International Journal of Environmental Sciences
ISSN: 2229-7359
Vol. 11 No. 17s, 2025
https://theaspd.com/index.php

A Sustainable Process For Brine Management And CO_2 Sequestration: An Experimental Study

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Abstract— *A sustainable process scheme for extracting valuable minerals from desalination reject brine while simultaneously sequestering* CO_2 *has been evaluated. The first part of the proposed multistage scheme involves the removal of calcium ions through reaction with* CO_2 *in the presence of NaOH in an inert particle spouted bed reactor (iPSBR). At NaOH dose of 11 g/L and gas flowrate of 1 L/min, 96% of the calcium ions and 16% of the magnesium ions were removed with a* CO_2 *uptake of around 10 g/L under ambient conditions. The solid products obtained from this step were filtered and analyzed confirming the formation of high-purity calcium carbonate,* $CaCO_3$*. Next, the filtrate from this reaction was reacted with NaOH to remove the magnesium ions. To accomplish this step, the magnesium ions present in the obtained calcium-free brine was precipitated through further addition of 8.22 grams of NaOH at ambient temperature. About 83% of the magnesium ions were removed in this step and the analysis of the solid products affirmed the formation of magnesium hydroxide,* $Mg(OH)_2$*. Both solid products obtained from the individual steps are commercially valuable. Moreover, the obtained* Ca^{2+}*,*Mg^{2+}*-free brine effluent can be eventually reused as a feed for an electrolysis unit. The addition of an electrolysis unit will significantly improve the scheme's economic feasibility as it will lead to the production of NaOH required for the ion removal steps. Thus, creating a self-sustaining process for the sustainable management of reject brine and* CO_2 *sequestration*

Keywords— Ca^{2+} *Removal,* CO_2 *Sequestration,* CO_2 *Uptake,* Mg^{2+} *Removal, NaOH, Reject Brine.*

I. INTRODUCTION

Seawater desalination has emerged as one of most sustainable alternative means of water supply that can effectively meet the global water demand [1, 2]. In a typical desalination process, seawater is used as feed to produce fresh water that can be used for both industrial and municipal applications. This technology is readily being used as the primary source of water in arid and semi-arid regions and is also being adopted by several countries as a secondary source of fresh water. This clearly shows the significance of the desalination technology for promoting sustainability within the modern world through ensuring the continuous supply of water [3]. Nonetheless, there are several environmental drawbacks of the desalination technology. One of which is the generation of high-salinity by-product known otherwise as Reject brine, which is continuously discharged from the desalination facility and thrown back into the adjacent water bodies leading to its contamination [4]. Moreover, the desalination process is energy-intensive and is commonly powered by fossil fuels which are notorious for their carbon dioxide emissions, thus, desalination technology inadvertently contributes as a CO_2-emitting source contributing to global warming [5, 6]. Studies have reported that there are currently more than 15 thousand desalination plants that are operating worldwide and together they emit about 76 million tons of carbon dioxide per year [7, 8]. Therefore, in order to tackle the adversities of the desalination process, the development of innovative and sustainable brine management strategy that can address the complications arise from the disposal of reject brine and its passive carbon dioxide emissions is paramount to protect the environment while ensuring sustainable supply of water. CO_2 mineralization using desalination reject brine has emerged as one of the technologies that can manage both desalination reject brine and carbon dioxide emissions in recent years [1, 9]. The Ca^{2+} and Mg^{2+} ions in reject brine can be used readily for carbon dioxide mineralization without requiring any pretreatment. The Solvay process is one of the leading processes that has already exhibited excellent potential for capturing carbon dioxide while decreasing the salinity of brine, converting them into economically viable products. In a typical Solvay process, NH3 is applied as a promotor to increase the pH of the feed which accelerates the reaction between carbon dioxide and sodium chloride producing ammonium chloride with precipitates of ammonium bicarbonate. However, NH3 has several environmental and health hazards which reduces the environmental feasibility of the

Solvay process. Additionally, compared to the other conventional CO_2 capture processes, the Solvay process exhibits lower carbon dioxide sequestration capability which needs to be substantially improved to increase the overall feasibility of the process. This led to a growing interest among the researchers to discover alternative promoters that are environmentally benign yet can substantially increase the overall feasibility of the Solvay process. Calcium oxide was studied as a potential promotor in a modified Solvay process in a study carried out by El-Naas et al. which showed a significant improvement in carbon dioxide capture capacity [10]. This approach was replicated by Mourad et al. in order to investigate the effect of using potassium hydroxide as a promotor. The results obtained in their study showed an increase in the CO_2 sequestration capacity and it also showed that the application of KOH enhances the removal of sodium, calcium and magnesium ions [10-12]. Alternatively, a number of research studies also explored the possibility of using amines as an alkali in a Solvay-like process which resulted in the production of high-quality carbonates [13]. The studies presented in the literature clearly indicate that both the CO_2 and the reject brine generated from the desalination facilities can be potentially remedied through the mineralization of CO_2 mineralization using reject brine while producing carbonates and other valuable products. However, none of the studies could successfully achieve all three targets of the process, i.e. maximize CO_2 capture, reduce brine salinity and obtain high quality marketable products, without compromising the other two. Compared to ammonia, NaOH is an alternative promoter that can be used in modified Solvay process with lower environmental implications. In this work, multistage process facilitating the treatment of reject brine and CO_2 has been studied to explore the possibility of generating highly pure $CaCO_3$ and $Mg(OH)_2$. The process will also generate low-salinity feed that can be further utilized as a feed for electrolysis (Fig.1). The successful implementation of the proposed scheme will present a new avenue for CO_2 sequestration and reducing reject brine salinity, thus, promoting sustainable management of these two major wastes within the GCC countries, and the world at large.

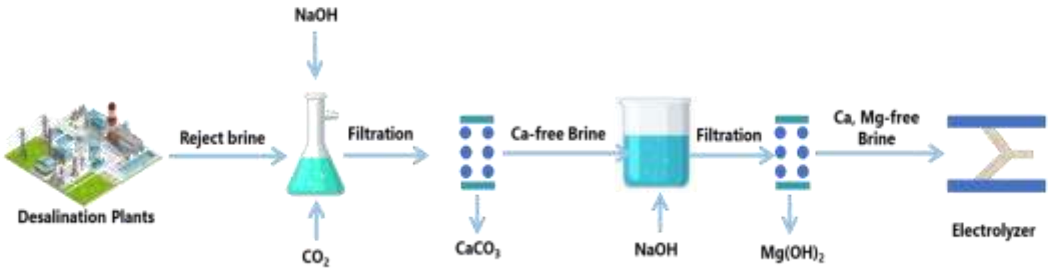

Fig. 1. An illustration of the scheme for the reject brine and CO_2 management.

II. EXPERIMENTAL MATERIALS

A. Materials

NaOH pellets (99% purity) were procured from Scharlau, UK. National Industrial Gas Plants (NIGP), Doha, Qatar supplied gas cylinders containing mixtures of 10% CO_2 balanced with nitrogen. The reject brine samples study were collected from MSF-based local desalination facility in Qatar. The pH and conductivity were measured using a Thermo Scientific Orion Star A325 meter, while major ion concentrations were determined using a Shimadzu 9800 ICP-OES. Table 1 shows the characterization of the brine samples.

TABLE I CHARACTERISTICS OF THE BRINE SAMPLE

Parameters	Units	Value
Sodium	mg/L	30350
Magnesium	mg/L	3040
Potassium	mg/L	1435
Calcium	mg/L	1005
TDS	mg/L	73700
Conductivity	mS/cm	90.81
pH	-	8.24

B. Description of the iPSBR and experimental procedure

Calcium removal experiments were conducted using a patented inert particle spouted bed reactor (iPSBR), which has been previously utilized in studies involving modified Solvay-based processes [1, 11, 12, 14]. The reactor consisted of a vertical stainless-steel vessel equipped with ports for gas and liquid inlet and outlet streams. A mass flow controller was used to introduce a mixture of gas at the bottom of the reactor. The vessel has a total volume of approximately three liters, of which one liter was used for performing experimental study. When the CO2-containing gas was introduced through a single spout at the base, it propelled the particles to move in an elliptical motion, increasing gas-liquid contact and promoting efficient mass transfer. The gas exiting from the top of the reactor passes through a moisture trap before entering CO_2 analyzer, which recorded CO_2 concentration (vol%) every second. A schematic diagram of the reactor setup is shown in Figure 2.

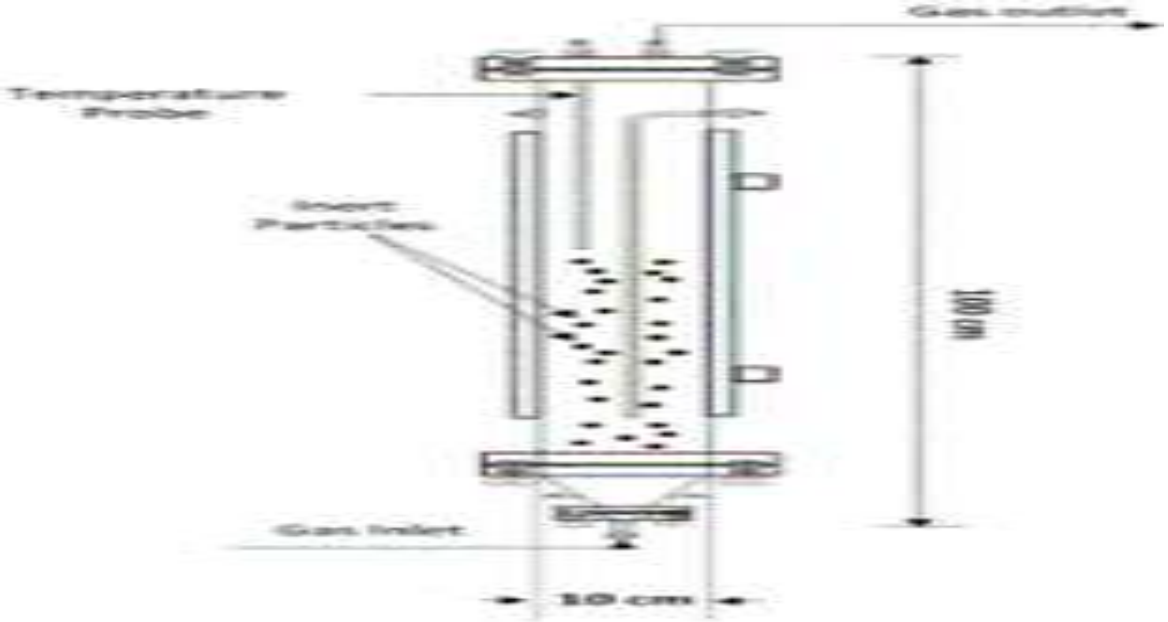

Fig. 2. A Schematic Diagram of the Patented iPSBR for Ca^{2+} Removal

It is important to note that the experiments were conducted at room temperature under atmospheric pressure conditions using untreated reject brine samples. The CO_2 uptake of the brine solutions was calculated from the CO_2 analyzer readings, plotted over time by measuring area under the curve using equation 1, which was then used to calculate the volume of CO2.captured [14].

$$V_{\mathrm{Uncaptured}} = \mathrm{Flowrate} \text{ x } \int_0^{t_F} \frac{CO_2\%}{100}\, dt \qquad (1)$$

An ICP-OES was used to measure the calcium and magnesium ion concentrations in the samples, and the resulting data were used to calculate the amount of target ions removed using the following equation 2.

$$\text{Ion Removal } \% = \frac{C_i - C_o}{C_o} \qquad (2)$$

The terms 'C_i' and 'C_o' in equation 2, represents the initial and final concentrations (mg/L), respectively. Following the reactions, about 100 mL of the filtrate was transferred into an Erlenmeyer flask to around 8.22 grams sodium hydroxide added and placed in a shaker and mixed thoroughly at 200 RPM for two hours. After mixing, the samples were filtered and analysed using ICP-OES. The amount of magnesium removed was calculated using equation 5. Solid products were filtered, and Thermogravimetric analysis of the samples was conducted in a N_2 environment by heating the samples up to 1000 oC. The weight loss was plotted against temperature to identify possible products and their purity

III. RESULTS & DISCUSSIONS

A. Experimental Results

Using the iPSBR, experiments were initially conducted at a CO_2 gas flow rate of 1 L/min, ambient temperature, and a NaOH dose of 11 g/L. The CO_2 uptake was recorded, and a plot of CO_2 uptake versus time was generated (see Fig. 3). The plot reveals two distinct zones, indicating the reactions occurring within the reactor system. A rapid increase in CO_2 uptake is observed in the 1st zone which indicates the formation of calcium carbonate. Following this, a steady increase in CO_2 uptake is observed, attributed to the formation of sodium bicarbonate, with a continued decrease in pH. Beyond this period, no further change in CO_2 uptake occurs, indicating the reactions have concluded and the pH approaches neutrality. Overall, in terms of the alkaline mass added, this CO_2 uptake corresponds to 0.90 g CO_2/g NaOH, comparable to the uptake reported in recent studies using Calcium Oxide (0.92 g CO_2/g CaO) and Potassium Hydroxide (0.31 g CO_2/g KOH) at room temperature [11, 12]. ICP analysis of the liquid products showed a 96% removal of Ca2+ ions and a 16% removal of Mg2+ ions. TGA of the solid products confirmed the formation of $CaCO_3$ (See Fig. 4).

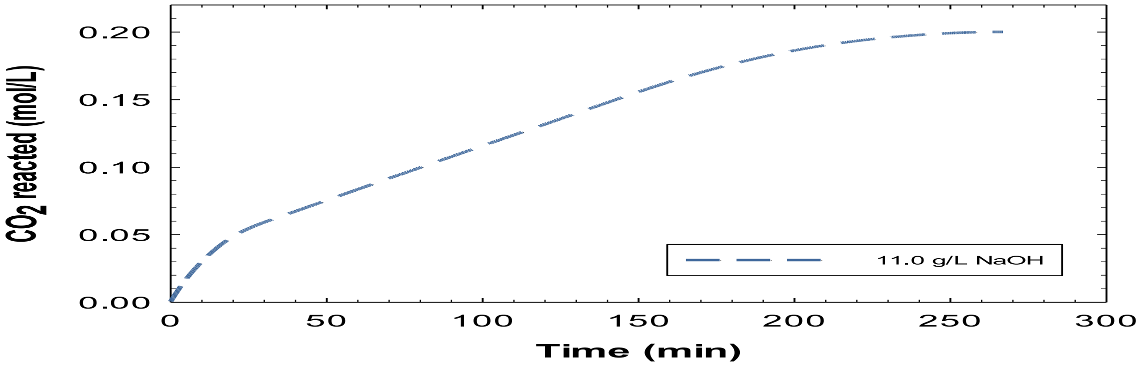

Fig. 3. Mol of CO_2 reacted vs Time curve obtained from the experimental run.

Based on a previous optimization study conducted on the removal of magnesium ions, around 8.22 grams of NaOH was added to the obtained Ca-free brine to induce the removal of Magnesium for the formation of magnesium hydroxide. This resulted in 83% removal of magnesium ions. This was slightly lower than the complete removal ions reported in the previous study [15]. This could be attributed to the fact that in the previous study, 8.22 grams of NaOH were directly added to the brine without any pre-treatment. However, in this study the brine was neutralized through the reaction with CO_2 which reduced the pH of the reject brine solution. Nonetheless, analysis of the solid products affirmed the formation of high-purity magnesium hydroxide (See Fig. 4).

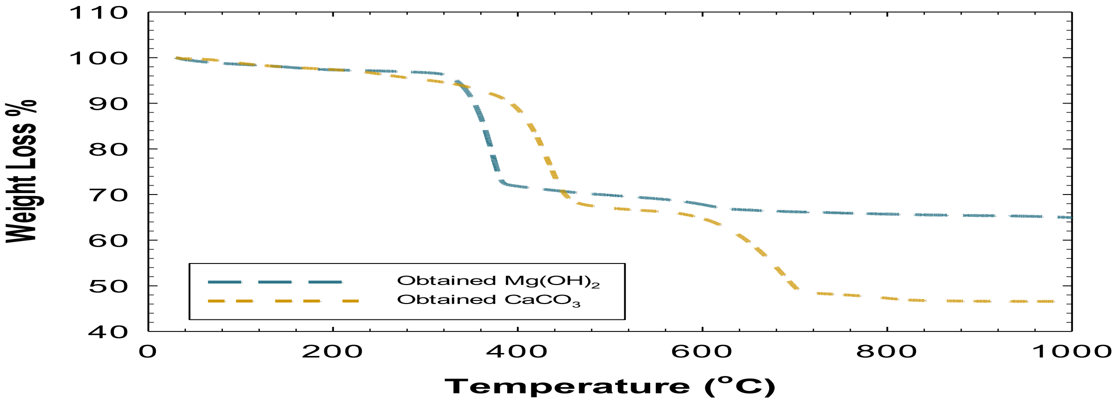

Fig. 4. TGA analysis of the obtained solid product

B. Techno-Economic Considerations for the Proposed Scheme

Based on the composition of the brine used in this work, it is estimated that one million tons of brine will contain approximately 3000 tons of Mg^{2+} ions and 1000 tons of Ca^{2+} ions. Assuming complete conversion of these ions into their respective products, about 7932 tons of $Mg(OH)_2$ and 2488 tons of $CaCO_3$ can be obtained following the proposed process scheme. Additionally, separating these products in two distinct stages enhances the potential for producing highly pure $Mg(OH)_2$ and $CaCO_3$, making them suitable for a variety of applications [15]. Moreover, the proposed scheme has the potential to sequester approximately 10000 tons of CO_2 for every million tons of brine treated. Therefore, successful implementation of this scheme could significantly contribute to achieving carbon neutrality by substantially reducing the carbon footprint associated with desalination processes. The proposed scheme will essentially require around 19220 tons of sodium hydroxide for the entire process for treating a million ton of reject brine. The requirement of sodium hydroxide as input for both stages might increase the cost of the overall process. However, this issue can be mitigated by incorporating an electrolysis unit into the process. The divalent ions-free effluent generated from the treatment of reject brine can be used as a feed for the electrolyzer unit. This effluent still contains significant amounts of sodium which can be converted into NaOH through electrolysis techniques. The NaOH generated from this step can then be recycled into the reactor and mixer units as shown in Fig. 1 within the proposed scheme to produce calcium carbonate and magnesium hydroxide, thereby significantly reducing capital costs and increasing the economic feasibility of the proposed scheme.

IV. CONCLUSIONS

An innovative process scheme for reducing the salinity of reject brine and CO_2 capture has been investigated. Consistent with the proposed scheme, an inert particle spouted bed reactor (IPSBR) was used to react with CO_2 with reject brine in the presence of NaOH and to observe the removal of calcium ions and CO_2 uptake. At NaOH dose of 11 g/L and CO_2 flowrate of 1 L/min, 96% of the calcium ions and 16% of the magnesium ions were removed with a CO_2 uptake of around 10 g/L under ambient conditions. The solid products obtained from this step were filtered and analyzed confirming the formation of high-purity calcium carbonate, $CaCO_3$. Next, the filtrate from this reaction was reacted with NaOH to remove the magnesium ions. To accomplish this step, the magnesium ions present in the obtained calcium-free brine was precipitated through further addition of 8.22 grams of NaOH at ambient temperature. About 83% of the magnesium ions were removed in this step and the analysis of the solid products affirmed the formation of magnesium hydroxide, $Mg(OH)_2$. Both solid products obtained from the individual steps are commercially valuable. A techno-economic evaluation of the combined process indicates the potential to produce 7932 tons of $Mg(OH)_2$ and 2488 tons of $CaCO_3$ while sequestering 10000 tons of CO_2 through the treatment of every million tons of brine. Additionally, the generated divalent ions-free brine effluent can be exhibits great potential to be used as feed for electrolysis to generate acid and base. The NaOH base produced can from this unit can be recirculating to eliminate the need of external addition, thus creating a self-sustaining process that presents a new avenue for CO_2 sequestration and reducing reject brine salinity, thus promoting sustainable management of these two major wastes within the GCC countries, and the world at large.

ACKNOWLEDGEMENT

The authors would like to acknowledge the support of Qatar University Student Grant # QUST-1-CENG-2025-368. The findings achieved herein are solely the responsibility of the authors.

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