

CO₂ capture and ions removal through reaction with potassium hydroxide in desalination reject brine: Statistical optimization

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ABSTRACT

Previous studies have investigated the overall performance of the modified Solvay process based on a new alkaline compound, namely, KOH. Preliminary results have confirmed its high reactivity and effectiveness in capturing CO₂ and managing reject brine. In this study, parametric sensitivity analysis has been carried out to optimize the operating conditions and thereby maximize CO₂ capture and ions removal from high-salinity brines. Response surface methodology (RSM) analysis using the central composite design (CCD) approach was implemented to statistically determine the impact of important operating conditions, including KOH concentration (30–110 g/l), CO₂ gas flow rate (400–1600 ml/min), gauge pressure (1–3 barg), and temperature (10–50 °C) on key response process output variables, such as CO₂ uptake and ions reduction. The importance of these parameters and their interactions were confirmed by employing analysis of variance (ANOVA) approach at a confidence level of 95% ($p < 0.05$). These analyses demonstrated that under the optimized conditions of a temperature of 10 °C, gauge pressure of 2.1 barg, CO₂ gas flow rate of 848.5 ml/min, KOH concentration of 110 g/l, and an inert mixing particle volume fraction of 15%, a maximum CO₂ uptake value of 0.58 g/g KOH, maximum sodium (Na⁺) removal of 44.1%, chloride (Cl⁻) removal of 40.1%, calcium (Ca²⁺) removal of 100%, and magnesium (Mg²⁺) removal of 99.8% were achieved. The characterization of the collected solid products at optimum conditions revealed the production of valuable and useful products, particularly sodium and potassium bicarbonates, in addition to KCl.

1. Introduction

CO₂ is the greenhouse gas with the most significant contribution to global warming. Power plants based on fossil fuels release considerable amounts of CO₂; indeed, they account for almost 60% of the total CO₂ emitted into the environment. Another major environmental concern is the discharge of highly concentrated brines as a by-product of desalination plants [1]. Conventional technologies to treat and manage these pollutants entail numerous limitations such as extensive energy requirements, high costs, and low efficiency. Hence, a combined approach dealing with both CO₂ and reject brines in a single reaction would be the most efficient way to manage these wastes. Proposed approaches should be effective, economically viable, and environmentally friendly [1].

The traditional Solvay process is utilized in producing soda ash

(Na₂CO₃). It involves the reaction of CO₂ with high-salinity water in the presence of NH₃, which results in the production of insoluble NaHCO₃ and soluble NH₄Cl (Eq. (1)) [2]. NH₃ is used only as a buffering reagent (not as a reactant) to increase the pH value to a basic level, and hence shift the reaction toward the production of NaHCO₃. Mohammad et al. [2] optimized the Solvay process with respect to CO₂ capture and sodium removal by applying response surface methodology (RSM) for optimization. Optimum conditions were attained at a gas flow rate of 1.54 l/min, a temperature of 19 °C, and a molar ratio of NH₃ to NaCl of almost 3.3:1; these parameters resulted in a maximum sodium removal of 33% and CO₂ capture efficiency of 86%. However, the NH₃-based Solvay process suffers from multiple disadvantages. For example, NH₃ is a relatively hazardous substance to both health and the environment. Furthermore, the regeneration of NH₃ as shown in Eq. (2) is an

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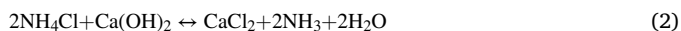
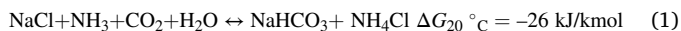
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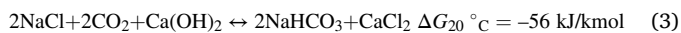
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energy-intensive step that substantially adds to the operating cost of the general process [3, 4].



The reaction between CO_2 and reject brine occurs at high pH values because high pH leads to an increased solubility of CO_2 in reject brine. Therefore, high alkalinity materials ($\text{pH} > 10$) are required to govern the combined process [4]. El-Naas et al. [3] modified the Solvay process by replacing NH_3 with CaO according to Eq. (3). $\text{Ca}(\text{OH})_2$ is produced following the addition of CaO to the brine, after which it reacts with CO_2 to produce NaHCO_3 and soluble CaCl_2 , which can be utilized in many industrial applications [3, 4]. The results demonstrated that the spontaneity of the CO_2 reaction with $\text{Ca}(\text{OH})_2$ is higher than that of the conventional Solvay process (Equation(1)) over the entire range of temperature (0–100 °C). Moreover, parametric sensitivity studies have been conducted using CCD approach-based RSM analysis to evaluate the impact of the most important parameters on process performance. The results indicated that at optimum operating conditions (temperature, gas flow rate, pressure, and CaO concentration of 20 °C, 1 l/min, 1 atm, and 20 g/l, respectively) an optimum CO_2 uptake value of 0.92 g CO_2 /g CaO and a reduction in sodium ions (Na^+) of 35% were achieved. Moreover, it was reported that under the same optimum conditions, the reaction sustained a pH value of 11.8, compared to the Solvay process pH of 11.2. However, increasing the CaO concentration to >20 g/l (less than stoichiometric amount) does not increase the pH but resulted in the formation of CaCO_3 rather than NaHCO_3 .

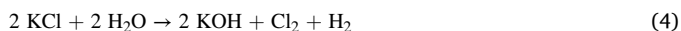


The efficiency of mixed Mg/Al oxides on the capture efficiency of CO_2 and ion removal from reject brine was investigated by Dindi et al. [5], who concluded that the absorption capacity of CO_2 was almost 0.082 g CO_2 /g carbonated solution at 25 °C and 1 bar. Moreover, the percentage reduction of (Na^+) and chloride (Cl^-) ions were 20% and 70%, respectively, resulting in the formation of NaHCO_3 with a yield of 0.044 g NaHCO_3 /g carbonated solution.

However, the use of mixed metal oxides in the modified Solvay process requires additional scrutiny, with the prime aims to identify conditions that enhance the efficiency of CO_2 capture and ion removal.

Moreover, note that certain industrial solid wastes exhibiting alkaline properties can be utilized to promote the pH of the brine solution and form useful products through reactions with CO_2 . This may also provide an alternative for stabilization and safe disposal of these wastes which is a major environmental concern. Ibrahim et al. [6] investigated the utilization of electric arc furnace bag house dust (EAF BHD) as a potential source of CaO and MgO in the modified Solvay process. RSM-CCD was used to optimize CO_2 uptake value under different operating conditions and indicated that an optimum CO_2 uptake of 0.22 g CO_2 /g BHD can be obtained at a temperature of 24 °C, pressure of 1 atm, solid to liquid ratio of 57 g/l, gas flow rate of 0.7 l/min, and inert particle volume fraction of 18%.

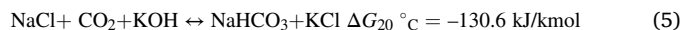
Furthermore, most technologies currently used to directly capture CO_2 from atmosphere require reactions with strong alkaline solutions, such as KOH , due to their strong binding affinities with CO_2 and the ability to maintain a high pH value in the solution [7]. This results in the production of useful carbonate solutions; note that KOH can be produced from the electrolysis of KCl solutions as per Eq. (4) with no CO_2 production, which is advantageous over the use of synthetic CaO produced together with the release of CO_2 gas [8].



Lombardia et al. [9] examined the absorption of CO_2 from landfill gas carried in a packed column (pilot plant scale) with a height of 0.990 m and a diameter of 0.080 m. The chemical reaction between KOH and

CO_2 resulted in the formation of an aqueous solution of K_2CO_3 , which has many industrial applications. The CO_2 capture efficiency was ~98% within the temperature range of 40–45 °C, at atmospheric pressure, with a solution flow rate of 60 l/h and KOH concentration of 53%. The effect of KOH treatment on the properties of activated commercial carbon surface has been studied by Lendzion-Bieluń et al. [10], who concluded that KOH results in an increase of hydroxyl groups at the surface, causing a 14% increase in CO_2 uptake at 0 °C. Mohammad et al. [11] used CCD-RSM to optimize CO_2 adsorption on modified activated alumina (2 g) with 30% KOH . Their results illustrated that, using the optimized conditions of 20 °C and 6 bar, the CO_2 adsorption capacity reached 0.131 g CO_2 /g adsorbent.

Recently, the potential of utilizing KOH to improve the overall performance of modified Solvay process according to Eq. (5) was investigated by Mourad et al. [12]. The reaction resulted in the formation of KCl ; this compound has applications in diverse industrial fields such as the production of crop fertilizers [7]. The results were compared under the same optimum conditions (temperature = 20 °C, a gas flow rate = 1 l/min, and atmospheric pressure) with others determined in a previous study by El-Naas et al. [3] in which CaO was utilized.



The results disclosed that the reaction of CO_2 with KOH was the more spontaneous than the traditional and modified Solvay reactions and had the highest pH value (greater than 13). The addition of KOH at a concentration of 70.57 g/l (*i.e.*, a stoichiometric amount below the solubility limit) resulted in an uptake value of 0.31 g CO_2 /g KOH ; however, the addition of 20 g/l of CaO (*i.e.*, more than the solubility limit and less than the stoichiometry ratio) resulted in an uptake value of 0.92 g CO_2 /g CaO . The percentage of ions removal (Na^+ , Mg^{2+} , Cl^-) was almost equal.

To the best of the authors' knowledge, literature provides no account on a parametric sensitivity analysis of the KOH -based modified Solvay process. To this end, the objective of this study is to investigate the effects of the underlying operating parameters such as temperature, gas flow rate, pressure, concentration of KOH in brine, and the volumetric ratio of mixing particles on the CO_2 uptake and ions reduction from reject brine. Hence, studying the aforementioned factors within the context of RSM will permit a statistically significant evaluation of the impact of the parameters and their interactions with responses (CO_2 uptake and ions reduction); in turn, these provide the optimum conditions for maximizing the efficiency of the overall process.

2. Materials and methods

2.1. Materials

The utilized KOH with a purity of 85%–100% and particle size of 5–7 mm was purchased from Scientific Progress Medical and Scientific Equipment Company, UAE. The gas mixture, which comprises 10% CO_2 and 90% air, was acquired from Abu Dhabi Oxygen Company, UAE.

2.2. Reject brine characterization

Samples of real reject brine were obtained from a local desalination plant located in Abu Dhabi, UAE, and operating multi-stage flash (MSF) desalination process. The reject brine samples displayed an average salinity of 58,560.3 mg/l. Ion concentrations of Ca^{2+} , Na^+ , K^+ , and Mg^{2+} in brine samples were determined by inductively coupled plasma (ICP) spectrometry (Varian 710-ES ICP optical emission spectrometer). Analyses of Cl^- was conducted using a HACH HQ40D portable meter equipped with a chloride ion-selective electrode (HACH IntelliCal™ ISECL181). To enhance the accuracy of each reading, chloride ionic strength adjustment buffer powder pillows were utilized. Moreover, HACH spectrophotometry (HACH-Lange DR5000) was employed

Table 1
Reject brine characteristics.

mg/l	Na ⁺ mg/l	K ⁺ mg/l	Mg ²⁺ mg/l	Cl ⁻ mg/l	SO ₄ ²⁻ mg/l	pH
933±3	23,063±7	877±4	2675±6	48,330±4	5284±3	8.02±0.01

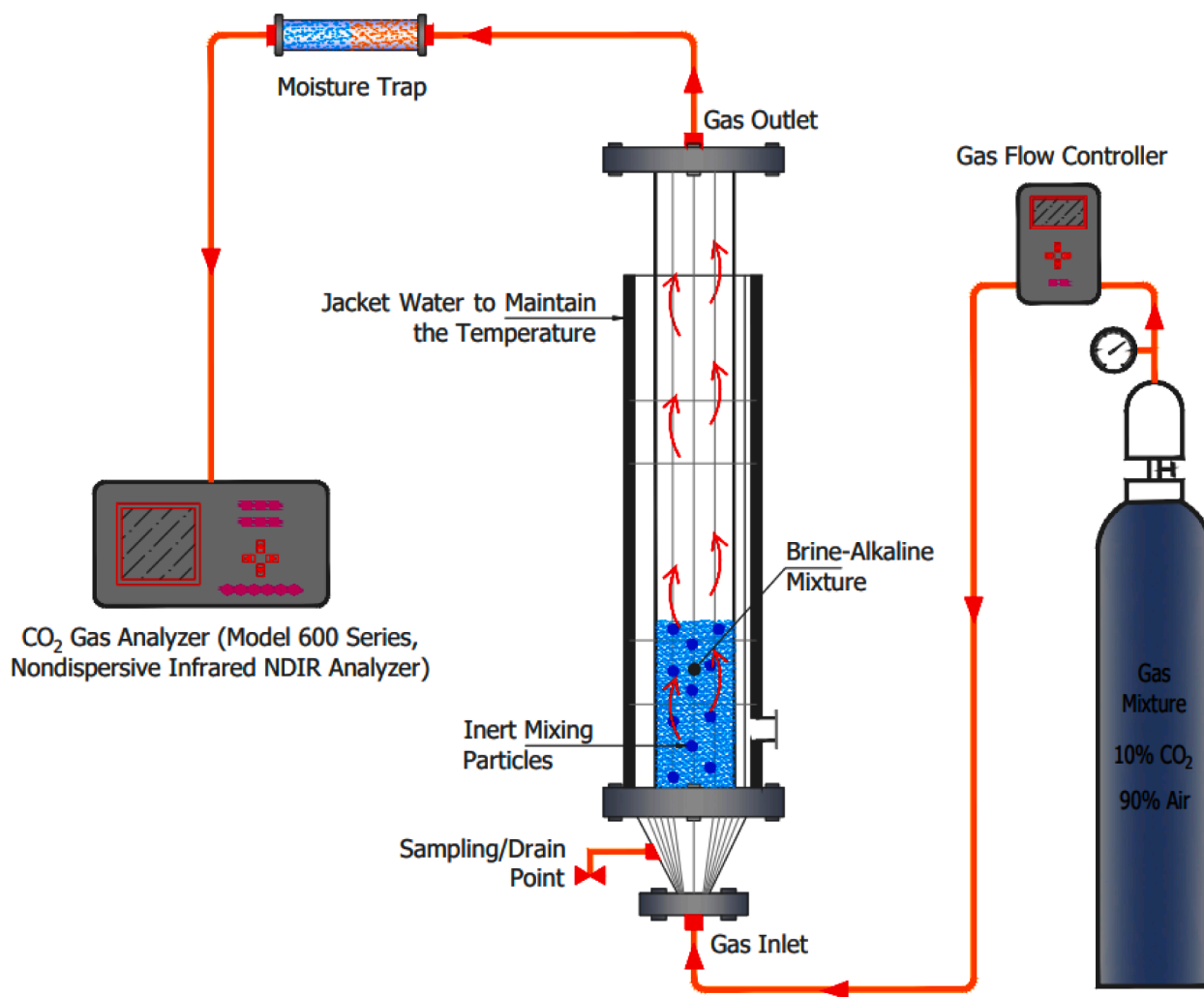


Fig. 1. A schematic diagram of the IPSBR system [12].

Table 2
Reactor dimensions and inter particles specifications [13, 14].

Reactor material	Stainless steel
Reactor internal diameter	78 mm
Reactor height	700 mm
Reactor total volume	300 ml
Orifice diameter	2 mm
Inert particles material	Polymethyl methacrylate
Particle average size	13 mm
Particle average weight	0.511 g/particle
Particle sphericity	0.62
Particle wet density	1.022 g/cm ³
Particle volume	0.5 cm ³ /particle
Particle surface area	4.92 cm ² /particle

to measure the concentration of SO₄²⁻ ions. The pH of reject brine was determined using a HACH portable pH metre (Model HQ11D53000000). All instruments were calibrated using standard solutions. Table 1 lists the concentrations of various ions in the reject brine before treatment.

All readings were determined by considering the averaged value for three collected samples before and after each experiment.

2.3. Experimental methodology and apparatus

In each experimental run, a pre-defined amount of KOH was mixed with 1 l of reject brine using a magnetic stirrer. The pH of the mixture was recorded before reaction with CO₂ gas. All experiments were performed in a novel inert particle spouted bed reactor (IPSBR). More details about the reactor system can be found in previous studies [6, 13–16]. The reactor consists of a cylindrical vessel with a conical base, inert particles, and a jacket to control the temperature. Fig. 1 shows a schematic diagram of the reactor. The dimensions of the reactor and particle specifications follow analogous values previously reported in El-Naas et al. [3] (Table 2).

Initially, the IPSBR system was filled with the prepared solution and exposed to a continuous gas flow (10% vol. CO₂) through an orifice at the bottom of the reactor. The flow rate of the gas was controlled by a mass flow controller. To determine the amount of CO₂ captured, the

Table 3
Independent factors and their levels.

Levels	Feed gas flow rate	KOH concentration	Temperature	Gauge Pressure
Units	l/min	g /l	°C	barg
Tag	F	C	T	P
Level - α	400	30	10	1.0
Level -1	700	50	20	1.5
Level 0	1000	70	30	2.0
Level +1	1300	90	40	2.5
Level + α	1600	110	50	3.0

outlet gas stream was passed through a CO₂ gas analyser. The gauge pressure was controlled using a back pressure regulator valve installed at the reactor outlet. At the end of each experiment, the internal pressure was gradually reduced to atmospheric pressure. Each experiment was concluded when the designated saturation point was reached, i.e., where the CO₂ concentration at the outlet is equal to its inlet value (10% vol.). Liquid samples were taken each 15 min, and the pH value of each sample was recorded. At the end of each experiment, the contents of the reactor were drained and the treated brine liquid was separated from precipitated solids by vacuum filtration using a Buchner funnel setup. Ion concentrations of treated brines were measured using ICP spectrometry. The percentage of ion (Ca²⁺, Na⁺, K⁺, Mg²⁺, Cl⁻) reduction, CO₂ moles reacted, and CO₂ uptake were calculated using Eqs. (6), (7), and (8), respectively [3]. Solid characteristics were investigated using the analytical techniques discussed in Section 2.5.

$$\text{Ion reduction\%} = \frac{X_i - X_f}{X_i} * 100 \quad (6)$$

where X_i denotes the initial concentration of ions in the feed brine (mg/L) and X_f stands for the final concentration of ions in the treated brine (mg/L).

$$\text{Moles of CO}_2 \text{ reacted} = \frac{\int_0^t \text{volume of CO}_2 \text{ captured (L/min).dt}}{\text{Molar volume of CO}_2 \text{ (L/mol)}} \quad (7)$$

$$\text{CO}_2 \text{ uptake} = \frac{\text{mass of CO}_2 \text{ reacted}}{\text{mass of KOH used}} \quad (8)$$

2.4. Experimental design by RSM

The optimization of the modified Solvay process based on KOH was carried out according to the RSM-CCD approach as implemented in Minitab 19.0 [17–19]. RSM is suitable for fitting a quadratic surface and aids in optimizing the process parameters with a minimum number of experimental runs [19–22]. In this study, RSM was used to select CCD in order to design a sequence of experiments to study the influence of the operating parameters on responses. A full-factorial CCD design was employed to investigate the effects of independent factors (i.e., pressure, feed gas flow rate, KOH concentration, and temperature) on responses (i.e., CO₂ uptake and ion reduction in reject brine) throughout the IPSBR. Table 3 lists the values of these independent parameters at each level. A total of 31 experimental runs were generated to analyse the impacts of the operating parameters on responses.

Experimental results were fitted following second-order polynomial equations to evaluate the relative influence of each factor on the studied responses. Moreover, ANOVA was implemented to validate the adequacy of the models developed. The response optimizer in RSM was used to determine the optimum operational conditions; its performance was evaluated based on the desirability parameter (d), that ranges from 0 to 1 [16, 22]. An optimal solution may be reached by the proposed model when the desirability value approaches its maximum (i.e., close to 1). For the purpose of this research, response optimizer requested targets were adjusted to maximize responses, CO₂ uptake, and the percentage of ions reduction.

Furthermore, the effect of residence time and height to diameter (H/D) ratio on the CO₂ uptake and ions reduction is also important. In their modelling of a semi-dry spouted bed, Haghnegahdar et al. [23] demonstrated that, despite increasing the amount of reactants, the removal efficiency of CO₂ decreases with decreasing the residence time of the reactive gas. On the other hand, the impact of increasing the bed height in powder-particle spouted bed reactor was investigated and found to increase the residence time between CO₂ and Ca(OH)₂ in the reactor, thereby increasing CO₂ removal efficiency. [24]. Moreover, Salam et al. [25] evaluated the hydrodynamics performance for two different spouted bed designs and dimensions, reporting that increasing the height of the spouted bed approach increases the minimum spouting velocity and thus the gas holdup and residence time. Other investigators [26–29] reported similar findings. As a result, changing the (H/D) ratio in the tested IPSBR system could affect the residence time distribution as well as the diffusion properties of the CO₂ gas. This point will be addressed in future studies in which different reactor sizes will be tested.

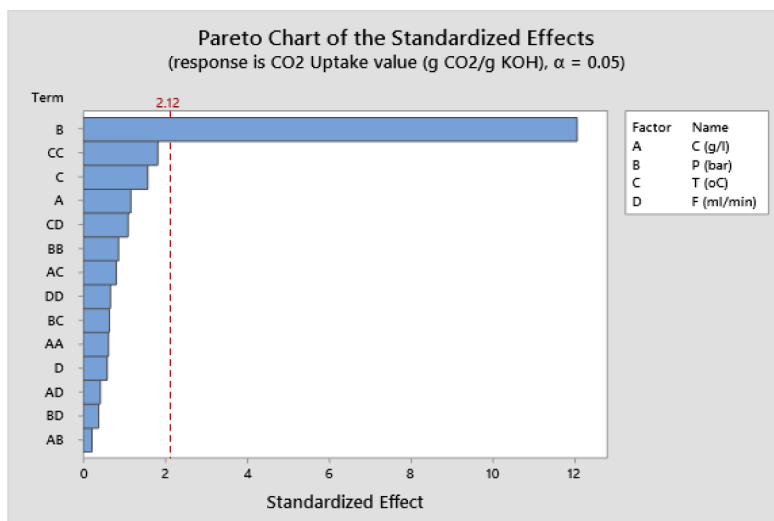


Fig. 2. Pareto chart of the standardized effects for CO₂ uptake value.

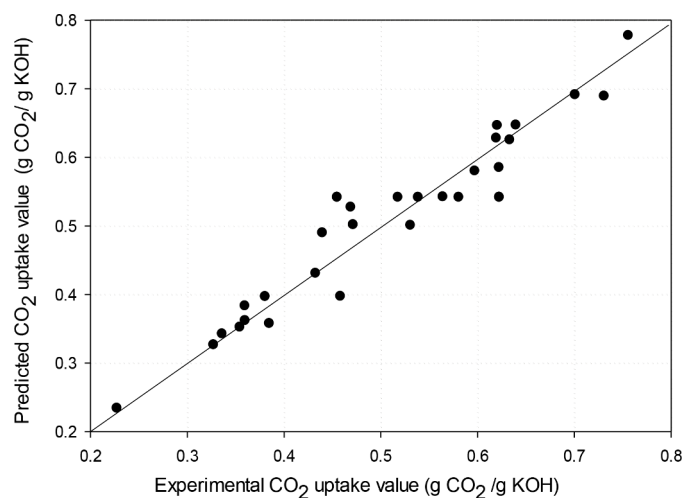


Fig. 3. Predicted (quadratic model) versus experimental CO₂ uptake values.

2.5. Characteristics of precipitated solids

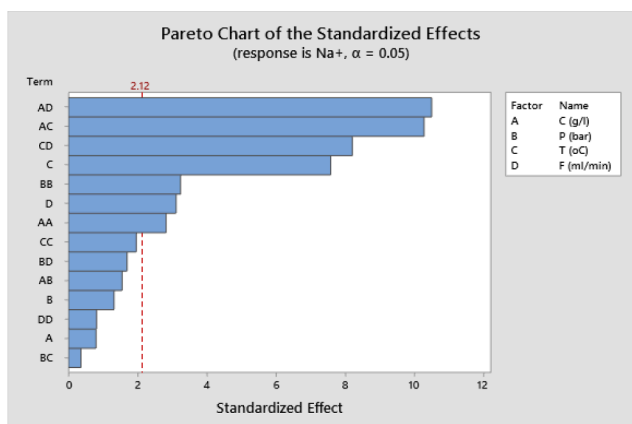
After each experimental run, the collected solids were oven-dried at 110 °C for 48 h. Powder X-ray diffractometer (XRD) was used to characterize and identify solid products. Samples were scanned from 10° to

70° (2 θ) min⁻¹ with a step of 0.013° and a scanning rate of 2° (2 θ) min⁻¹. To determine the surface morphology, texture, and shape of the produced solids, scanning electron microscopy (SEM) was employed at three different areas. Before SEM analysis, the sample was coated with a thin gold film, and energy-dispersive X-ray spectroscopy (EDS) analysis was performed to identify the main components in the solid products. Subsequently, Fourier transform infrared (FTIR) spectroscopy was conducted to investigate the chemical properties and functional groups of the solid samples. An IRTrace-100 FTIR spectrophotometer (Shimadzu, Kyoto, Japan) was used for these analyses. Spectral results were recorded within a range of 500–4000 cm⁻¹ with a 4 cm⁻¹ spectral resolution and 34 scans.

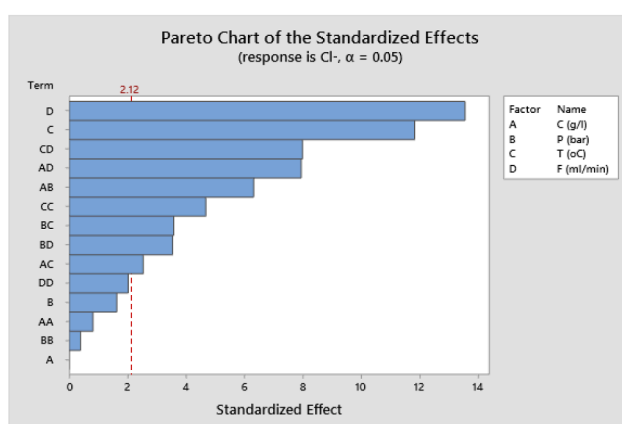
3. Results and discussion

3.1. RSM analysis for CO₂ uptake value

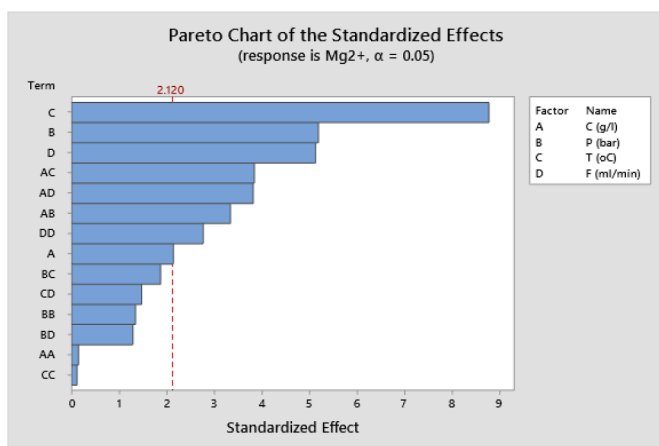
Table S1 presents the experimental and predicted results for CO₂ uptake under different operating factors designed by the RSM-CCD (Supplementary materials). Note that all the experiments were conducted based on an initial inert particle volume of 6%, which was confirmed to be the optimum value by El-Naas et al. [3]. Based on the experimental responses, Eq. (9) shows a second-order polynomial response equation containing the significant factors of KOH concentration (g/l), gas flow rate (ml/min), pressure (bar), and temperature (°C)



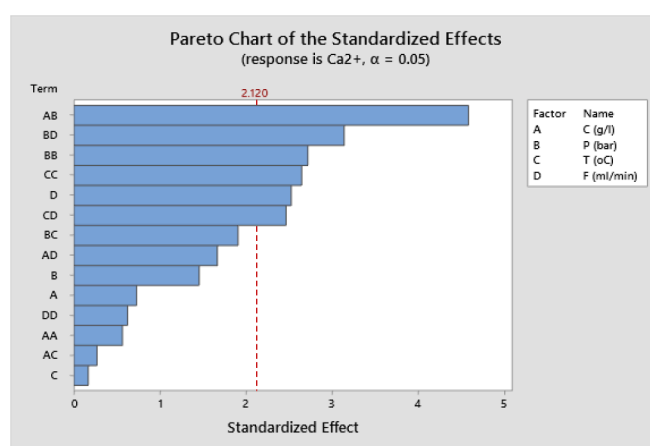
(a)



(b)

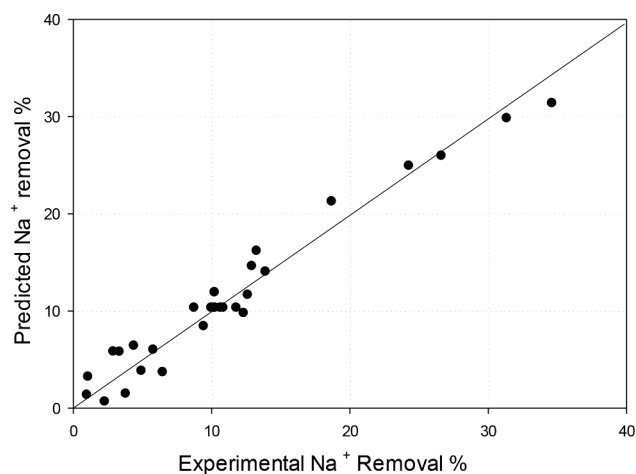


(c)

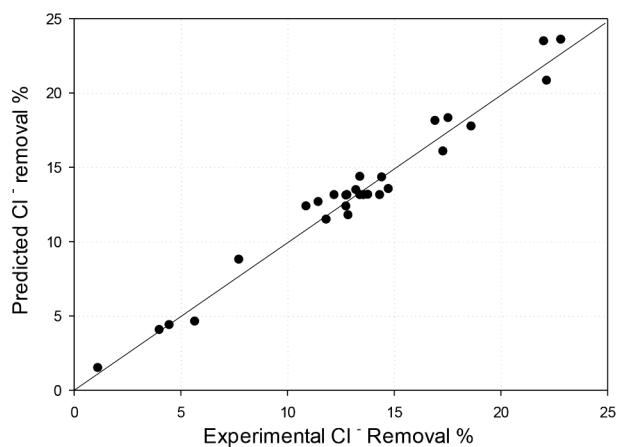


(d)

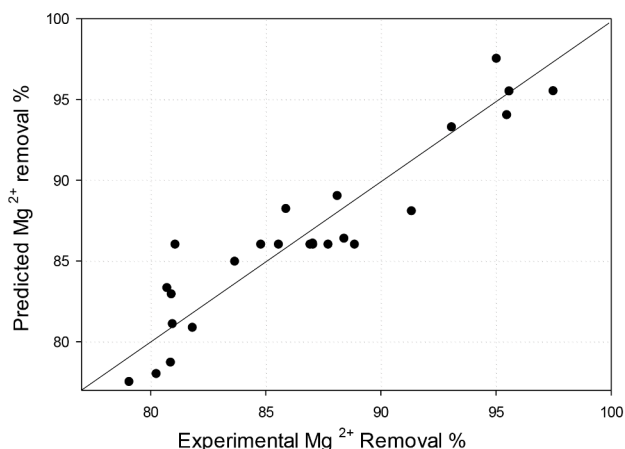
Fig. 4. Pareto chart showing the standardized effects for (a) Na⁺ ion reduction, (b) Cl⁻ ion reduction, (c) Ca²⁺ ion reduction, and (d) Mg²⁺ ion reduction.



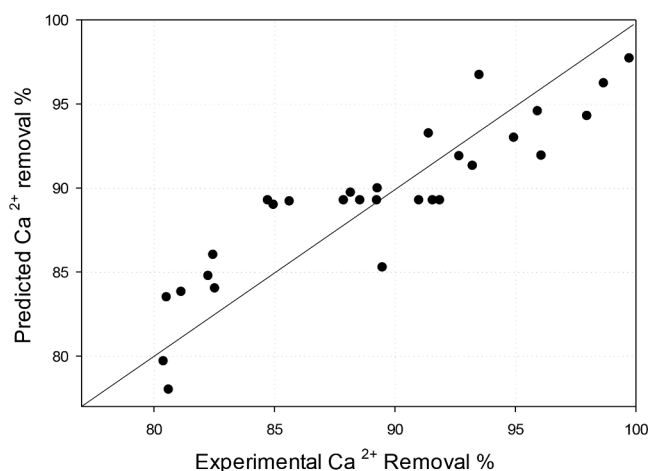
(a)



(b)



(c)



(d)

Fig. 5. Predicted (quadratic model) versus experimental (a) Na^+ ion reduction, (b) Cl^- ion reduction, (c) Mg^{2+} ion reduction, and (d) Ca^{2+} ion reduction.

affecting the response (CO_2 uptake) and interaction between the parameters.

R^2 value of 0.9072 reveals that 90.72% of the data fit the regression model. A small standard deviation value of 0.055 confirms that the

$$\begin{aligned} \text{CO}_2 \text{ uptake value} = & -0.581 + 0.00158 C + 0.413 P + 0.0142 T + 0.000326 F - 0.000016 C^2 - 0.0358 P^2 \\ & - 0.000189 T^2 - 0.000000 F^2 + 0.00030 C * P + 0.000056 C * T - 0.000001 C * F \\ & - 0.00177 P * T + 0.000035 P * F - 0.000005 T * F. \end{aligned} \quad (9)$$

ANOVA was used to evaluate the statistical significance of the developed models such that the model is considered statistically significant when the p-value is <0.05 at the 95% confidence level. If the p-value is >0.05 , it is considered to be insignificant at the 95% confidence level [16, 30, 31]. Table S2 (Supplementary materials) shows that pressure is the only factor that exerts a significant effect on CO_2 uptake, whereas the other factors have trivial effects in the presence of pressure. Moreover, the Pareto plot shown in Fig. 2 confirms that pressure is the most important factor influencing CO_2 uptake values. Note that most parameters had a significant impact on the response. Moreover, the high

model is statistically adequate. The insignificant lack of fit (p-value >0.05) affirms that the model fits well to the experimental data [22].

Figure S1 (Supplementary materials) shows the convergence of the fitted proposed model. The model was also confirmed using the residual plots (SI) [22]. The normal probability plot and histogram indicate that the data are normally distributed. Moreover, residuals versus fits and versus order plots fluctuate, verifying the assumption that the residuals exhibit constant variance and are independent of each other. Fig. 3 shows the additional confirmation of the adequacy of the model. All predicted responses show good agreement with experimental values.

3.2. RSM analysis for ion removal

The experimental design according to CCD, experimental results of ion reduction, and predicted results are shown in Tables S3, S4, S5, and S6 (Supplementary materials) for Na⁺, Cl⁻, Mg²⁺ and, Ca²⁺, respectively. To relate the measured ions removal to specific variables, a full quadratic model was fitted to the data as expressed by Eqs. (10), (11), (12), and (13).

$$\begin{aligned} \text{Na}^+ \text{reduction\%} = & -24.8 - 0.735 C + 26.07 P + 2.985 T - 0.0077 F + 0.00317 C^2 - 5.81 P^2 \\ & + 0.00879 T^2 - 0.000004 F^2 + 0.0932 C * P - 0.03082 C * T + 0.001049 C * F - 0.043 P * T \\ & - 0.00676 P * F - 0.001640 T * F \end{aligned} \quad (10)$$

$$\begin{aligned} \text{Cl}^- \text{reduction\%} = & 60.2 - 0.619 C - 15.03 P + 0.595 T - 0.01968 F - 0.000474 C^2 + 0.365 P^2 \\ & + 0.01086 T^2 - 0.000005 F^2 + 0.1962 C * P - 0.00393 C * T + 0.000411 C * F - 0.2222 P * T \\ & + 0.00733 P * F - 0.000827 T * F \end{aligned} \quad (11)$$

$$\begin{aligned} \text{Mg}^{2+} \text{reduction\%} = & 130.4 - 0.528 C - 24.2 P + 0.289 T + 0.0088 F - 0.00019 C^2 - 2.74 P^2 \\ & - 0.00062 T^2 - 0.000016 F^2 + 0.2271 C * P - 0.01306 C * T + 0.000433 C * F \\ & + 0.255 P * T + 0.00586 P * F - 0.000334 T * F \end{aligned} \quad (12)$$

$$\begin{aligned} \text{Ca}^{2+} \text{reduction\%} = & -22.1 + 0.889 C + 86.2 P - 1.209 T + 0.0365 F + 0.00096 C^2 - 7.41 P^2 \\ & + 0.01803 T^2 - 0.000005 F^2 - 0.4174 C * P + 0.00122 C * T - 0.000253 C * F \\ & - 0.347 P * T - 0.01906 P * F + 0.000749 T * F \end{aligned} \quad (13)$$

The importance of the fitted regression models, main parameters, and their interactions was assessed using ANOVA as shown in Tables S7, S8, S9, and S10 (Supplementary materials) for percentage reductions of Na⁺, Cl⁻, Mg²⁺ and, Ca²⁺, respectively. The Pareto plots illustrated in Fig. 4(a)-(d) are shown to detect the most significant factors and interactions that affect ion reduction. The p-values of all models are <0.05 at the 95% confidence level, indicating that the fitted equations are capable of explaining observed variations in response and can be used to optimize the operating conditions. The independent parameters of temperature, flow rate, and two-level interactions between temperature and flow rate, concentration and flow rate, and concentration and temperature all have a significant effect on the removal of Na⁺ ions. It is also evident that the primary variables of flow rate and temperature, as well as the two-way interactions between these variables, affect the Cl⁻ percentage reduction significantly, whereas the other coefficient terms

induce rather trivial effects. Data illustrated in Fig. 4(c) and Table S9 reveal that all independent factors and interactive cross-product coefficients of concentration and pressure, concentration and temperature, and concentration and flow rate had a significant effect on the removal of Mg²⁺ ions. However, the gas flow rate was detected as the only major factor influencing the reduction of Ca²⁺ ions. Moreover, the interaction effects between concentration and pressure, pressure and gas flow rate, and temperature and gas flow rate are all significant because their corresponding p-values are <0.05.

Note that the R² values for all four models (Tables S7, S8, S9, and S10) are >90%, indicating a good agreement between experimental and

predicted results. Furthermore, the p-value for the lack-of-fit is > 0.05,

denoting that the lack of fit is insignificant, that the model fits well the experimental data, and that the factors have considerable effects on the

response.

The residual plots illustrated in Figures S2, S3, S4, and S5 confirm the adequacy of the results. The normal probability plots demonstrate the independency and normality of the residuals. The residual *versus* order plots illustrate a random uniform scatter of points and an absence of obvious patterns in data. Moreover, Fig. 5(a)-(d) shows that the experimental data are in accord to the predicted data line, indicating an accurate prediction of responses and a close fit between modelled and collected data.

3.3. Process optimization and validation

3.3.1. Optimization of CO₂ uptake combined with maximum ion removal

In this study, the response optimizer in Minitab was utilized to determine the optimum operating conditions for the KOH-based Solvay process. The objectives for optimization were to maximize CO₂ uptake value and ions (Na⁺, Cl⁻, Mg²⁺ and, Ca²⁺) percentage reduction.

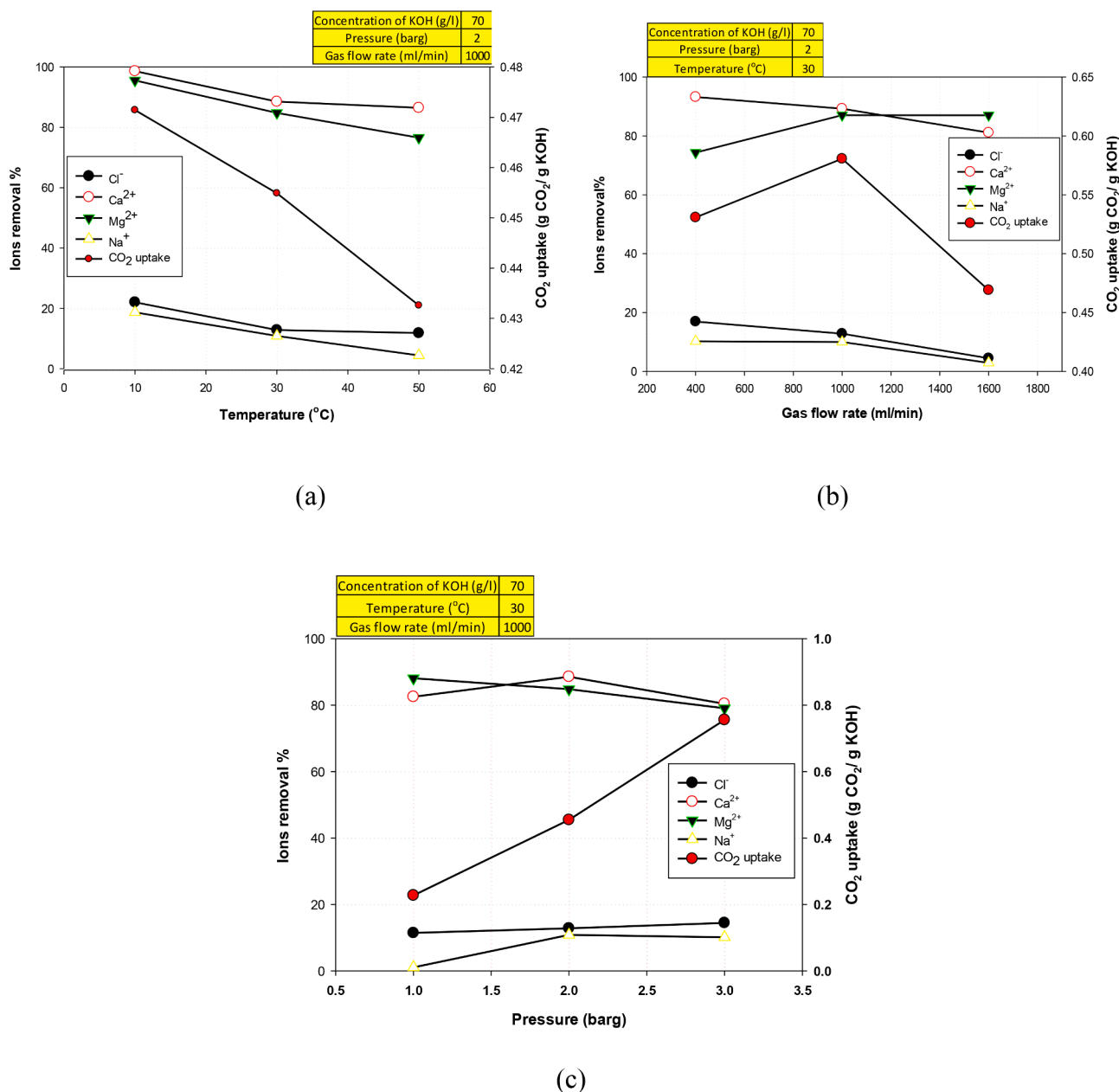


Fig. 6. Effects of (a) temperature, (b) gas flow rate, and (c) pressure on CO₂ uptake and ion removal under constant concentration, pressure, and CO₂ gas flow rate.

Optimization was accomplished by obtaining individual desirability values for each response. Table 4 shows the optimum conditions, confidence interval (CI), and desirability function. The desirability value is almost 1, confirming an optimal performance for the studied factors [16]. From Table 4, it was predicted that, at low temperatures of 10 °C, high KOH concentration of 110 g/l, moderate gauge pressures, and a gas flow rate of 2.1 bar and 848.5 ml/min, the optimum condition could be achieved. Thus, low temperatures increase CO₂ solubility and forward reactions are dominant, causing a decrease in ion solubility by increasing solid precipitation [3], as shown in Fig. 6(a).

According to Eq. (5) and as demonstrated in Fig. 6(b), a high gas flow rate can positively influence CO₂ uptake values by enhancing the reaction rate and shifting it toward the products. However, flow rates higher than 1000 mL/min appeared to have a negative effect on CO₂ uptake, which can be explained by decreasing the gas residence time in the reactor and hence decreasing the reaction rate. Consequently, an intermediate value of gas flow rate was predicted by the response optimizer. Furthermore, pressure exhibits a linear relationship with CO₂

uptake and a limited effect on ion removal as illustrated in Fig. 6(c). Therefore, a gauge pressure value of ~2.1 barg was fitted by Minitab.

3.3.2. Experimental validation of predicted optimized conditions

The optimum conditions were experimentally validated under the same predicted conditions. The results obtained for CO₂ uptake, Na⁺, Cl⁻, Mg²⁺, and Ca²⁺ percentage removal were 0.56 g CO₂/g KOH, 38.8%, 36.1%, 99.8%, and 100%, respectively. These results all reside within the CI of 95% as presented in Table 4, confirming the ability of the model to predict process performance at several operating parameters. Note that Cl⁻ % reduction was greater than the predicted value.

3.4. Optimized conditions under atmospheric pressure

All experimental runs were conducted under a certain gauge pressure (1–3 barg), suggesting a positive influence on CO₂ uptake. The effects of atmospheric pressure on CO₂ uptake and ion reduction were examined under the same optimum conditions obtained in Section 3.3.1, such that

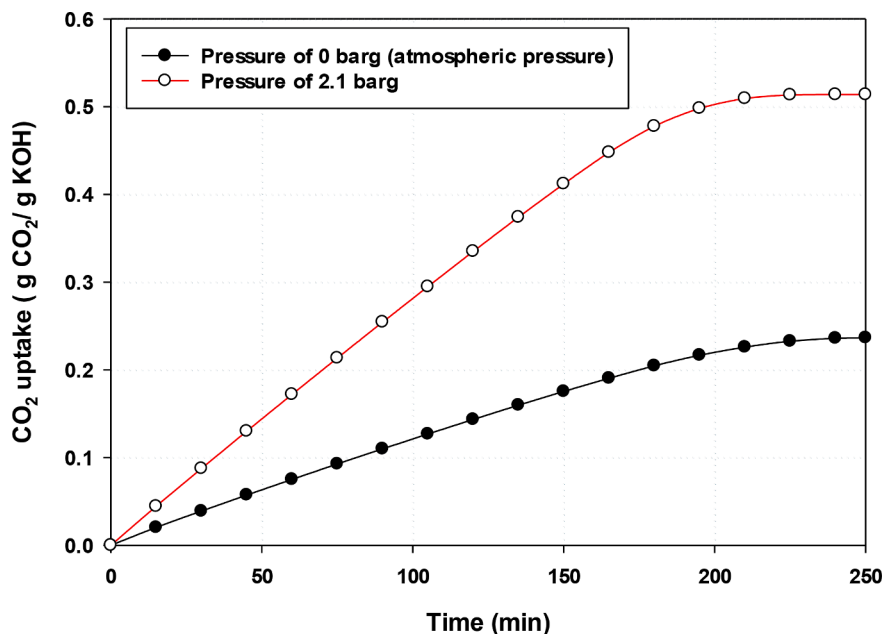


Fig. 7. Effect of atmospheric pressure on CO₂ uptake at the optimum conditions of 110 g/l concentration, 848.5 ml/min gas flow rate, and 10 °C.

the process was optimized based on the combined responses of CO₂ and ion removal. The results were compared as shown in Figs. 7 and 8. Fig. 7 shows the comparison of the uptake of CO₂ with and without pressure. As expected, the CO₂ uptake increased from 0.24 to 0.56 g CO₂ / g KOH when the gauge pressure increased from atmospheric pressure (0 barg) to ~2.1 barg. However, the effect of pressure on ion reduction, as shown in Fig. 8, is almost negligible. K⁺ ion reduction reached up to 63.5% under the optimized conditions. Although KOH is used as the primary alkaline at high concentrations (110 g KOH/l brine), high reduction percentages were achieved in the treated brine. This confirms the high reactivity of the alkaline (KOH) in the presence of CO₂ gas and brine and the formation of valuable products, such as KCl, KHCO₃, and K₂CO₃ under optimized conditions as described in the following sections.

3.5. Influence of inert mixing particle volume fraction

The effects of inert particles on the modified Solvay process in IPSBR

were investigated experimentally in a previous study [3]. It was reported that inert particles create vigorous mixing and circular motion inside the reactor, thereby enhancing the gas–liquid interfacial area and improving mass and heat transfer between the two phases. These results show that inert particles are very effective means of increasing the gas–liquid interfacial area and consequently the mass transfer rate. El-Naas et al. [3] concluded that the optimum condition of CaO-based modified Solvay process was achieved for an inert particles volume fraction of ~6.6%. Therefore, the above-mentioned experiments were conducted based on a particle volume fraction of ~6%. The inert particle volume fraction was obtained using Eq. (14) [6] as follows:

$$\text{Inert particle volume fraction} = \frac{\text{volume of particles}}{\text{volume of reject brine}} * 100\% \quad (14)$$

In this study, the impact of the mixing particle volume fraction was addressed under optimized conditions (Section 3.3.1) such that the KOH concentration is 110 g/l, temperature is 10 °C, pressure is 2.1 barg, and

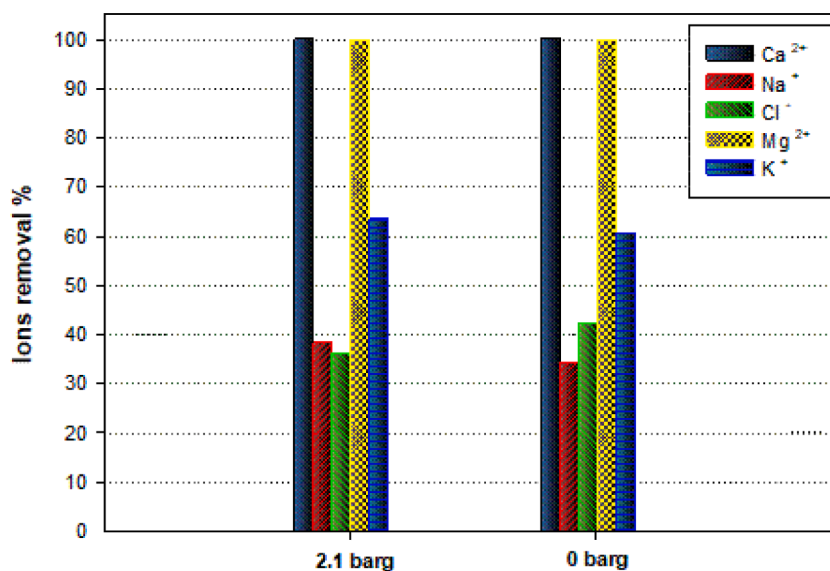


Fig. 8. Effect of atmospheric pressure on ion reduction at the optimum conditions of 110 g/l KOH concentration, 848.5 ml/min gas flow rate, and 10 °C.

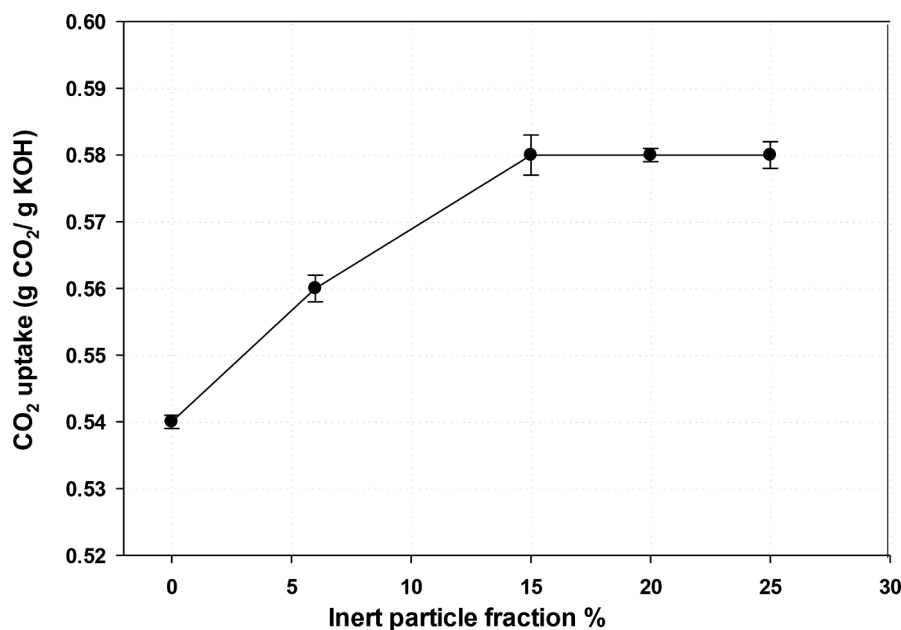


Fig. 9. The effect of particle volume fraction on CO₂ uptake at optimum conditions of 110 g/l KOH concentration, 848.5 ml/min gas flow rate, pressure of 2.1 barg, and temperature of 10 °C.

CO₂ flow rate is 848.5 ml/min. The mixing particle volume fractions employed were in the range of 0%–25%. Figs. 9 and 10 show the influence of inert particle volume fractions on CO₂ uptake and percentage of ion reduction, respectively. As illustrated in Fig. 9, particle fraction has no significant impact on CO₂ uptake. This is evident based on the fact that the value slightly increased from 0.54 (at 0% mixing particles) to 0.58 (at 15% mixing particles). However, adding particles with volume fractions greater than 15% did not further improve CO₂ uptake.

Fig. 10 shows the effect of the particle volume fraction on the reduction of ion concentrations. By increasing the fraction from 0% to

15%, Cl⁻ removal increased from 31.2% to 40.1%, after which it remained almost constant. Similarly, Na⁺ reduction increased from 34.1% at 0% to 44.5% at 15%–25%. The percentage removal of Mg²⁺ at 15%–25% was ~99.8%, slightly higher than that obtained at 0% (93.1%). However, no variation was detected in the reduction percentage of both Ca²⁺ and K⁺, which maintained almost the same values of 100% and 63.5%, respectively. Thus, it can be concluded that the addition of particles primarily enhances the percentage reduction of Mg²⁺, Na⁺, and Cl⁻, until reaching a constant value at a particle volume

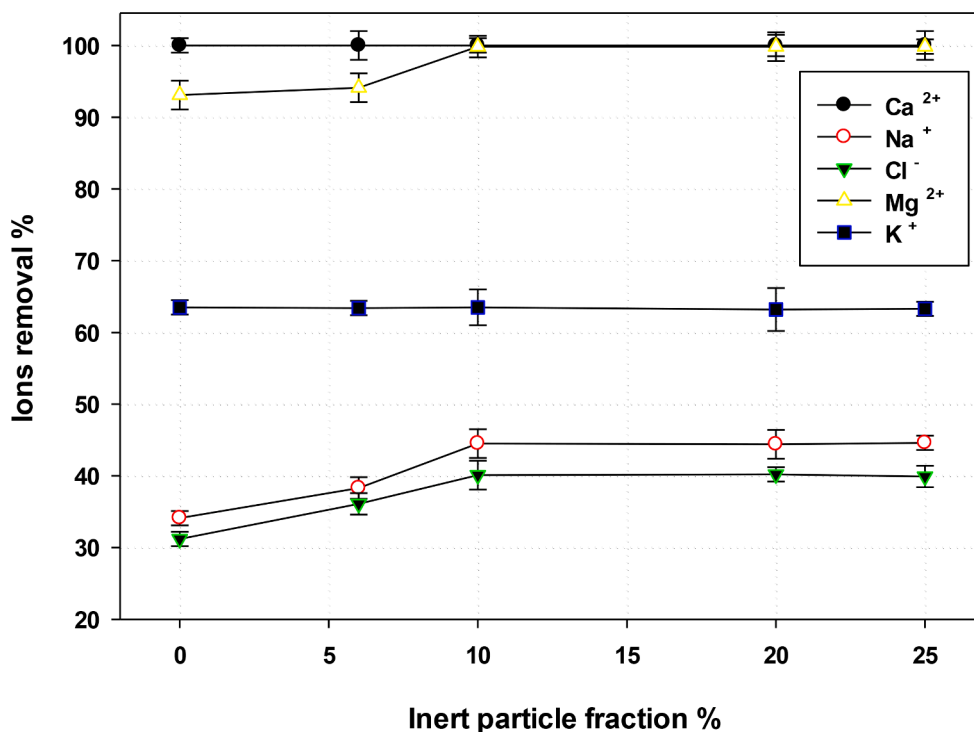


Fig. 10. Effects of particle volume fraction on ions percentage reduction at optimum conditions of 110 g/l KOH concentration, 848.5 ml/min gas flow rate, pressure of 2.1 barg, and temperature of 10 °C.

Table 4
Optimum conditions and fitted responses for CO₂ uptake combined with ion removal.

Optimum operating conditions		Response	Fits	95% CI	Composite desirability
C (g/l)	110	CO ₂ uptake (g CO ₂ / g KOH)	0.542	(0.3629, 0.7604)	0.984
P (barg)	2.1	Na ⁺ % reduction	41.7	(34.00, 49.41)	
F(ml/min)	848.5	Cl ⁻ % reduction	23.6	(19.58, 27.56)	
T (°C)	10	Mg ²⁺ % reduction	97.6	(88.83, 100)	
		Ca ²⁺ % reduction	99.5	(87.77, 100)	

fraction of 15%. This indicates that optimum conditions can be reached at a volume fraction of 15% without additional particle addition (Table 4).

3.6. Modeling of CO₂ uptake using Aqion software

With the use of the Extended Debye-Hückel electrolytic model for activity coefficients [32] as implemented in the Aqion program [33], the gaseous CO₂ absorbed in the KOH-rich brine solution has been simulated. Input to the model are the concentration of ionic species enlisted in Table 1 and the employed partial pressure of CO₂ (0.21 barg). It is predicted that gaseous CO₂ branches into the following dissolved inorganic carbon species (DIC): 0.5 mmol CO₂ (aq)/, 911 mmol HCO₃⁻ and 896 mmol CO₃²⁻. The high yield of HCO₃⁻ and CO₃²⁻ species coincides with the slight increase in the pH value (9 to 11) and the formation of CaCO₃, NaHCO₃, and KHCO₃ as main products. The total load of DIC translates into ~109.35 g/l. Considering the KOH concentration of 110 g/l, the predicated total uptake is 0.99 g CO₂/g KOH, which is much higher than the corresponding experimental value of 0.58 g CO₂/g KOH. The difference calls for the implementation of a more robust electrolytic model in future studies. It is worth noting, however, that the predicated CO₂ uptake based on the stoichiometric ratios of Eq. (5) and taking into account CO₂ solubility is about 0.80 gCO₂ /g KOH.

4. Cost estimation

It is important to note that the modified Solvay process based on different alkaline metals, such as CaO and KOH [3, 34], resulted in a net positive economic benefit by eliminating the associated cost with NH₃ regeneration in the traditional Solvay process (Eq. (1)) [2]. According to El-Naas et al. [3], the modified Solvay process eliminates all NH₃ regeneration energy requirements, which are estimated to be about 30% of total operating energy. Furthermore, by eliminating the work required for steam consumption and compression, which accounts for approximately 70% of the total equivalent work, the modified process significantly reduces the equivalent work.

Table 5 summarizes the estimated cost of consumed alkaline materials for traditional and modified Solvay processes. It also shows the operating conditions, CO₂ capture efficiency, and metals reduction percentage. It is worth noting that the cost of consumed KOH saves approximately 66% and 94% of the cost of consumed CaO and NH₃,

Table 5
Estimated cost of consumed alkaline materials for traditional and modified Solvay processes.

Reference	Alkaline /NaCl molar ratio	Reaction temperature	Reaction pressure	CO ₂ flow rate	Ions reduction%	CO ₂ capture efficiency/ CO ₂ uptake	Cost
Mohammad et al. [2]	3.3 NH ₃ :1NaCl	19 °C	1 bar	1.54 l/min	Na ⁺ : 33% Ca ²⁺ : 91.4% Mg ²⁺ : 97.2%	86%	7 US\$/m ³ treated gas
El-Naas et al. [3, 4]	0.3CaO:1 NaCl	20 °C	1 bar	0.76 l/min	Na ⁺ : 35% Mg ²⁺ :99%	0.92 g CO ₂ /g CaO	1.28 US\$/m ³ treated gas
Current Study	1.95 KOH: 1 NaCl	10 °C	1 bar 2.1 bar gauge	0.848 l/min	Na ⁺ : 44.1% Ca ²⁺ : 100% Mg ²⁺ : 99.8%	0.25 g CO ₂ /g KOH 0.58 g CO ₂ /g KOH	0.4329 US\$ / m ³ treated gas

respectively. Although KOH has a lower CO₂ uptake than some other alkaline, it is superior in high metal ions reduction (44.1% Na⁺, 40.1% Cl⁻, and ~100% for Ca²⁺ and Mg²⁺). Due to its high reactivity and good ions removal compared to other alkaline metals, KOH could be a good alternative, making it more competitive in the global market.

5. Characterization of the solid product under the optimized conditions

Fig. 11(a) shows the structural properties of samples collected at optimized conditions and were determined using X-ray diffractometry with Cu K α radiation ($\lambda = 1.54 \text{ \AA}$). The diffraction pattern was operated at a tube current of 30 mA and a target voltage of 40 kV. The scanning range for 2 θ values was set from 10 ° to 70 °, in order to cover all significant diffraction peaks, using a scan speed of 2°/min. The analytical XRD diffraction peaks for the collected solids were identified based on the theoretical intensity of the three most intense peaks for that mineral in a pure state [35, 36]. The solids collected at optimum operational conditions confirmed the recovery of the brine salts primarily in the form of NaHCO₃, CaCO₃, KCl, KCO₃, and KHCO₃. The produced solids have many applications in diverse industrial fields. NaHCO₃ can be used as a pH-buffering agent, baking soda, and cleaning agent. In addition, it can be utilized as a desulfurization agent in flue gas treatment [3, 37, 38]. The salt (KCl) is widely used as a major ingredient in the production of potassium-fertilizers [36, 39] (95% of fertilizers in the USA are supplied in the form of KCl [39]); it also has some medical applications [40]. Moreover, K₂CO₃ and/or KHCO₃ are widely used as cleaning and emulsifying agents, respectively [41]. Recently, Mohammad et al. [42] investigated the effect of new oil-adsorbent material produced by a modified Solvay process. The solids are mostly made up of NaHCO₃, calcium chloride (CaCl₂), Mg(OH)₂, and potassium carbonate (K₂CrO₄). It was determined that the developed adsorbents can be utilized in oil spill cleanup.

The surface morphology, texture, and shape of the collected solid samples were characterized using SEM observations. SEM micrographs of cross sections of collected samples displayed clear morphological structures as shown in Fig. 11(b). Under optimum conditions, a mixture of agglomerated and scattered unified shape crystals were observed. EDS analyses were also conducted, confirming the results obtained by XRD and showing that the main species in the collected solids were Na, Ca, Mg, C, O, K, and Cl (Fig. 11(b)).

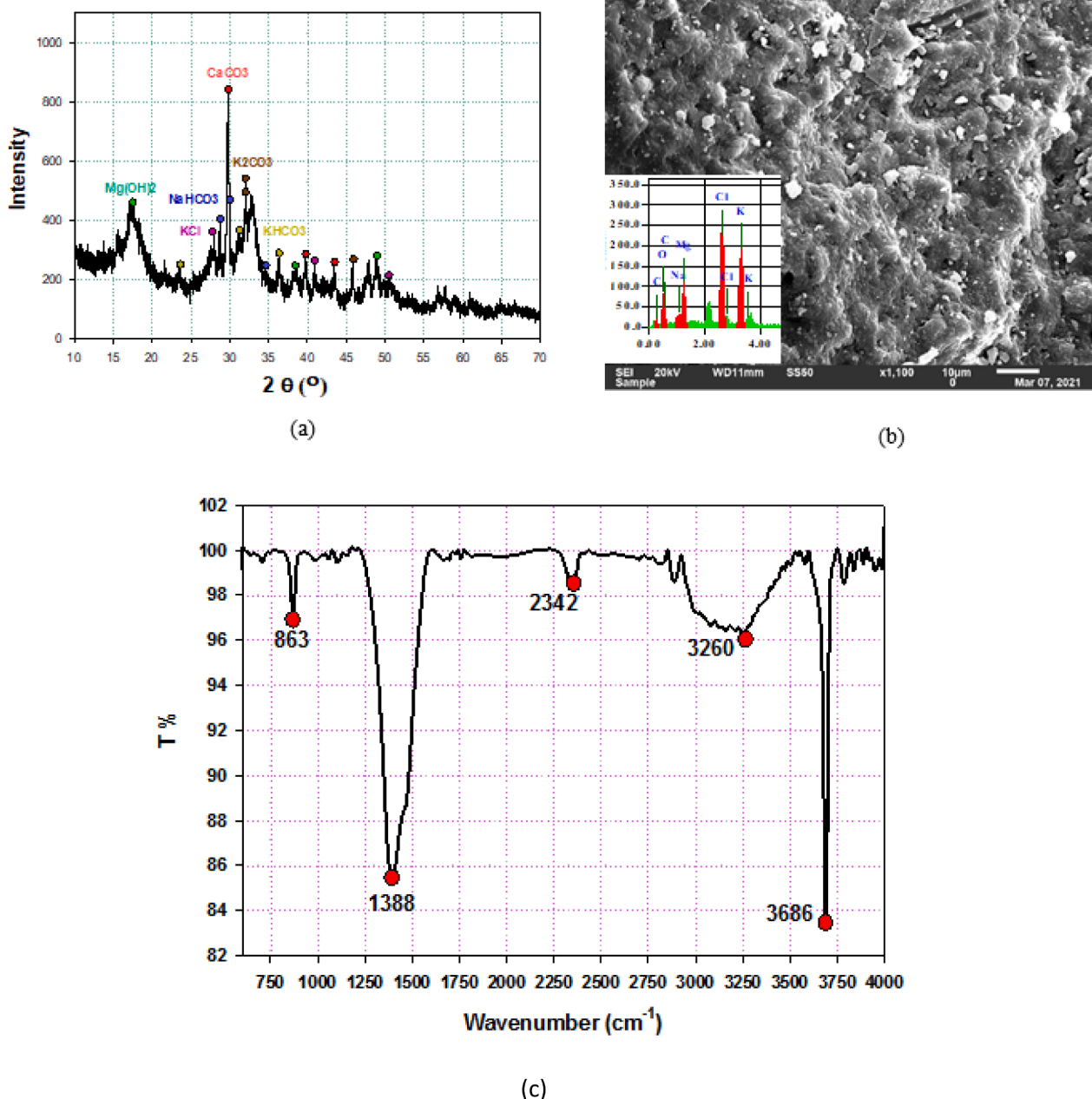


Fig. 11. Solid characteristics determined by (a) X-ray diffraction; (b) SEM, and (c) FTIR under optimized conditions.

Solid samples collected at optimum operational conditions were examined using FTIR to investigate the presence of functional groups. Spectra were acquired across the range of 500–4000 cm^{-1} at a spectral resolution of 4 cm^{-1} and using 34 scans. As shown in Fig. 11(c), many absorption bands with a clear difference amongst collected solid samples were identified. At a wavenumber of 3250–3750 cm^{-1} , a peak signifying the OH^- group was observed. A peak in the range of 2750–3000 cm^{-1} representing C–H stretching was observed in all samples. An absorption band for the carbonate group CO_3^{2-} was clearly detected at 1250–1750 cm^{-1} . At a wavenumber of 800–1200 cm^{-1} , a peak signifying the C–C group was also detected.

6. Conclusions

The optimization of the KOH-based modified Solvay process was investigated to obtain the maximum CO_2 uptake and ion recovery from reject brine. CCD-RSM approach was utilized to predict and optimize the

parameters of four key factors, namely, KOH concentration (30–110 g/l), gauge pressure (1–3 barg), temperature (10–50 $^\circ\text{C}$), and CO_2 gas flow rate (400–1600 ml/min). The experimental results were represented by a second-order regression model and the predicted responses agreed well with the experimental results ($R^2 < 0.95$). The optimum operating conditions were found to be KOH concentration of 110 g/l, gauge pressure of 2.1 barg, CO_2 flow rate of 848.5 ml/min, and temperature of 10 $^\circ\text{C}$. Under these conditions, the maximum CO_2 uptake was ~ 0.58 g CO_2 /g KOH, and the maximum reductions of Na^+ , Cl^- , Ca^{2+} , and Mg^{2+} were 44.1%, 40.1%, 100%, and 99.8%, respectively. The effect of the mixing particle volume fraction was also examined between 0 and 25%, such that the optimum conditions were improved at mixing particle percentages of 15% compared with 6%, while no improvement was detected beyond 15%. The collected solids were characterized using SEM, XRD, and FTIR. Obtained spectra by these surface characterization techniques confirm the production of valuable solid products, such as NaHCO_3 , KHCO_3 , K_2CO_3 , and KCl .

CRedit authorship contribution statement

Aya A.-H.I. Mourad: Conceptualization, Methodology, Software, Data curation, Writing – original draft, Visualization, Investigation, Writing – review & editing. **Ameera F. Mohammad:** Conceptualization, Methodology, Software, Data curation, Visualization, Investigation, Writing – review & editing. **Ali H. Al-Marzouqi:** Visualization, Investigation, Supervision. **Muftah H. El-Naas:** Conceptualization, Software, Investigation, Supervision, Writing – review & editing. **Mohamed H. Al-Marzouqi:** Visualization, Investigation. **Mohammednoor Altarawneh:** Software, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi: [10.1016/j.cep.2021.108722](https://doi.org/10.1016/j.cep.2021.108722).

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