desalination process. The system contains humidifier where produced water meets with flowing air for evaporation and dehumidifier for clean water creation and collection. The unique feature of this process is that co-produced geothermal energy or inexpensive solar energy could be deployed. In addition, direct internal heat transfer has greatly enhanced latent heat recovery.

In this research, the concept of produced water desalination by using new HD process was laboratory investigated through a lab-constructed HD unit. The influences of the operation parameters such as feed water temperature and flow rate, carrying air flow rate on purified water quality, productivity, and water recovery were investigated. The research indicated that feed water temperature, water flow rate, as well as carry air flow rate show dramatic influence on water productivity and ion removal efficiency. Over 98% of dissolved salt was removed in a tubing-shell structured HD unit. Considerable enhancement in water productivity was achieved by deploying built-in capillary bundle as dehumidifier. The water productivity was increased from 48 to 311 ml/(m².h) with water recovery reached 20.7% at 80 °C.

Desalination experiments with actual coal bed methane produced water indicated that both salt and dissolvable organics were removed efficiently by the HD process: the total dissolved solid was reduced from 1.98×10^4 to 76.75 mg/L while the total organic carbon was declined from 470.2 to 17.83 mg/L. The purified produced water is suitable for many beneficial uses, such as agricultural irrigation and industry use.

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CHAPTER 1 INTRODUCTION AND LITERATURE REVIEW

1.1 Introduction

Produced water embodies the primary waste stream of oil, natural gas and coalbed methane production. It is very saline, sometimes nearly six times as salty as seawater, and contains dissolved hydrocarbons and organic matter as well. Produced water disposal at the production site for land restoration or other beneficial uses will be the primary option for cost-effective produced water management, due to the limitation in storage capacity and distribution technologies available at each particular site. For the purpose of direct disposal or land restoration, the deployment of advanced technologies for removing salts and dissolvable organics is generally required for attaining surface water discharge standards or reuse criteria [Dallbauman et al., 2005]. For example, in New Mexico, any disposal of produced water must meet the standard promulgated by the Oil Conservation Division (OCD) of the New Mexico Energy, Minerals and Natural Resource Department (NMEMNRD). Unfortunately, ample experiments and demonstration tests have indicated that no cost-effective technologies can effectively treat produced water to substantial quality that meets the direct disposal quality criteria. As a result, the bulk of produced water (>90%) is currently managed through a three-step process: (1) lifting produced water to the surface, (2) transportation to the disposal site, and (3) deep well injection or evaporation [Veil, 2004]. Both transportation and deep well injection are costly and offers potential contamination to groundwater systems, and thus face strict regulation. The average disposal cost for produced water in NM is ~\$2.5/bbl, with a major part of this cost attributed to its transportation in most of the producing areas of NM [Veil, 2004].

The economic burden posed by produced water disposal can potentially convert an uneconomic production from otherwise beneficial wells, particularly those marginal wells with poor establishment in water transportation systems. As a result, some of the operations were forced to be abandoned. In addition, many oil/gas exploration activities render land more vulnerable to degradation, because of the scarcity of water. The effects of this potential degradation threaten to restrict energy exploration, and the cost of the subsequent infrastructure degradation could be substantial. Purified produced water can be a valuable clean water resource for restoring the landscape and maintaining native vegetation. Considering the undesirable effects of high costs and land degradation, it is desirable to develop a method of cleaning produced water for directly disposal at wellhead.

High concentration of salts and hydrocarbons are generally present in produced water. The conventional oil/water separations used in oil/gas industry, such as gravity separation and microfiltration process, have no influence on the concentration of dissolved components in produced water. However, the presence of dissolved components shows a major impact on the receiving environment due to their toxicity. The primary concern is the high salt concentration which causes soil degradation and serious corrosion and dissolved hydrocarbons. Many of the dissolved hydrocarbons are toxic, particularly the chemicals with small molecule aromatics, such as toluene, benzene, ethylbenzene and xylene (BTEX). In some cases, dissolved metal ions and radioactive elements are also dissolved in produced water and must be removed for disposal. Typical dissolved components in produced water are shown in Table 1-1.

Table 1-1 Typical chemical composition of dissolved components in produced water

Components	
Dissolved salt	Cations: Na^+ , K^+ , Ca^{2+} , Mg^{2+} , Ba^{2+} , Sr^{2+} and Fe^{2+} Anions: F^- , Cl^- , SO_4^{2-} , CO_3^{2-} and HCO_3^- .
Dissolved organics	Fatty acids Aliphatic Aromatics (benzene, toluene, and ethylbenzene) Phenols
Heavy metals	Cadmium (Cd), Chrome (Cr), Copper (Cu), Lead (Pb), Mercury (Hg), Nickel (Ni), Silver (Ag), and Zinc (Zn).
Radioactive elements	Radium 226 and radium 228

In the past decade, many efforts have been made to purify produced water to substantial quality for disposal or beneficial use. The conventional demineralization technologies include reverse osmosis (RO), distillation, electrodialysis, freeze desalination, and ion exchange [Cox et al., 1993]. Technologies for dissolved organic removal include air stripping, activated carbon adsorption, synthetic zeolite adsorption, membrane filtration, biological treatment and wet air oxidation [Hansen et al., 1992]. Each technology has its limitation in produced water purification due to the complex chemical composition. Table 1-2 summarizes the approaches of dissolved component removal from produced water and characteristics of each technology.

Table 1-2 Technologies for the removal of dissolved components from produced water

Technologies	Application	Efficacy	Challenges	Ref.
Adsorption	Organic removal	>99% BTEX removal	TOC<100 mg/l	Noll, K.E., 1992
Biological treatment	Organic removal	>98% BTEX removal	TOC<100 mg/l TDS<3.6×10 ⁴ mg/l	Hickey, et al., 1999; Tellez and Nirmalakhandan, 1992
Freeze demineralization	Ion removal	>95% ion removal	Site specific and large land requirement	Boysen et al., 2002
Ion exchange	Ion removal	Efficient in softening water	Fouling by high concentration of Ca ²⁺ and Mg ²⁺ . TDS<5000 mg/L	Brown and Sheedy, 2002.
Electrodialysis	Ion removal	Low pressure operation, good fouling resistant	TDS<15000 mg/L, organic removal	Tsai, et al., 1995
Reverse osmosis	Ion and organic removal	>95% ion/organic removal	Serious membrane fouling	Rousseau, 1987
Precipitation	Multivalent ion removal			

Even with rapid advancement in water demineralization technologies, water purification at the wellhead would need to be tailored to meet the specific characteristics of water production in each individual well. First, the amount of water at each particular site is limited by well production and by available storage capacity and distribution pipelines at the site. The

desalination technology used must be efficient for application in small or medium-scale water treatment scenarios and insensitive to the variability in water chemistry. Secondly, formation and production history will have a dramatic influence on produced water quality and how the purification technology can be deployed. The produced water purification process must be highly efficient for produced waters with variable chemistry. Finally, any sophisticated pretreatments must be avoided due to the high operation cost; the novel strategy of produced water purification must be tolerant of solid suspensions and floating oil. Unfortunately, the membrane technologies, such as reverse osmosis and electrodialysis, generally require multistage pretreatment for fouling removal and are only economically competitive for large quantities of water (i.e. $>1000 \text{ m}^3/\text{d}$) [Ettouney et al., 2004]. Thermal processes have advantages of high purity product but are limited by the high energy intensity associated with phase changes. Water purification tactics must be adapted to meet the unique characteristics of produced water production from oil/gas and CBM production field, particularly addressing the limitations in well production and water availability at each particular site.

To overcome these technical and economic barrier of produced water desalination by conventional technologies, novel processes that renewable energy can be used are reviewed for potential application in produced water desalination. A specific feature of produced water is that a large quantity of geothermal energy could be co-produced during oil production. By deploying the low-cost energies, a considerable decline in operating cost is expected. In addition, many oil gas active areas, such as western of United States, are enriched with solar radiation. The high solar radiation intensity and the deep reservoir formation (located in southeastern New Mexico) make it feasible for deploying or integrating solar and coproduced geothermal energies for produced water heating and desalination.

1.2 Desalination Process

1.2.1 Desalination Process Multi-stage flash (MSF) process

Multi-stage flash process (MSF) is a water desalination process that distills salty water by flashing feed water into steam in multiple stages. Multistage Flash Desalination has a market share of over 60% of the worldwide desalination market and this share jumps to almost 80% in middle-east [Borsani et al., 2005]. The MSF process is characterized by large size of its production plants and high “gain ration” which means ten pounds of water could be produced from each pound of steam [Al-Enezi et al., 2006; Othmer, 1969].

Figure 1-1 gives a schematic diagram of multi-stage desalination process. As shown in the figure, a MSF desalination system includes several chambers in which pressure decreases from the first chamber to the last one. After salty water is heated by brine heater, it will be introduced into the first desalination chambers known as the "a stage", where the surrounding pressure is lower than that in the brine heater. Because of the pressure reduction, water will be boiled rapidly here and flashed into steam. However, only a small percentage of water will be converted into steam. The remaining water will be sent to a series of additional stages with increased vacuum. The generated steam is condensed into fresh water and collected as clean water. Seawater is heated in the heat exchanger by condensation of the vapor produced in each stage. The cold seawater enters into the desalination chamber in a counter flow with the waste brine and distillate water. The heat energy released from condensation is used to preheat the cold seawater.

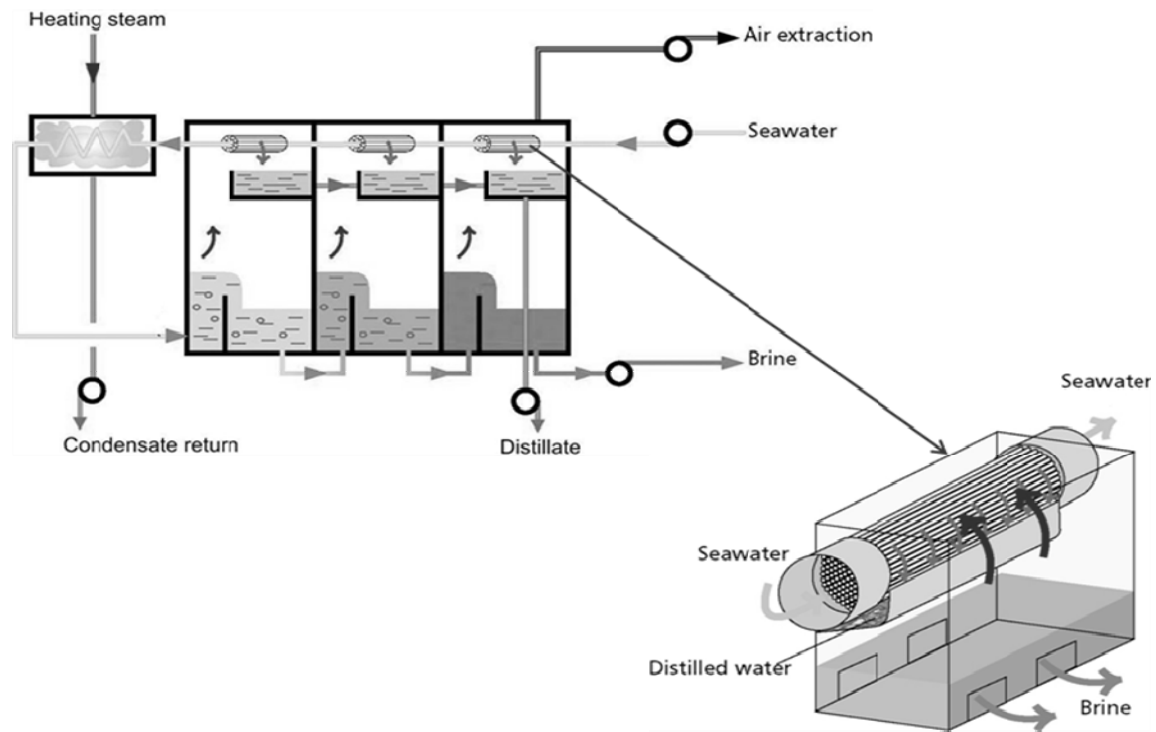


Figure 1-1 Diagram of multi-stage flash process (MSF) desalination process.

1.2.2 Multi-effect boiling (MEB) process

Multiple-effect boiling distillation is a water desalination process that salty water is boiled in a series of chambers by the submerged tubes with steam circulated inside. Figure 1-2 shows a schematic diagram of multi-effect boiling process. The MEB system consists of several effect cells in which the surrounding pressure and temperature decrease continuously. As shown in Figure 1-2, brine is sprayed into individual cells from the top, meanwhile heated steam is introduced into the tube bundles which function as “boiler”. Salty water is warmed up by the energy provided by the heated steam in the tube bundle and partly evaporates. The steam flows

into the steam tube on the top of cell which can be used for next cell's energy source. At the bottom of each cell, there are two outlet exits: concentrated water outlet and condensate water outlet. The concentrated water outlet is used for draining un-evaporated water. The condensate water outlet is used to collect clean water after condensation. The latent heat from the water condensation could be reused to warm up feed water and reduce the energy consumption.

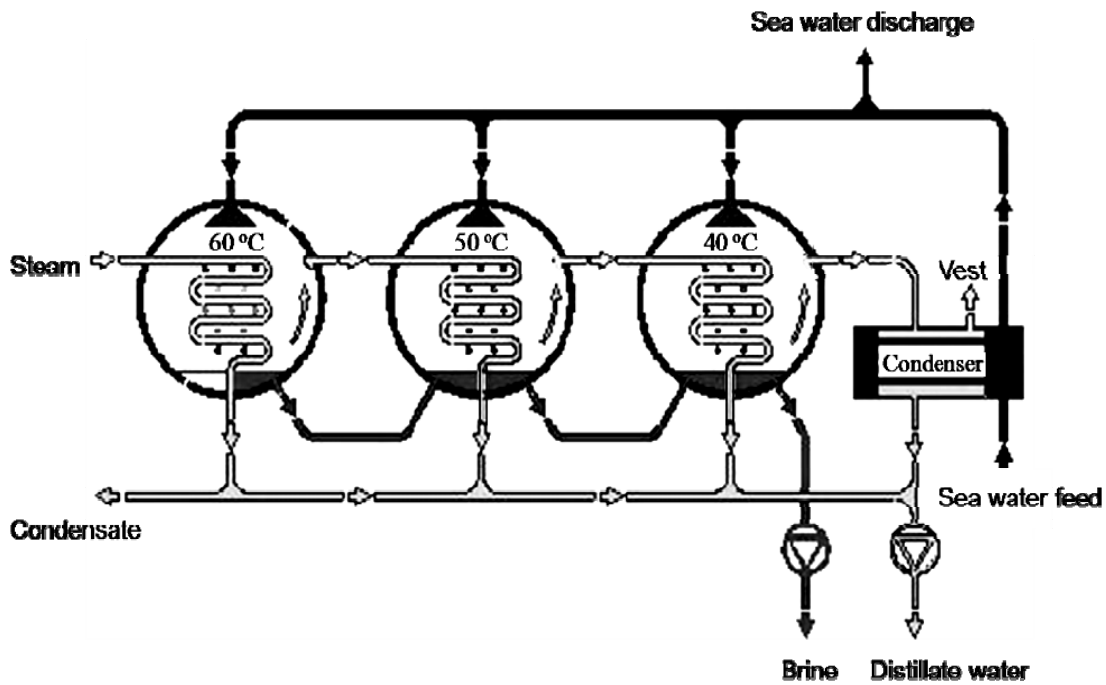


Figure 1-2 Diagram of multi-effect boiling (MEB) desalination process.

1.2.3 Humidification-dehumidification process

Humidification-dehumidification is a heat-based desalination process involving enhanced water evaporation in the presence of flowing air and water condensation upon subsequent

cooling or capillary condensation. The typical characteristics of the humidification-dehumidification process include flexible capacity, atmospheric pressure operation, and use of low-temperature energies such as solar, geothermal, and waste heat from industries. Conventionally, humidification dehumidification has been carried out in two separate columns, one for humidification and the other for dehumidification. In one aspect, it will increase the complexity of the structure of the unit and capital cost for construction [Bourouni et al., 1997; Nawayseh et al., 1997; Al-Hallaj et al., 1998; Dai et al., 2004; Hashemifard et al., 2004]. In another aspect, the phase-change invoked energy consumption only come from sensitivity heat carried by feed water or carrier gas limited water evaporation capacity. At the same time, large quantity of latent heat will be released during vapor condensation process in a separate column. Even specific designs are generally deployed to reuse the latent heat; only partially latent heat can be reused. To overcome the high energy consumption during the phase conversion process, the latent heat released by the dehumidification process could be deployed for feed water preheating. In recent years, Beckman et al., has developed a Dewvaporation process in which the heat released from vapor condensation can be reused directly for water evaporation and dramatically enhances the efficacy of latent heat reuse. In the dewvaporation process, humidification and dehumidification occur continuously in two chambers with an internal liquid heat exchanger on the evaporation side. Room temperature air is used as carrier-gas to evaporate salty water and to form pure condensate from dew at constant atmospheric pressure. The heat needed for evaporation is supplied by the heat released by dew fall condensation on opposite side of a heat exchange wall [Hamieh et al., 2006]. This process is formed by three major parts: humidifier, where the air was drove into and humidified to saturation condition; condenser,

where the water vapor can be condensed for clean water creation; and water pre-heat system, where the feed water is further heated to elevate temperature by waste energy.

One limitation of the referenced desalination process is its low water recovery and high sensitivity in heat loss [Parekh et al., 2004]. More recently, a process named AltelaRain™ technology has been utilized by Altela, Inc [Bruff, 2006]. Water was evaporated in the presence of flowing air, forming humidified air. Purified water is then collected as the air is subsequently cooled. The challenge for their distillation process is the high energy intensity needed for phase conversion from liquid to gas. Use of latent heat recovery for improved energy efficiency has been investigated [Parekh, et al., 2004]. As a consequence of water vapor condensation, large amount of latent heat will be released and collected for water preheating [Hamieh, et al., 2006].

CHAPTER 2 RESEARCH OF PRODUCED WATER DESALINATION

2.1 System description and operation principle

Various low-temperature desalination units have been constructed base on the principle of humidification-dehumidification. The basic principle of all these techniques is to convert salt water to humidified air and then condense the water vapor for clean water creation.

The low-temperature distillation is based on the fact that humidified air at elevated temperatures (60-80 °C) carries a large amount of water vapor. Water-saturated hot air at 30 °C and 80 °C has a water partial pressure of 31.8 mm Hg and 355.1 mm Hg; a large amount of water (~517 g/Kg air) can be condensed as clean water when humidified air is cooled from 80 °C to 30 °C. The difference in water vapor pressure at varied temperatures gives the estimate of water treatment capacity, as shown in Figure 2-1.

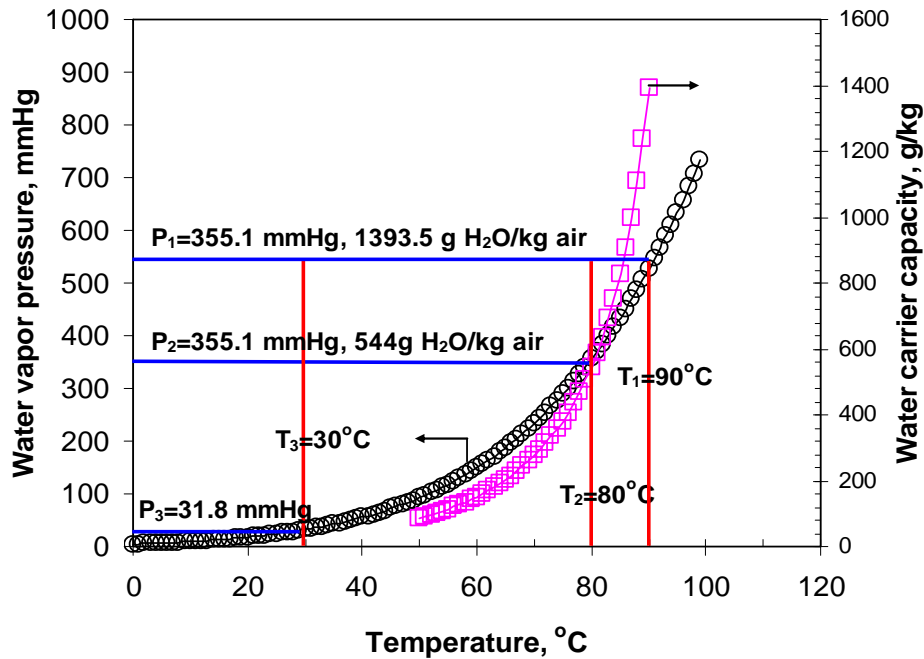


Figure 2-1 Water partial pressure as a function of temperature.

The specific energy consumption of the air-enhanced water distillation includes water heating, evaporation, and mechanical energy for pumping water and blowing air. To create 1.0 Kg clean water from produced water, the specific energy requirements for water heating, evaporation and the air blower are ~229.9 KJ, 2260 KJ, and 7.9 KJ, respectively [Parekh, et al., 2004]. The large amount of latent heat expected in phase conversion (i.e. water evaporation) is the main cause of the energy intensity of conventional distillation processes. One way to lower energy consumption is to reuse the latent heat for water heating and evaporation [Hamieh et al., 2006; Bourouni, et al, 2001].

From the three main parts of this system, operation principle would be concluded as: (1) improving external heat equipment for deploying different kind energy at each situation or

location, (2) enhancing energy efficacy of desalination system by increasing humidity efficiency, condense capacity or latent heat reuse.

2.2 Objective of study

The primary aims of this study are: (1) to conduct experimental studies for the concept of produced water desalination by humidification-dehumidification water distillation process, (2) to determine the influence of operation parameters on humidification-dehumidification separation performance, (3) to characterize the water production and energy efficiency and improve the energy efficacy by using built-in capillary condenser, (4) to investigate the purification application of humidification-dehumidification process in coalbed methane produced water purification.

2.3 Scope of study

This research focuses on produced water purification at the wellhead and beneficial uses in oil production, i.e., drilling fluid, stimulating fluid, and water flooding. Experiments have been carried out to evaluate the humidification-dehumidification process for produced water desalination. First, sequences of desalination test on NaCl solutions were tested for optimization of operating parameters, such as air/water ratio, feed water temperature, and air/water flow rate. Then, technologies for enhancing energy efficiency were investigated. Measures undertaken for enhancement in latent heat recovery and energy efficiency include: (1) built-in water condensers with different configurations for enhancement of latent heat recovery, (2) optimizing process for enhancement of heat/mass transfer, and (3) deployment of renewable energies, including solar

energy and coproduced geothermal energy, for driving the water desalination process. Finally, produced water desalination by humidification-dehumidification process has been tested. Separation performance including ion removal efficiency and impact on organic removal were studied.

CHAPTER 3 EXPERIMENTAL SETUP

3.1 Experimental setup

The produced water humidification-dehumidification purification system consisted of a water heating and delivery system, an evaporation and condensation chamber, and the clean water collection and concentrate water recycle system. Both the feed and clean water were collected in a time period and stored at 5 °C for chemistry analysis. Figure 3-1 shows a schematic diagram of this system.

The water heating and delivery system includes a cole-parmer temperature bath and a Masterflex pump. Feed water was heated to 60 °C, 70 °C or 80 °C in a water bath. When temperature reached a preset value, produced water was introduced into the top of humidification-dehumidification chamber by a Masterflex pump at a fixed flow rate. The feed water drained down through a water distributor to form thin water film. Meanwhile, the air supplied by a centrifugal blower moved in a counter direction from the bottom of evaporation chamber to the condensation chamber and further contacted with the water film. Humidified air formed during the counter movement of air and water film. The humidified air kept flowing into the condensation chamber and formed condensate upon cooling and capillary condensation. Condensate purified water exited from the bottom of humidification-dehumidification chamber to a clean water collection bottle. At the same time, the concentrated wastewater was circulated through a pipe to the produced water tank. Distilled water was added manually into the feed water to maintain a constant ion concentration during the whole experimental process. Both the feed water and purified clean water were collected every two hours for chemistry analysis.

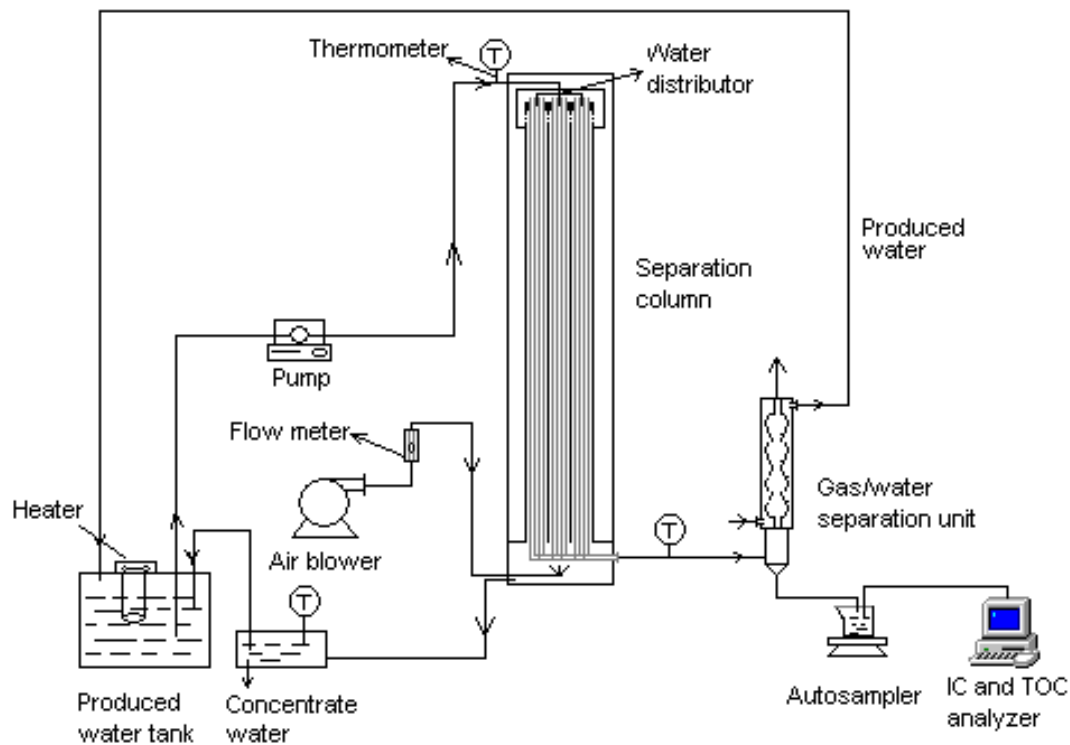


Figure 3-1 Schematic diagram of air-enhanced humidification-dehumidification process for produced water desalination.

The produced water humidification-dehumidification system has the similar structure described by Xiong and coworkers [Xiong, et, al. 2005]. The humidification and dehumidification chambers were constructed by plastic shell and 124 copper tubes. Because of two different humidification-dehumidification column design, this produced water desalination experiments were carried out by using two different humidification-dehumidification media:

The first separation column was built by plastic shell column and copper pipes as humidifier and heat exchanger. Figure 3-2 gives the schematic diagram of the copper tubing

separation column. Copper tubes with outside diameter of 6.35 mm and length of 1.8 m were bundled and embedded into a plastic column. Produced water was directed through a water distributor which was made by 124 microbore tubes with inner diameter of 0.25 mm. On the bottom of the column, there are dry air inlet, clean water outlet and concentrated water outlet pipes which connected with produced water tank for feed water circulation. Produced water was directed through the water distributor to inner surface of the copper tubes and contact with up-flowing dry air which was blown from the bottom of the column. Humidified air will be generated during the counter movement of falling water film and up-flowing air stream inside the copper tubes. Humidified air stream flow to the condensation chamber and clean water start to condense at the outside walls of the copper tube because of temperature difference between inside and outside wall of the copper tubing. As the condensate is generated, large quantity of latent heat will be released and will transport to the inside wall surface of the copper pipes. As a result, the heat released by water condensation will compensate water-evaporation provoked temperature decline and enhanced water evaporation. The total heat exchange area of 124 copper tubes in this experiment is 4.5 m^2 .

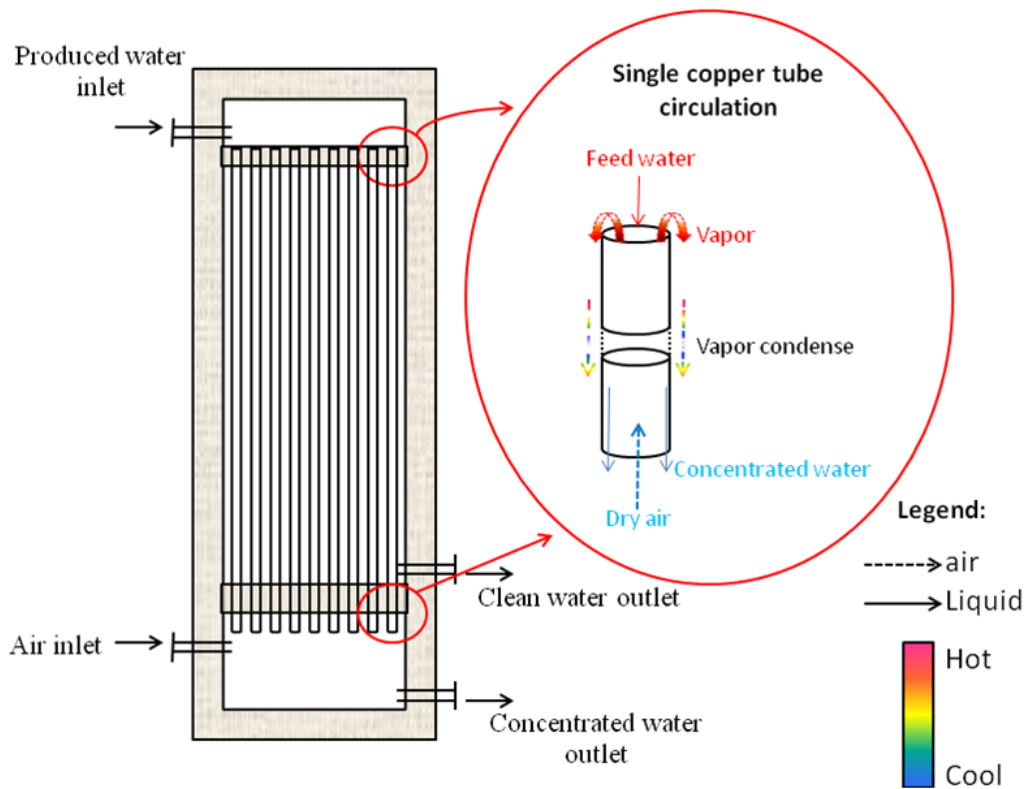
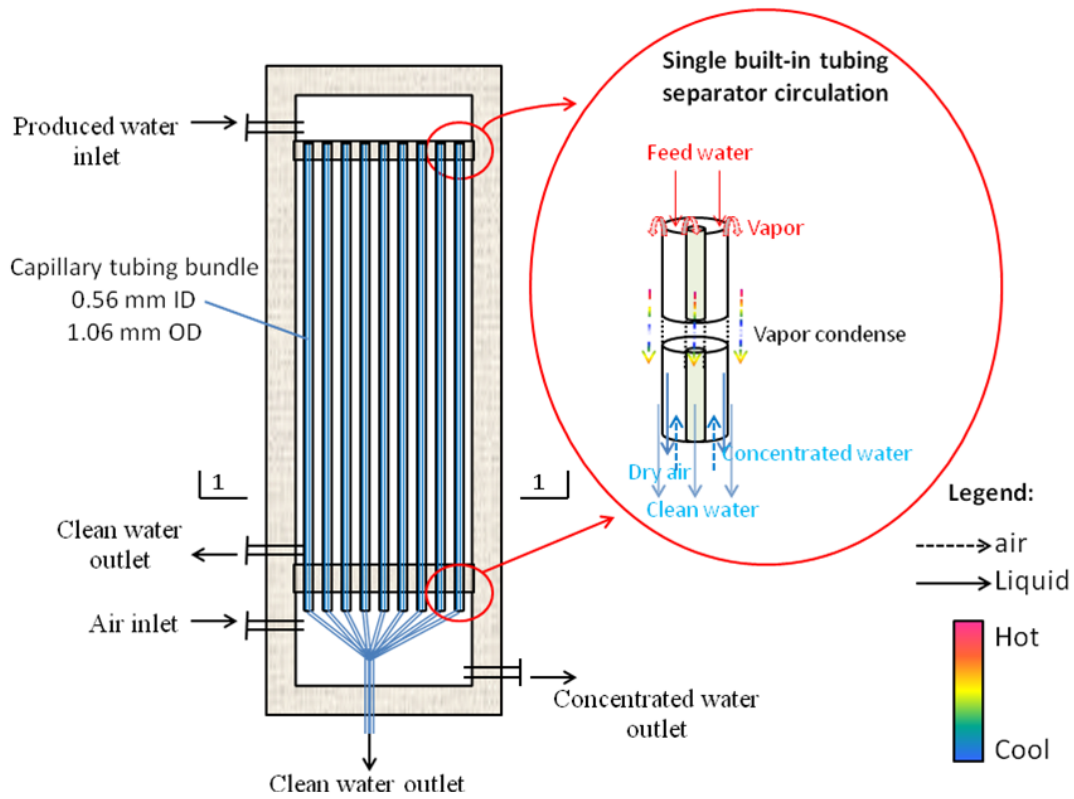
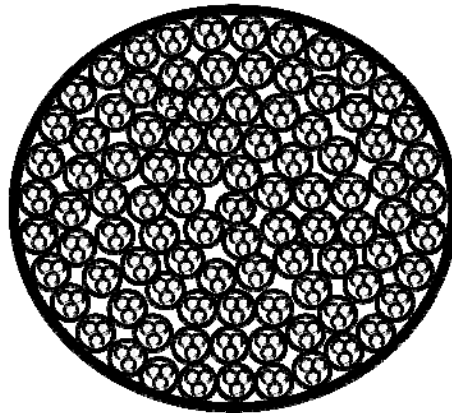


Figure 3-2 Schematic diagram of the copper tubing separation column

To enhance the water productivity and heat efficiency, capillary tubing bundles were embedded in the copper tubes and function as built-in condensers. The capillary tubing used here has an inside diameter (ID) of 0.56 mm and outside diameter (OD) of 1.06 mm. Each copper tube contains three built-in capillary tubes, giving an overall heat exchange surface area of 1.2 m². Figure 3-3 schematically shows the built-in capillary tubing bundle embedded in a copper casing.



(a)



(b)

Figure 3-3 Schematic diagram of built-in capillary bundle tubing for enhanced water. (a) Built-in capillary bundle tubing separator and circulation, (b) Separator column transect 1-1.

As shown in Figure 3-3, the feeding water formed a thin water film on the external surface of the built-in capillary tubing. Air was forced to flow in the interspacing of capillary tubing in a countermovement by a centrifugal air blower. The up-flowing dry air further contacted with water film, generating humidified air stream. The humidified air was then directed to the interior cavities of capillary tubing and moved downward. In that process, water condensed at the interior cavities of capillary tubing upon cooling and capillary condensation. The purified produced water was collected at the bottom of air/water outlet.

3.2 Chemicals and equipment

Chemicals used in this study include NaCl (ACS, >99%), cation standard (3600 ppm), anion standard (5000 ppm). Coalbed methane produced water samples were taken from the local disposal site of San Juan Basin of New Mexico State. The produced water contains 19792.8 mg/L total dissolved solid, 99.6 mg/L total suspended particulates and 470.2 mg/L total organic carbon.

A water bath (17L, Polystat®) was used to heat salty water and produced water to desirable temperatures. The variable Autotransformer (Type: 3PN1010, Staco Energy Products CO.) and centrifugal blower (Cole-Parmer) were used for water and air delivery.

3.3 Analysis

Ion concentration of both feed and purified water samples were analyzed by ion chromatograph (IC, DX-120, Dionex). All water samples were filtrated by sterilizing filters (0.2

µm, Fisher) to remove suspended particulates floating oil. Water samples were diluted to desirable concentrations (~50 mg/L) before manually injected into the IC for cation and anion analysis.

A dynamic light scattering particle analyzer (Nanotracs NPA 150) was used for study of particle size distribution of the produced water. Concentrations of dissolved organics were represented by the TOC (Total Organic Carbon) and analyzed by a TOC analyzer (Shimadzu, TOC-V). Metal ions were analyzed by the flame atomic absorption (Varian Model 110).

CHAPTER 4 PROCESS OPTIMIZATION FOR ENHANCED WATER RECOVERY

Water productivity of the humidification-dehumidification process was investigated with the two condensers: (1) copper tubing condenser and (2) built-in capillary bundles. Few experiments were carried out to test the performance of these two separation column with different condensers such as productivity, water recovery and heat efficiency. Compare with two separation column, the one with high productivity, high water recovery and high heat efficiency is suitable for oil filed use.

4.1 Copper tubing condenser

Water purification by the HD process with copper tubing condenser was tested under certain conditions and water recovery at different operation parameters was investigated. The influence of feed water temperature, flow rate of inlet water and flow rate of inlet air were investigated.

(1) The effect of carrying air flow rate test: this group test was made at constant feed water temperature of 80 °C and flow rate of 20 ml/min . The carrying air flow rate was varied from 1250 LPH (liter per hour) to 1500 LPH and to 2500LPH.

(2) The effect of feed water temperature test: the flow rate of feed water and carrying air were set at 20 ml/min and 1250 LPH respectively. Feed water was heated up to 60 °C, 70 °C and 80 °C for three separate tests.

(3) The effect of feed water flow rate test: the effect of feed water flow rate test were tested at constant feed water temperature of 80 °C and carrying air flow rate of 1250 LPH. Feed water flow rate was set at 20 ml/min, 27 ml/min and 32 ml/min respectively. 0.1 mol/L NaCl solution was used for all the testes. It was observed that the system needed about 4 hours to warm up. Thus, the first water sample was collected after starting the test for at least four hours. Then water samples were collected every three hours and water chemistry was analyzed. Each separation test was running for over 12 hours at constant room temperature until three consecutive water samples gave the same water recovery: an indication of stabilized operation.

Water productivity, water recovery, ion remove efficiency and organic remove efficiency are four general parameters to test the quality of the water purify unit. Water productivity (WP) is defined by following equation,

$$WP = \frac{Q_w}{A \times \Delta t} \quad (4-1)$$

where Q_w is the amount of water collected from a dehumidifier in the time period Δt , which Q_w and Δt are expressed as kg and hour respectively. A is the effective humidification-dehumidification area, m^2 . In copper tube condenser test, the area is $4.5 m^2$, which is the overall heat exchange surface area of the unit. Water recovery is defined as the ratio of purified water quantity to feed water quantity. Figure 4-1, 4-2 and 4-3 show the effects of operation parameters on water productivity and water recovery.

Figure 4-1 reveals the influence of carrying air flow rate on water productivity and water recovery at feed water temperature of 80 °C and flow rate of 20 ml/min. The purified water productivity decreased from $0.0194 \text{ kg}/(\text{h}\cdot\text{m}^2)$ to $0.0159 \text{ kg}/(\text{h}\cdot\text{m}^2)$ with carrying air flow rate

increasing from 1250 LPH to 2500 LPH. Water recovery decreased from 7.3% to 6.0%. The productivity and water recovery decreasing is explained by large amount of cool carrying air entering the system. Dry carrying air was driven into the separator column at room temperature (25 °C) which is remarkable different from feed water temperature (80 °C). As the feed water mix with cool air, the temperature of the system decreased, resulting in a decline of water evaporation. Also, increasing dry air flow rate decreased the humidity at the top of humidifier which resulted in a decline in water productivity. Figure 4-2 and Figure 4-3 give the water productivity as a function of feed water temperature and flow rate. The decline of water recovery with increase of feed water flow rate suggests that the efficiency of productivity decreased with increase of feed water flow rate.

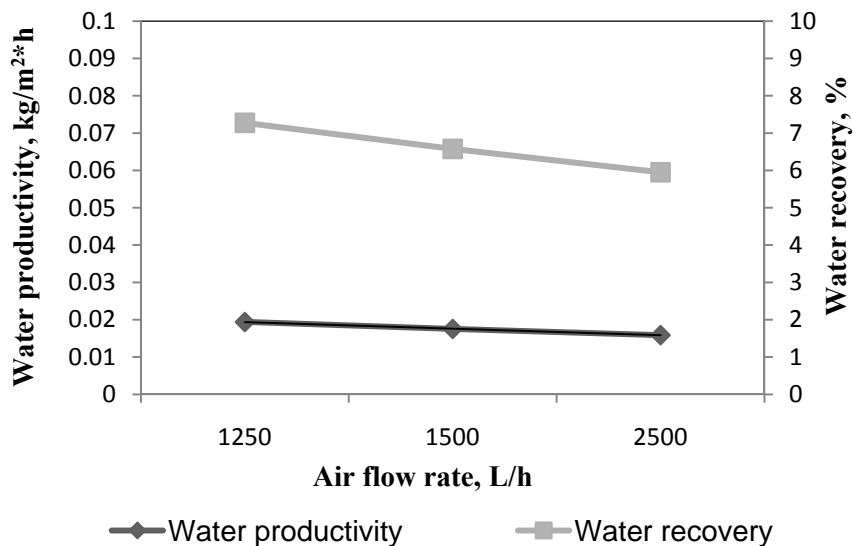


Figure 4-1 Water productivity and water recovery at varied carrying air flow rate.

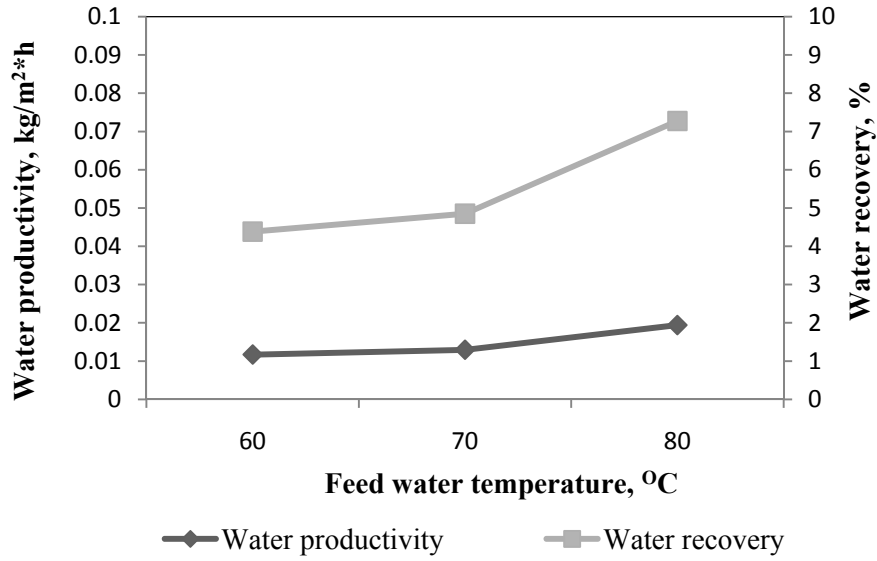


Figure 4-2 Water productivity and water recovery at varied feed water temperature.

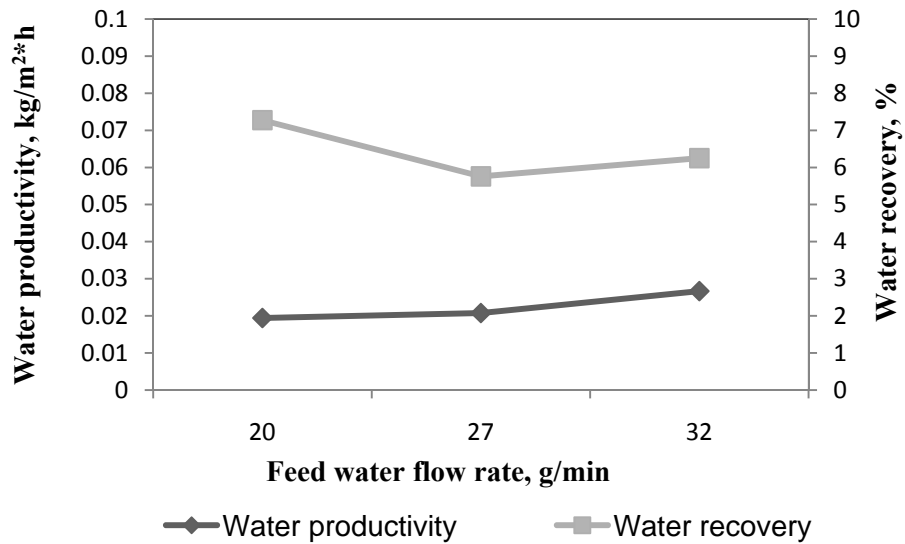


Figure 4-3 Water productivity and water recovery at varied feed water flow rate.

Purified water and feed water samples from cooper tubing condenser test was analyzed in this experiment. The ion removal efficiency is defined as ion concentration difference of the feed and purified water versus feed ion concentration. It present as equation as follow:

$$Efficiency_{ion} = \frac{C_f - C_p}{C_f} \quad (4-1)$$

where C_f and C_p are ion concentrations of the feed water and purified water respectively, ppm.

Table 4-1 shows the quality records of the system during the period of operating time. All the results show ion removal efficiency is larger than 98% which ion concentration of Na^+ decline from about 3000 ppm to less than 10 ppm. And in some conditions, ion removed almost 100% by this process.

Table 4-1 Ion removal efficiency at varied carrying air flow rate, feed water temperature and flow rate for separator with copper tubing condenser

	Carrying air flow rate (LPH)	Ion removal efficiency (%)
Ion remove efficiency at varied carrying air flow rate	1250	99.91
	1500	99.93
	2500	99.78
	Feed water temperature (°C)	Ion removal efficiency (%)
Ion remove efficiency at varied feed water temperature	60	99.85
	70	99.93
	80	99.91
	Feed water flow rate (g/min)	Ion removal efficiency (%)
Ion remove efficiency at varied feed water flow rate	20	99.91
	27	98.02
	32	98.43

4.2 Built-in capillary tubing condenser

Figure 4-4 and 4-5 give the water productivity as a function of feed water temperature and flow rate. Increasing feed water temperature or flow rate will enhance purified water production. Especially when the feed water inlet flow rate was high such as 30 mL/min, water productivity observably increased with feed water temperature increase. The productivity of the copper tubing condenser separator system were ranged from 0.006 kg/(h.m²) to 0.05 kg/(h.m²) when the feed water temperature varied from 60 °C to 80 °C and flow rate increased from 10 mL/min to 30 mL/min. And the deployment of built-in capillary tubing bundles shown considerably enhancement in water evaporation and subsequent water productivity. As shown in Figure 4-5, the built-in capillary tubing condenser separator system produced amount of purified water range from 0.03 kg/(h.m²) to 0.31 kg/(h.m²) which significantly enhanced water

production about 6 times compare with copper tubing condenser separator system. As shown in Figure 4-6, it is also interesting to observe that the water recovery was increased with the increase of feed water temperature and flow rate. We attributed such an improvement in water productivity to the fast heat transfer and enhanced energy efficiency. The condensation-released latent heat at internal surface of capillary tubing will transport across the thin walls to the external surface and compensate the evaporation-provoked cooling effect. Apparently, large air-water contact surface area and direct heat reuse will benefit the energy efficiency.

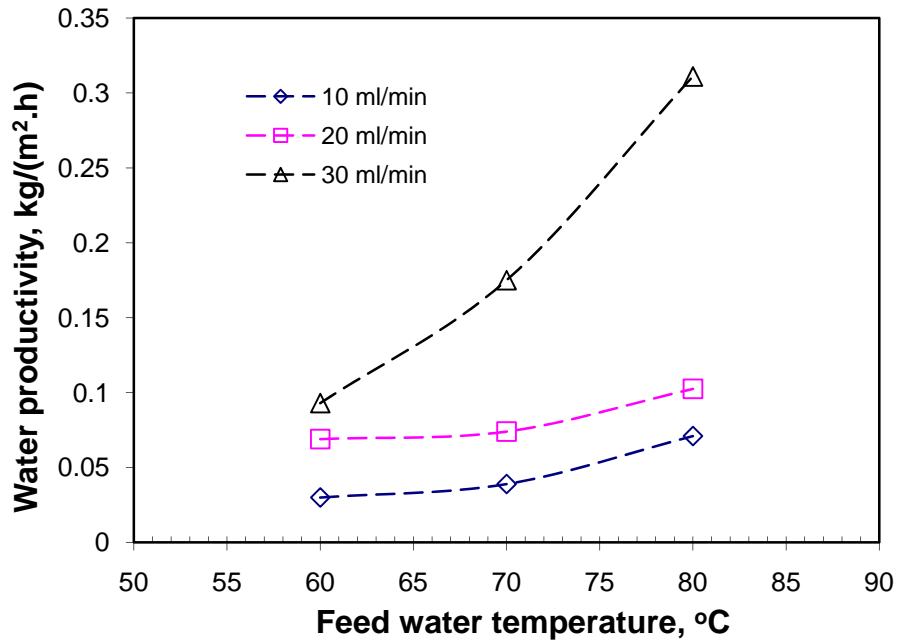


Figure 4-4 Water productivity at varied feed water temperature and flow rate for separator with built-in capillary tubing condenser.

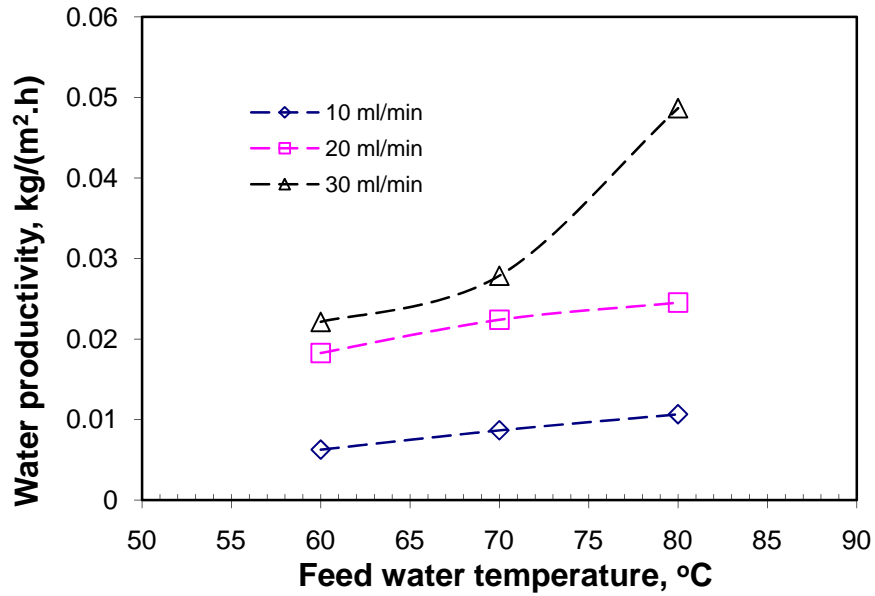


Figure 4-5 Water productivity at varied feed water temperature and flow rate for separator with copper tubing condenser.

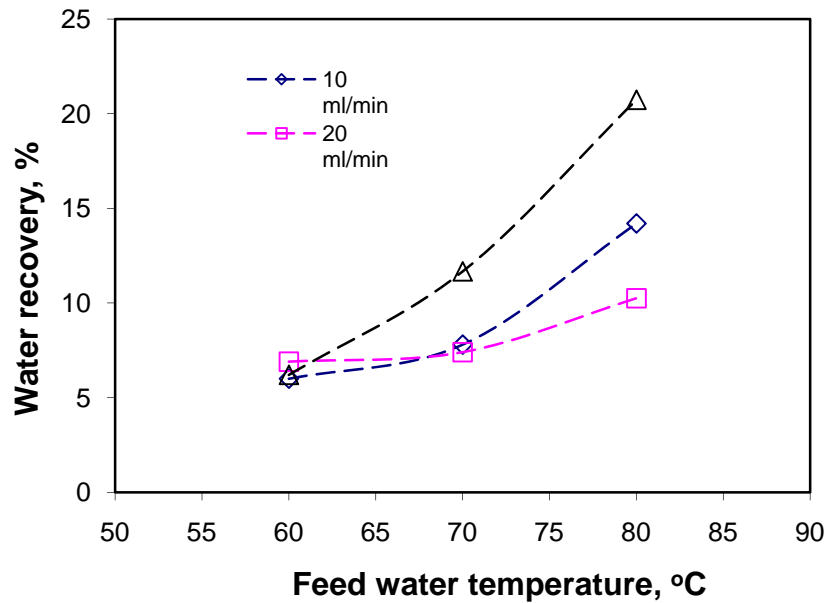


Figure 4-6 Water recovery at varied feed water temperature and flow rate for separator with built-in capillary tubing condenser.

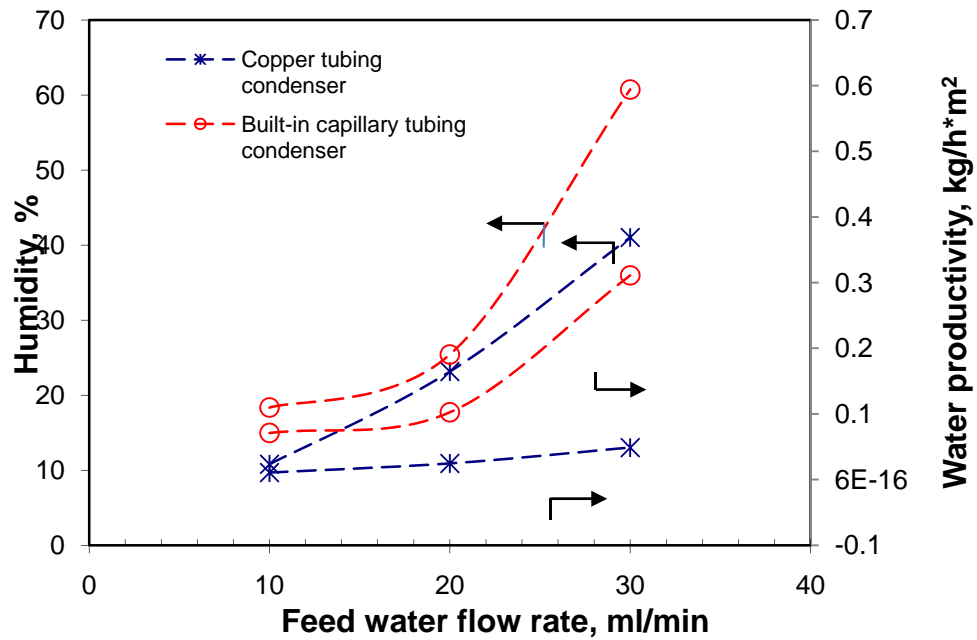


Figure 4-7 Deployment of built-in capillary tubing condenser for enhancement of water productivity.

Figure 4-7 gives the water productivity and humidity at the top of humidifier as a function of feed water flow rate. This test ran at a constant temperature of 80 °C and varied feed water flow rate ranging from 10 to 30 mL/min, carrying air flow rate was set at 1000 LPH. The relative humidity of an air-water mixture is defined as the ratio of the partial pressure of water vapor in the mixture to the saturated vapor pressure of water at a prescribed temperature. Relative humidity is normally expressed as a percentage and is defined in the following manner [Perry and Green, Handbook]:

$$Humidity_R = \frac{P_{water-vapor}}{P_{saturated-vapor}} \times 100 \quad (4-3)$$

where Humidity_R is the relative humidity of the air-water mixture, %; P_{water-vapor} is the partial pressure of water vapor; and P_{saturated-vapor} is the saturated vapor pressure of water at the temperature of the air-water mixture. In this experiment, saturated vapor pressures are 149.4 mmHg, 233.7 mmHg, and 355.1 mmHg at 60 °C, 70 °C, and 80 °C, respectively. Partial pressure is defined as, in a mixture gases, the pressure which one gas would have if it alone occupied the volume of the mixture gases [Henrickson, 2005]. By the definition of partial pressure, the total pressure of a gas mixture is the sum of the partial pressures of each individual gas in the mixture.

Total absolute pressure is 1 atm (atmospheric pressure) which is equal to 680 mmHg. Volume fraction of water vapor is calculated as:

$$Volume\text{-}fraction_{water\text{-}vapor} = \frac{V_{water\text{-}vapor}}{V_{water\text{-}vapor} + V_{carrying\text{-}air}} \quad (4-4)$$

where V_{water-vapor} and V_{carrying-air} are volume of water vapor and dry carrying air inlet in unit time respectively. We assume that all the water vapor at the top of humidifier was condensed and collected as purified water. The water vapor volume can be converted to water productivity.

As shown in Figure 4-6, the water humidity at top of the copper tubing humidifier increased from 10.8% to 41% as the feed flow rate was increased from 10 ml/min to 30 mL/min, resulting in monotonically increase in clean water productivity. The deployment of built-in capillary tubing bundle show considerably enhancement in water evaporation and subsequent water productivity. The water humidity was increased from 18.4% to 60.7%, resulting in higher water productivity upon subsequent cooling from 0.048 to 0.311 kg/(h.m²). Different with copper tubing humidifier, built-in capillary tubing makes 1.2 m² additional heat exchange area which accelerate latent heat transport from humidify side to dehumidify side. As the dehumidify side

cooling down faster and condense area surface increased, purified water was accumulate much more than copper tubing separator in the same time. Also increasing feed water temperature and feed flow rate increased water humidity at top of humidifier, resulting in enhancement in water productivity [Hamieh et al., 2006]. Apparently, the unit energy consumption of produced water purification declines with increase of operating temperature, suggesting the preferred operating conditions at high temperature and high water flow rate.

4.3 Heat efficiency

Humidification-dehumidification process was developed to purify produced water because of the advantage of using the co-produced energy and high energy efficiency. It used elevated temperature produced water to humidify dry air and retrieved the heat from water vapor condensation along the wall of tube. And mass exchange also may increases the heat flux so that heat reuse efficiency will be enhanced. [Holzhauer, 1979; Leidenfrost et al., 1979; Guinn et al., 1981; Papaefthimiou et al., 2006]

Figure 4-8 schematically shows the mass flow of the produced water purification unit. To quantify the energy input during the operating process, dehumidified air at 25 °C was used as carrier gas. The feed water was heated to elevated temperature at 60 °C and 80 °C. The gas and liquid exiting the system include: (1) concentrated wastewater, (2) purified clean water, (3) water vapor, and (4) air. Temperatures at each exiting port were recorded for energy efficiency analysis.



Figure 4-8 Schematic diagram of mass flow in produced water purification by a humidification-dehumidification process.

The water evaporation and condensation occurred interior the separation unit, resulting in a direct reuse of latent heat released by water condensation. The energy of the system was calculated by performing energy balance with the following equation:

$$\begin{aligned}
 & F_{w-feed} \cdot C_w \cdot (T_{w-in} - T_{w-ref}) + G_{air} \cdot C_{air} (T_{air-in} - T_{air-ref}) \\
 & = F_{w-conc} \cdot C_w \cdot (T_{w-conc} - T_{w-ref}) + F_{w-pure} \cdot C_w (T_{w-pure} - T_{w-ref}) \\
 & \quad + (F_{w-feed} - F_{w-conc} - F_{w-pure}) \cdot \lambda_w \cdot (T = T_{air-out}) \\
 & \quad + G_{air} \cdot C_{air} (T_{air-out} - T_{air-ref}) \\
 & \quad + Q_{loss}
 \end{aligned} \tag{4-5}$$

where F is the water flow rate, kg/min. G is the flow rate of carry air, LPH. C_w is specific heat capacity of water, 4.18 kJ/(kg.°C). C_{air} is specific heat capacity of air, 1.006 kJ/(kg.°C). T is temperature, °C. λ is specific heat of vaporization of water, 2260 kJ/kg. Q_{Loss} is defined as heat

loss rate during the operation process, kJ/hr. The T_{w-ref} and $T_{air-ref}$ are the reference temperature of water and air respectively, both at 25 °C in this study.

The heat balance was calculated from equation (4-5) and the results are summarized in Table 4-2.

Table 4-2 Energy balance of produced water desalination by the humidification-dehumidification process with different heat exchanger

No.	F_{w-in} , ml/min	T_{w-in} °C	T_{w-conc} °C	T_{w-pure} °C	Heat balance, KJ/hr					
					E_{in}	ΔH_{w-conc}	ΔH_{w-pure}	ΔH_{air}	$\Delta H_{w-vapor}$	Q_{loss}
Cu-1	10	60	19	22	87.8	-14.34	-0.35	-3.91	54.47	51.92
Cu-2	10	80	20.5	23	137.9	-10.38	-0.40	-2.60	58.16	93.17
Cu-3	20	80	29.5	33	275.9	20.50	3.69	10.42	106.59	134.68
Cu-4	30	80	35	39	413.8	66.09	12.82	18.23	151.63	165.06
Cp-1	10	60	23.8	22.7	87.8	-2.83	-0.35	-3.00	58.87	36.08
Cp-2	10	80	27.1	24.3	137.9	4.52	-0.25	-0.91	63.86	70.72
Cp-3	20	80	30.5	30	275.9	23.38	3.82	6.51	89.09	153.07
Cp-4	30	80	35.5	36	413.8	62.62	17.16	14.33	127.47	192.24

The humidification-dehumidification operation results in the formation of a temperature gradient along the separation unit: the top has the highest temperature while the bottom has the lowest temperature. The temperature gradient varies with the feed water temperature and feed flow rate. As shown in Table 4-2, in circumstances of low water flow rate or low operating temperature, the outlet temperatures are lower than those at the inlets because of the evaporation-provoked cooling effect. Increasing feed water temperature or feed flow rate increases the temperature of the whole system as well as outlet air and liquid. As a consequence, the heat loss caused by water evaporation and radiation will increase. Even though, the energy consumption for purifying unit produced water will decrease, resulting in enhanced energy efficiency and

water recovery as shown in Figure 4-6. Also energy lost of the separator unit with built-in capillary tube is much less than copper tube separator, because the larger heat exchange area can accelerate heat transfer to achieve reduced energy loss.

CHAPTER 5 PRODUCED WATER DESALINATION

EXPERIMENTS

The coalbed methane produced water sample was obtained from local water disposal facility at Farmington in New Mexico. The original produced water contained large number of suspended particulates, floating oil droplets and dissolved components. A coarse filtration with 100 mm filter paper was first carried out. The purpose of the coarse filtration was to remove the large particulates and oil droplets which can potentially block the microbore water distributor. Figure 5-1 shows the picture of produced water filtration cake. The orange and dark yellow color on the white filter paper is suspended particles accumulated [Lu, 2008]. The filtered produced water still has high concentration of particulate suspension which present as light yellow color. A dynamic light scattering particle analyzer was deployed for study of particle size distribution of the produced water. Figure 5-2 gives the particle size distribution of the filtrated produced water.

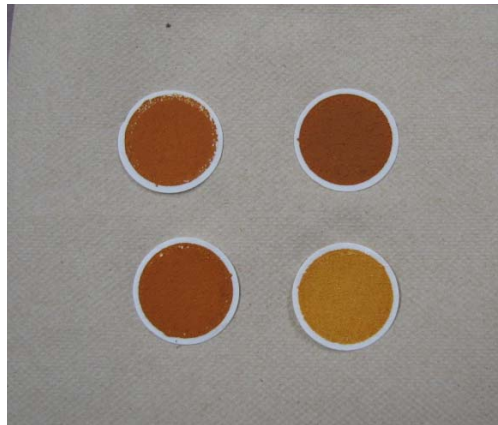


Figure 5-1 Filtration cake of the produced water.

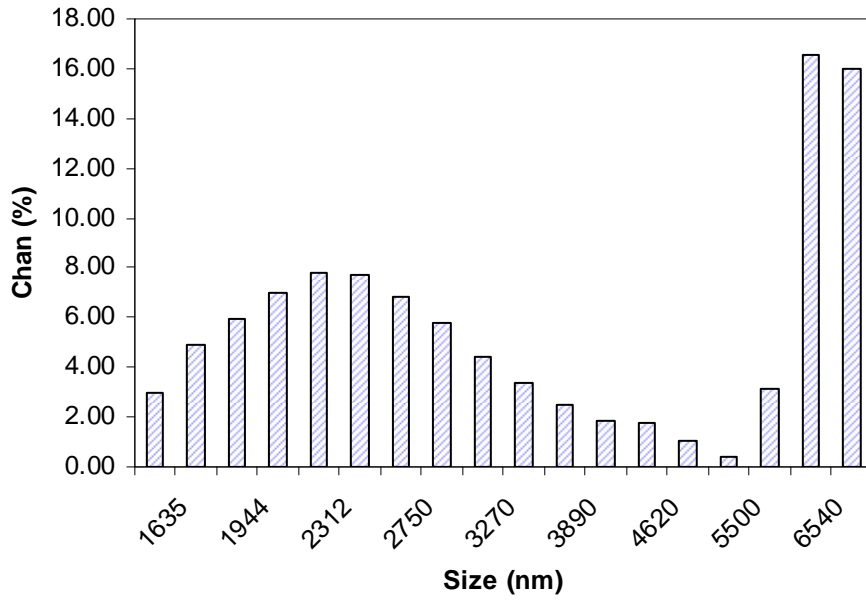


Figure 5-2 Particle size distribution of suspensions in produced water.

The produced water had a total dissolved solid (TDS) of 1.98×10^4 mg/L and total organic carbon (TOC) of 470.2 mg/L. Table 5-1 gives the chemical composition of major dissolved components in the produced water and the total suspended particles.

**Table 5-1 Chemical composition of CBM produced water in Farmington,
NM**

Ion species	Concentration, mg/L	Trace metal ions	Concentration, mg/L
Sodium (Na ⁺)	6765.7	Barium (Ba)	10.9
Ammonium (NH ₄ ⁺)	350.0	Copper (Cu)	0.21
Potassium (K ⁺)	427.9	Iron (Fe)	0.27
Magnesium (Mg ²⁺)	32.7	Lithium (Li)	4.7
Calcium (Ca ²⁺)	46.1	Silica (SiO ₂)	11.7
Chloride (Cl ⁻)	10542.9	Strontium (Sr)	8.9
Sulfate (SO ₄ ²⁻)	1590.7	Nickel (Ni)	0.16
Total dissolved solid (TDS), mg/L		19792.8	
Total suspended particulates, mg/L (0.22µm < dia.< 100µm)		99.6	
Total organic carbon (TOC), mg/L		470.2	

5.1 Ion removal efficiency for produced water

Produced water desalinations by humidification-dehumidification process were carried out at an air flow rate of 1000 LPH and water flow rate of 40 mL/min. The water temperatures were varied from 60 °C to 80 °C. The purified water samples were collected at a time interval of 3 hours. Each separation was running for over 12 hours until three consecutive water samples shown identical water productivity. Figure 5-3 gives the individual ion removal efficiency of the produced water as a function of feed water temperature. The ion removal efficiency of NH₄⁺

declined from 97.4% to 95.4% as water temperature increased from 60 °C to 80 °C. In aqueous solution, ammonium ions exist at equilibrium with ammonia ($\text{NH}_3 + \text{H}_2\text{O} \leftrightarrow \text{NH}_4^+ + \text{OH}^-$). Increasing temperature drives the reaction to the left side due to the exothermic reaction. As a result, more ammonia will evaporate with air and condensate to the clean water upon subsequent cooling. All other ions show >98% removal efficiency, resulting in a decline in total dissolved solid from 1.98×10^4 mg/L to 92.1 mg/L at 60 °C.

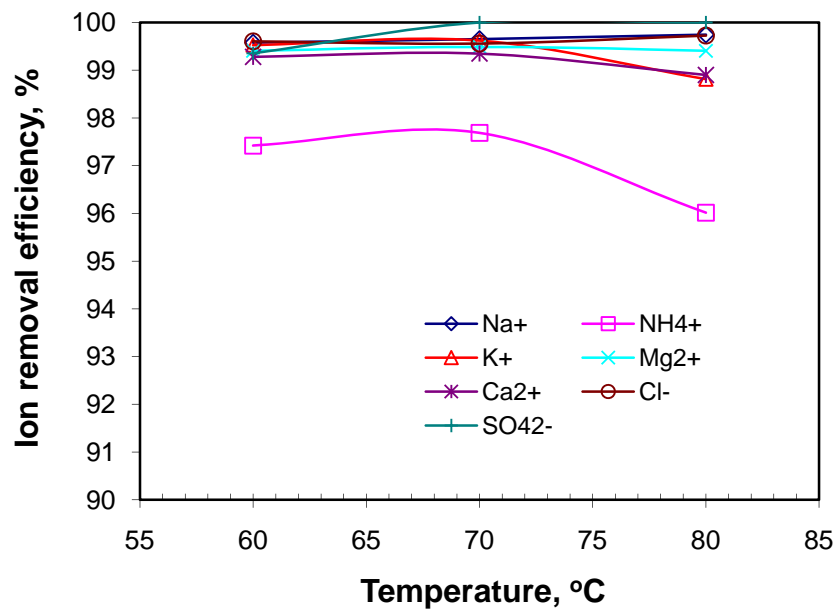


Figure 5-3 Ion removal efficiency of individual ions in produced water at temperatures of 60 °C, 70 °C and 80 °C, respectively.

Presence of ions in the purified produced water is explained by the mist formation during air-enhanced water evaporation process. Small water droplets will be carried with by the counter-flowing air stream and precipitate in the purified water upon cooling [Hamieh et al., 2006].

Increasing the feed water temperature increases the water partial pressure at top of humidifier while the mist formation is independent on water humidification. As a result, ion concentration declines slightly with increase in operating temperature, as shown in Figure 5-4.

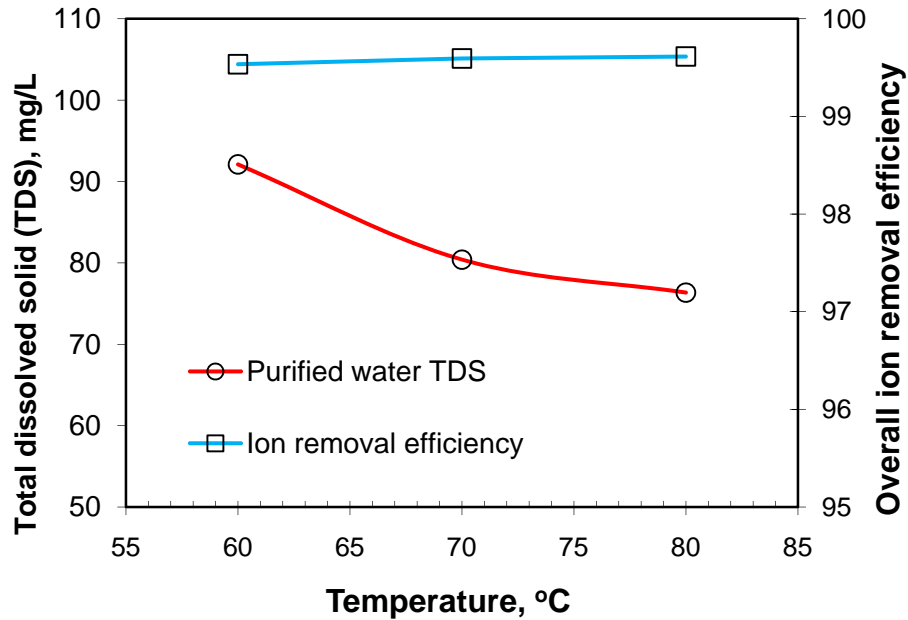


Figure 5-4 Total dissolved solid in purified water and overall ion removal efficiency at different operating temperatures.

5.2 Organic removal efficiency for produced water

Presence of organics in produced water was quantified by concentration of total organic carbon. The produced water sample treated by a coarse filtration had a TOC of 470.2 mg/L. Over 95% of organic carbon was removed by the humidification-dehumidification process with a decline of TOC from 470.2 mg/L to 21.5 and to 17.8 mg/L at 60 °C and 80 °C, respectively.

Figure 5-5 gives the TOC of purified water and TOC removal efficiency at different operating temperatures.

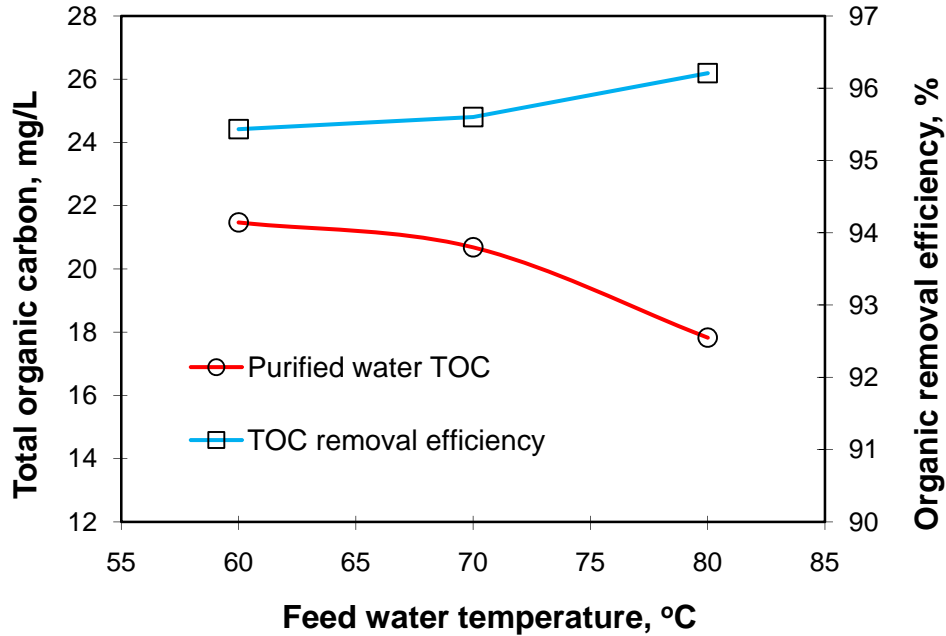


Figure 5-5 Total organic carbon of purified waters and organic removal efficiency as a function of feed water temperature.

The organic components in produced water typically contain fatty acid (~300 mg/L), polycyclic aromatic hydrocarbons (PAHs, ~5 mg/L), phenols (~10 mg/L), and volatiles such as benzene, toluene and xylene (BTX, ~8 mg/L) [Hansen et al., 1994; Santos et al., 1997]. The organic composition varies over the lifetime of oil/gas production. Other factors such as temperature, production history, and type of produced water also show influences on organic composition in produced water. Generally, produced waters from gas field or CBM production have a higher concentration of volatile hydrocarbons than those in oilfield [Veil et al., 2004].

During the humidification process, organics will evaporate accompanying the water evaporation, forming an organic-containing humidified air. Generally, the organic concentration in humidified air is far below the saturation, particularly for the volatile organics. The subsequent cooling for water condensation forms purified water while the organic vapor will exit to the atmosphere. All experiments show high organic removal efficiencies (>95%) with slightly variation at different operating conditions. For example, the organic removal efficiency increased from 95.4% to 96.2% as feed water temperature was increased from 60 °C to 80 °C. The influence of feed water temperature on organic removal efficiency has the same trend as that on inorganic salts, which can be explained by the small water droplet formation at presence of strong air flow.

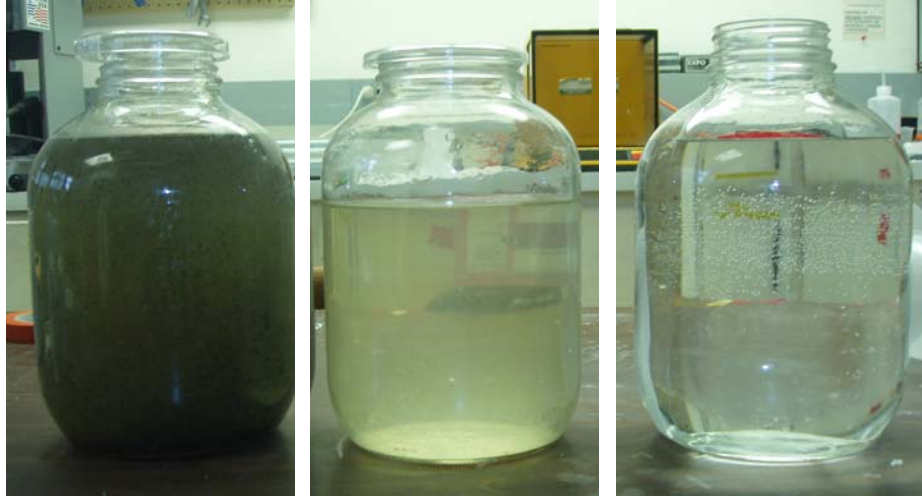
5.3 Water quality and beneficial use

Produced water contains large varieties of inorganic salts, heavy metals and organic contaminants. Direct disposal of produced water poses the potential for ground water contamination and thus may not be discharged to ground water system unless they are being purified to substantial quality [Santos et al., 1997]. Quantity and quality of purified produced water are the most important factors governing the beneficial uses when produced water is substituted for conventional water resource. Generally, beneficial use of produced water, such as irrigation, livestock watering, and power cooling, must satisfy all special considerations necessary for water quality requirement. Table 5-2 summarizes the general requirement of reclaimed water quality for the industry or agriculture applications.

Table 5-2 Beneficial use of reclaimed produced water and quality requirements

Beneficial use	Water quality requirements					Main concerns
	TDS, mg/L	Hardness or trace elements	pH	Cl ⁻	Alkalinity	
Irrigation (Rowe et al., 1995)	500-2000	Boron <0.75 Cu <0.2 Fe<5.0 Ni<0.2 Zn<2.0	6	NA	NA	Salinity, trace elements, chlorine residual, and nutrient
Cooling water (EPA, 2004)	<2700 (conductivity)	Hardness<450	6.5-7.5	350	280	Corrosion, biological growth, and scalling
Chemical process (EPA 2004)	1000	Hardness<250 Fe<0.1 Mn<0.1	6.2-8.3	500	125	Low turbidity, suspended solids and silica

Figure 5-6 shows the picture of produced water sample as received, filtrated, and purified by the humidification-dehumidification process. Large number of oil droplets and particulates were observed on the produced water as received. A course filtration with 100 μm Whatman □ filter removed the oil droplets and large particulates, but left all dissolved components and fine particles with detail composition shown in Table 5-1. The purification by the humidification-dehumidification removed majority of salts and organics. The detail chemical composition of the purified water is shown in Table 5-3.



(a)

(b)

(c)

Figure 5-6 Produced water and purified water samples. (a) Produced water as received, (b) Produced water with large particle and oil droplet removal (particle size $>100 \mu\text{m}$), (c) Purified produced water.

Table 5-3 Chemical composition of purified produced-water

Composition	Concentration, mg/L
Sodium (Na ⁺)	17.23
Ammonium (NH ₄ ⁺)	13.95
Potassium (K ⁺)	5.08
Magnesium (Mg ²⁺)	0.19
Calcium (Ca ²⁺)	0.51
Chloride (Cl ⁻)	29.07
Sulfate (SO ₄ ²⁻)	10.3
Total dissolved solid (TDS), mg/L	76.35
Total organic carbon (TOC), mg/L	17.83

The purified produced water has the TDS of 76.35 mg/L and TOC of 17.83 mg/L. All metal ions in the purified water are below the detection limit. Compare with Table 5-2 and Table 5-3, the purified produced water show much higher quality than the criteria for general irrigation, tower cooling and chemical processing.

CHAPTER 6 CONCLUSIONS AND FUTURE WORK

6.1 Conclusions

Humidification-dehumidification is an efficient desalination process for produced water purification. Low temperature operation and mobile device make it suitable for most oil field that locates at remote region. Otherwise, solar energy and co-produced geothermal energy from reservoir can be used in this process which makes it environment friendly and economic.

The humidification-dehumidification process for produced water desalination was studied for removal of dissolved components from produced water, and influence of operation parameter on system performing efficiency. Humidification-dehumidification separator was re-designed to improve the performance of this process. The outcomes of the studies of total dissolved solid and organic removal efficiency; water productivity and heat efficiency are summarized below.

A bench scale experiment with coalbed methane produced water indicated that over 99.5% of dissolved salt can be removed with the total dissolved solid decline from 1.98×10^4 mg/L to 76.3 mg/L. The humidification-dehumidification process also showed remarkable organic removal efficiency by reducing the total organic carbon from 470.2 mg/L to 17.8 mg/L. The water productivity is insensitive to the feed water quality and chemical composition and thus is particularly useful for purification of concentrated- or particulate-enriched produced waters.

A built-in capillary tubing bundle was deployed as condenser for enhancement in water productivity and heat efficiency. The water productivity was increased from 48 to 311 ml/(hr.m²). The influence factors, such as feed water temperature and feed water/air ratio, on the

water productivity were also investigated. Increasing feed water temperature or feed flow rate increases the heat loss, but the water productivity and recovery increase as a result of more efficiency heat use.

The purified produced water has a higher quality than most of requirement of agriculture and industry uses. The humidification-dehumidification shows promise for reclaiming produced water for beneficial uses such as irrigation, tower cooling and chemical processing.

6.2 Recommended future work

In this research, we have experimentally demonstrated the desalination capacity of humidification-dehumidification process on produced water. Water recovery and energy efficiency have been significantly improved by using built-in hollow tube condensers. The next step will be further investigate and understand the influence of operation parameter on water productivity and heat efficiency, and desalination system usage in the oil field.

(1) Upscale the separation unit for field application of produced water desalination by using low-temperature energies.

(2) Investigation of the influences of operation parameters on the purified water productivity. In order to find the best operation condition for different size desalination unit, the influences on desalination capacity need to be tested by enlarging the range of numerical value of each operation parameter,

(3) Development of numerical model to simulate the mass and heat transfer and optimize an engineering design of the separation unit.

(4) Investigation of deployment of solar energy and co-produced energy sources.

(5) Conducting of economic analysis for projection of equipment costs, operating costs and total water costs. Also, the water treatment cost should be a function of plant size, production rate, as well as waste or solar heat deployment.

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