

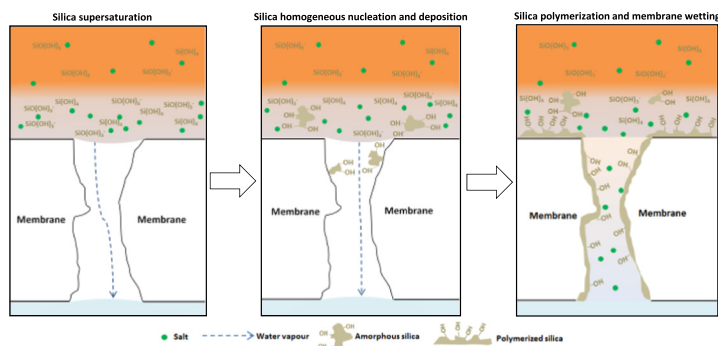
Silica fouling during direct contact membrane distillation of coal seam gas brine with high sodium bicarbonate and low hardness

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GRAPHICAL ABSTRACT



ABSTRACT

The presence of silica in coal seam gas (CSG) reverse osmosis (RO) brine complicates the membrane distillation (MD) process due to silica polymerization and metal silicate formation at high water recovery. This study reported that direct contact MD (DCMD) of CSG RO brine at electrical conductivity (EC) of 17.8 mS/cm achieved 90% brine recovery (overall recovery of 95%) with distillate flux decline of 16%, and distillate EC of 15 μ S/cm over 150 h of operation. A series of experiments were conducted using synthetic solutions to evaluate the effect of CSG RO brine water quality on silica fouling in DCMD process. Results showed that CaCO_3 formation was fast and did not co-precipitate with dissolved silica, while magnesium and silica were co-precipitated as magnesium silicate compounds. Results also revealed that polymerized silica formed a non-porous layer on the membrane surface. This fouling layer not only significantly reduced distillate flux but also led to a dramatic increase of distillate EC. Further study on silica fouling mitigation suggested that rapid cooling and filtration/clarification could be adopted for removal of silica from MD brines containing high silica concentration. It also showed that disodium ethylenediaminetetraacetic acid (Na_2EDTA) was effective to hinder the formation of CaCO_3 and magnesium silicate in MD.

1. Introduction

As conventional gas resources decline, unconventional sources of gas, like coal seam gas (CSG) or coal bed methane (CBM), is in increasing demand in many countries [1]. Australia has significant CSG resources, particularly in the states of New South Wales and Queensland. Continued CSG production growth in Australia is expected until

2030 [2]. CSG produced water is considered as a by-product in CSG extraction process. It is estimated that up to 7500 GL of water will be produced from Australian CSG industry in the next 25 years [1]. CSG produced water quality varies from site to site and generally has potential to cause surface water, underground water, and ecosystem damage [1]. Therefore, the management and disposal of CSG produced water is seen as the greatest challenge of the growing CSG industry in

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Australia [3].

High pressure membrane desalination technologies such as nanofiltration (NF) and reverse osmosis (RO) have been adopted to separate impurities from CSG produced water before treated water can be conveniently re-used, for applications such as irrigation, dust suppression, reinjection into depleted underground aquifers, etc. [2]. A significant problem concerning the application of NF/RO is the waste concentrate (brine) disposal, particularly at inland CSG sites where the brine is traditionally disposed through evaporation ponds. The cost of constructing and managing evaporation ponds can be quite substantial, leading to the need for a technology that can further reduce the volume of waste brine at inland desalination plants.

In the last decade, membrane distillation (MD) has emerged as a possible option for CSG brine management. MD is a thermal membrane separation process that involves transport of vapour through microporous hydrophobic membranes and operates on the principle of vapour-liquid equilibrium as a basis for molecular separation [4]. MD can be operated in four basic configurations, including direct contact MD (DCMD), sweeping gas MD (SGMD), air gap MD (AGMD) and vacuum MD (VMD) [5]. MD has several advantages for brine minimisation, such as low operating pressure, moderate temperatures, potential for 100% rejection of non-volatile solutes and small footprint [6, 7]. In addition, solar energy and waste heat (e.g., from gas compressors) could be utilised as the thermal energy source for MD processes at inland CSG sites, and this makes MD potentially more attractive than other brine minimisation technologies. In the case of valuable salt (e.g., NaOH) recovery from CSG RO brine, MD will provide a more concentrated brine stream for subsequent processes [8, 9].

However, MD fouling and wetting are the major operational issues for concentrating hyper-saline RO brine [10]. MD fouling is the accumulation of unwanted materials on the membrane surface or inside the pores, and this could pose a very detrimental effect on MD long-term stable operation. MD fouling can alter the hydrophobicity of membrane surface, leading to the intrusion of brine into membrane pores and, thus, deteriorating distillate quality. This phenomenon is called membrane wetting. To date, only limited studies have been conducted on MD fouling in CSG RO brine treatment. Duong et al. [11] performed a pilot study on concentrating CSG RO brine using Aquastill's (Sittard, The Netherlands) spiral-wound AGMD system. This study demonstrated stable operation of MD system at 80% of RO brine recovery (overall recovery of 95%), and salt rejection of 99.5%. However, concerns were raised that membrane fouling due to silica and calcium carbonate may restrict efficiency of MD long-term operation. Dissolved silica may not be very high in CSG produced water, but can easily exceed its solubility in a RO-MD hybrid system operates at high water recovery. Duong et al. [11] did not further explore the kinetics of silica or metal silicate formation in MD process. Zhong et al. [12] evaluated silica fouling with synthetic CSG produced water using a submerged VMD system equipped with capillary hydrophobic polypropylene (PP) membranes. The synthetic solutions employed in Zhong's study were silica rich (200 mg/L) with high Mg^{2+} (62.5–200 mg/L) and Ca^{2+} (40–300 mg/L), but low $NaHCO_3$ (1.2 g/L). The submerged VMD was operated at 70 °C under constant total dissolved solids (TDS) concentration at 37.2 g/L and pressure of –95 kPa. Membrane wetting was observed in Zhong's study. They also reported that calcite and magnesium silicates were the major foulants on the membrane, and membrane cleaning with a diluted hydrochloric acid solution of 0.32 wt% at 24 h intervals only restored distillate flux to 80% of its original value after 72 h of operation. Compared with other MD configurations, VMD is considered more sensitive to membrane wetting due to the hydrostatic pressure drop across the membrane which acts as a driving force for liquid into the membrane pores.

Dissolved silica in water exists as orthosilicic acid ($Si(OH)_4$) and silicate anions ($H_2SiO_3^{2-}$, $H_2SiO_3^-$) [13]. The optimum amorphous silica solubility is $pH > 9.5$ or < 6.5 since dissolved silica exists in solution predominantly in either neutral or anionic species [14]. When

the concentration of dissolved silica exceeds its solubility (at a given temperature and pH), silica polymerization (heterogeneous nucleation) will occur, and form colloidal amorphous silica. The colloidal amorphous silica deposited on membrane surface will further dehydrate and finally become glasslike scale, and it is very difficult to remove by conventional chemical cleaning approaches [15]. Temperature is an important factor in MD process, and it can influence silica solubility. Silica solubility in solution increases with increasing temperature, and vice versa [2]. MD temperature and concentration polarizations could result in lower temperature and higher dissolved silica concentration near the membrane surface [16], and potentially promote silica supersaturation and cause precipitation of colloidal amorphous silica. Studies have shown that at neutral pH, presence of multivalent cations (e.g., Ca^{2+} , Mg^{2+}) facilitate silica polymerization and this was possibly due to dissolved silica surface charge neutralisation and compression of double charged layer induced by multivalent cations [5, 17]. Study has suggested that dissolved silica has stronger bond with Mg^{2+} than with Ca^{2+} [12]. Moreover, CSG produced water's alkaline environment favours reaction between dissolved silica and Ca^{2+}/Mg^{2+} to form metal silicates which have significantly less solubility than amorphous silica, and this would increase the likelihood of particulates (e.g., Mg_2SiO_4 , Ca_2SiO_4) depositing on the membrane surface [14]. Researches have showed contrasting observations in the effect of anions (e.g., CO_3^- , Cl^-) on silica solubility. Koo et al. [18] concluded that CO_3^- was more significant in facilitating silica polymerization as compared to Cl^- , while Marshall et al. [19] reported that use of carbonate salts increased amorphous silica solubility as compared to chloride salts. Therefore, there is still a need for understanding silica fouling kinetics in MD process before this technology can be commercially realised in CSG RO brine treatment.

In this study, DCMD performance and membrane fouling were assessed over a relative long period of operation for concentrating CSG RO brine. The influence of divalent cations (Mg^{2+} and Ca^{2+}), anions (HCO_3^-/CO_3^{2-} and Cl^-) and temperature on silica fouling were examined individually. Moreover, the efficiencies of MD silica fouling control using commercial RO antiscalants and disodium ethylenediaminetetraacetic acid (Na_2EDTA) were assessed.

2. Experimental

2.1. DCMD set-up and membrane material

The experiment set-up is shown in Fig. 1. Feed water (brine) was withdrawn from the feed container (6) and transferred to the DCMD module (3) through a heating unit (2) followed by a cartridge filter (4). The feed water was then recirculated to feed water container via the DCMD module (3). Distillate from the membrane was collected in the distillate container (5) and then withdrawn from the container and cycled back to the DCMD module through a cooler (7). Thermal insulation foam was applied on both containers (including lids) to minimize heat loss and water evaporation. Feed water container was under 400 rpm magnetic stirring to provide agitation during experiments to prevent precipitated solids from settling at the bottom of container. The solution EC and pH were continuously monitored at feed and distillate container, respectively. The inlet and outlet temperatures of the DCMD module were measured by thermocouples, and outputs were recorded in a computer via a multichannel datalogger. Both feed and distillate flows were operated in countercurrent mode within the DCMD module at a fixed flowrate of 1000 ml/min. Feed and distillate inlet temperatures were controlled at 50 and 25 °C, respectively.

According to the supplier's membrane specification, polytetrafluoroethylene (PTFE) flat sheet membrane (Changqi Co. Ltd., China) used in this study has effective area of 0.0169 m² (130 mm × 130 mm), nominal pore size of 0.3 μm, thickness of 0.12 mm and air permeability of $1.4 \times 10^{-3} \text{ mol m}^{-2} \text{ Pa}^{-1} \text{ s}^{-1}$. To enhance heat and mass transfer, Dow Filmtec™ SW30–4040 RO membrane feed mesh spacers were

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