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Statistical Analysis and Optimization of a Process for CO_2 Capture

Muftah H. El-Naas, Ameera F. Mohammad, Mabruk I. Suleiman, Mohamed Al Musharfy, Ali H. Al-Marzouqi

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***Abstract*—**CO_2 capture and storage technologies play a significant role in contributing to the control of climate change through the reduction of carbon dioxide emissions into the atmosphere. The present study evaluates and optimizes CO_2 capture through a process, where carbon dioxide is passed into pH adjusted high salinity water and reacted with sodium chloride to form a precipitate of sodium bicarbonate. This process is based on a modified Solvay process with higher CO_2 capture efficiency, higher sodium removal, and higher pH level without the use of ammonia. The process was tested in a bubble column semi-batch reactor and was optimized using response surface methodology (RSM). CO_2 capture efficiency and sodium removal were optimized in terms of major operating parameters based on four levels and variables in Central Composite Design (CCD). The operating parameters were gas flow rate (0.5–1.5 L/min), reactor temperature (10 to 50 °C), buffer concentration (0.2-2.6%) and water salinity (25-197 g NaCl/L). The experimental data were fitted to a second-order polynomial using multiple regression and analyzed using analysis of variance (ANOVA). The optimum values of the selected variables were obtained using response optimizer. The optimum conditions were tested experimentally using desalination reject brine with salinity ranging from 65,000 to 75,000 mg/L. The CO_2 capture efficiency in 180 min was 99% and the maximum sodium removal was 35%. The experimental and predicted values were within 95% confidence interval, which demonstrates that the developed model can successfully predict the capture efficiency and sodium removal using the modified Solvay method.

***Keywords*—**Bubble column reactor, CO_2 capture, Response Surface Methodology, water desalination.

I. Introduction

REDUCING CARBON dioxide emissions from fossil fuel combustion power plants is becoming essential to minimize global warming created by the greenhouse effect [1]. Many countries have agreed to mitigate the global warming and climate change problems by decreasing CO_2 emission to the atmosphere. The CO_2 capture from fossil-fueled power plants is a potential method for controlling greenhouse gas emissions, where fossil-fueled power plants are producing about 40% of total CO_2 emissions [2]. Many efficient approaches have been developed to capture CO_2 from flue gases in power generation process [3]. CO_2 can be captured at different combustion levels such as: post-combustion capture, pre-combustion capture and oxyfuel combustion capture [4]; however, there are some disadvantages for these approaches, such as the extensive energy required in such systems. Currently, more efficient and less-energy intensive processes have been considered. The shortage of water supplies for drinking and irrigation purposes is already a serious problem in many parts of the world. Severe water shortages may occur in many countries of the European Union and the Northern Mediterranean by 2020 [5]. There is an urgent need to develop a new process for the management of desalination reject brine that can be used by desalination plants since reject brine has to be utilized and the environmental effects associated have to be sufficiently considered. The chemical reaction of reject brine with carbon dioxide is believed to be a new effective, economic and environmental friendly approach [6]. The chemical reactions are carried out based on the Solvay process to convert the reject brine into useful and reusable solid product (sodium bicarbonate). At the same time, the treated water can be used for irrigation and other industrial applications. Another advantage for the Solvay process is capturing CO_2 gas from industrial exhausts or flue gases. The reactions of CO_2 with ammoniated brine can be optimized at 20 °C and can achieve good conversion using different forms of carbon products [6]. The ammonia in this process is not involved in the overall reaction; it only buffers the solution at a basic pH and increases the precipitation of sodium bicarbonate. From this point view, the Solvay process has been modified, where ammonia is replaced by a buffering agent, which is used to raise the pH level more than 11 and capture the CO_2 by reaction with sodium chloride to form a precipitate of sodium bicarbonate. The process has many benefits such as decreasing salinity in the reject brine and reducing carbon dioxide emissions to the atmosphere. In addition, the process eliminates the need for ammonia recovery which is an energy intensive step in the Solvay process. Accordingly, the effect of process parameters on CO_2 capture and brine desalination is mainly significant. RSM is a statistical technique used for optimizing process factors and their interactions. In addition, a suitable polynomial equation for describing the response surface can be employed [7].

Many studies have investigated the application of RSM to CO_2 capture processes. The effect of temperature and CO_2 partial pressure on CO_2 capture capacity and activated carbon's breakthrough time using response surface method have been tested by [8]. Amine based CO_2 capture process has been optimized using RSM by [9]. Chunfeng Song optimized cryogenic CO_2 capture process using RSM and investigated

Muftah H. El-Naas is with Gas Processing Center, Qatar University, Doha, Qatar (Corresponding author, phone: +974 4403 7695; fax: +974 4403 4371; e-mail: muftah@qu.edu.qa).

Ameera F. Mohammad and Ali H. Al-Marzouqi are with Chemical and Petroleum Engineering Department, UAE University, Al Ain, UAE (e-mail: a.fares@uaeu.ac.ae, hassana@uaeu.ac.ae).

Mabruk I. Suleiman and Mohamed Al Musharfy are with Takreer Research Center, Abu Dhabi, UAE (e-mail: missa@takreer.com, musharfy@takreer.com).

the effect of gas flow rate, temperature and operating time [10]. Mulgundmath and Tezel studied the effect of purge/feed flow ratio, purge time, purge gas temperature and adsorption pressure on CO_2 recovery in a temperature pressure swing adsorption system using RSM [11].

The main objective of the present work is to optimize the desalination of the reject brine and CO_2 capture efficiency based on the modified Solvay process and evaluate the optimum conditions for CO_2 capture and sodium removal using real reject brine.

II. Materials and Methods

A. Gas Mixture, Chemicals and Brine Samples

A gas mixture of (10% CO_2 and 90% air) was obtained from Abu Dhabi Oxygen Company, UAE. Sodium chloride and buffer (purity 99.9%) were purchased from Scientific Progress Medical and Scientific Equipment, UAE. Reject brine samples with salinity range of 70,000 mg/L to 80,000 mg/L were obtained from a local desalination plant utilizing multi-stage flash (MSF) desalination process.

B. Experimental Set-Up

The system consisted of a contact reactor (a stainless steel jacketed, bubble column reactor (SSR)), CO_2 gas analyzer (Model 600 series, Non-Dispersive Infrared (NDIR) analyzers), water bath circulation unit to control temperature through the jacket, piston pump, gas mixture cylinder and gas flow controller. The contact reactor operated in a semi-batch mode, where the buffer-brine mixture was exposed to a continuous flow of carbon dioxide mixture with air. A SCADA panel was installed to control and monitor the process parameters such as: temperature, pressure, liquid level, and gas and liquid flow rates.

C. Experimental Methods

Synthetic brine samples with a specific sodium chloride concentration have been prepared. One liter of the synthetic brine was mixed with a specific amount of buffer for five minutes, and the mixture was then fed to the reactor at a controlled-temperature and atmospheric pressure. A gas mixture containing 10% CO_2 and 90% air was bubbled into the reactor at a controlled flow rate for 120 minutes. Brine samples were collected and then tested for sodium removal using Inductively Coupled Plasma spectrometry (Varian 710-ES ICP Optical Emission Spectrometer). Meanwhile, the effluent gas was continuously passed through CO_2 gas analyzer to detect the CO_2 percentage.

D. Experimental Design

RSM which is a mathematical and statistical technique was used to optimize the process. The aim of this statistical technique is to determine the optimum condition that results in the maximum CO_2 capture efficiency and sodium removal through careful design of experiments [12]. Full factorial design was used for fitting a second-order model, which is defined as the following:

$$Y = \beta_o + \sum_{i=1}\beta_i X_i + \sum_{i=1}\beta_{ii} X_i^2 + \sum_{i=1}\sum_{j=i+1}\beta_{ij} X_i X_j$$

where Y is the response function, β_o the offset term, β_i the coefficient of the linear effect, β_{ii} the coefficient of squared effect, X_i the coded value of variable i, X_j the coded value of variable j and β_{ij} the coefficient of interaction effect [13]. The CO_2 capture and sodium removal based on the modified Solvay method was optimized using RSM. The numerical optimization was followed by analyzing the critical factors and their interactions. The design of runs was in accordance with CCD. The reaction time was investigated in a screening study and set to be two hours, since maximum sodium removal was achieved at this time. The four major factors which affect both CO_2 capture efficiency and sodium removal were temperature, buffer concentration, brine salinity and gas flow rate; these factors were operated in the range of 10 to 50 ºC, 2 to 26 g/L (0.2-2.6%), 25 to 197 g NaCl/L and 0.5 to 1.5 L/min, respectively. The experimental conditions for the CCD runs are presented in Table I. The optimal temperature, gas flow rate, buffer concentration and brine salinity for CO_2 capture and sodium removal have been found by the response optimizer.

TABLE I
Range and Level of Independent Variables for CCD Runs

Factors	Tag	Symbol	Units	Level -α	-1	0	1	+α
Temperature	T	X_1	°C	10	20	30	40	50
Buffer concentration	B	X_2	g/L	2	8	14	20	26
Salinity	S	X_3	g NaCl/L	25	68	111	154	197
Gas flow rate	F	X_4	L/min	0.5	0.75	1	1.25	1.5

E. Calculations

The following equations have been used to calculate the sodium removal percentage (1) and CO_2 capture efficiency (2)-(4).

$$\textit{Sodium removal } \% = \frac{X_i - X_f}{X_i} \times 100\% \quad (1)$$

$$CO_2 \text{ capture efficiency} = \frac{\text{Moles of } CO_2 \text{ captured}}{\text{Moles of } CO_2 \text{ loaded to the reactor}} \times 100\% \quad (2)$$

$$\text{Moles of } CO_2 \text{ captured} = \frac{\int_0^t \text{Volume of } CO_2 \text{ captured}\left(L/\min\right).dt}{\text{Molar volume of } CO_2 \ \left(L/\text{mol}\right)} \quad (3)$$

where X_i is the initial sodium concentration in the feed brine (mg/L), X_f is the final sodium concentration in the treated brine (mg/L), t is the time in minutes.

III. Results and Discussion

A. Statistical Analysis

The analysis of variance in Minitab 17.0 software was used for regression analysis for the obtained data to estimate the coefficient of the regression equation. The fitted polynomial equation was expressed as three dimensional (3D) surface in order to visualize the relationship between the responses and experimental levels of each factor and to infer the optimum conditions. A total of 31 runs for optimizing the four individual parameters in the CCD were undertaken. Experimental conditions, experimental results and predicted results according to the factorial design are shown in Table II.

TABLE II
FULL FACTORIAL CCD FOR CO_2 CAPTURE AND SODIUM REMOVAL

Run #	Experimental conditions				Results			
					CO_2 capture efficiency %		Sodium removal %	
	X_1 T	X_2 B	X_3 S	X_4 F	Exp.	Pred.	Exp.	Pred.
1	30	14	111	1	97.4	96.1	13.5	14.1
2	40	20	154	0.75	91.8	93.2	10.0	8.3
3	30	14	197	1	97.3	95.9	12.3	9.8
4	30	14	111	1	96.1	96.1	16.3	14.1
5	30	14	111	1.5	89.4	88.7	7.8	7.0
6	30	14	111	1	95.7	96.1	13.0	14.1
7	30	14	111	1	95.2	96.1	13.2	14.1
8	20	8	68	0.75	91.2	91.3	18.4	16.5
9	40	8	154	0.75	78.7	78.0	8.4	8.2
10	20	20	68	0.75	99.0	99.1	33.6	31.9
11	30	2	111	1	71.7	71.4	2.1	3.5
12	30	14	111	1	96.5	96.1	12.9	14.1
13	40	8	68	0.75	73.7	74.4	6.8	6.2
14	30	14	111	0.5	94.5	93.3	16.4	17.4
15	20	20	68	1.25	92.7	94.2	22.1	23.0
16	30	14	111	1	95.4	96.1	15.3	14.1
17	40	8	154	1.25	77.4	78.4	5.9	6.7
18	40	20	68	1.25	89.2	89.1	7.8	6.7
19	30	14	111	1	96.7	96.1	14.7	14.1
20	40	20	68	0.75	90.3	90.9	12.2	12.6
21	10	14	111	1	99.9	98.9	27.6	26.8
22	30	26	111	1	93.7	92.0	18.7	17.5
23	20	8	154	1.25	90.3	90.5	9.2	9.6
24	40	20	154	1.25	90.4	91.1	2.7	5.3
25	20	20	154	1.25	94.3	94.5	17.6	17.2
26	40	8	68	1.25	74.8	75.0	4.4	1.9
27	20	8	154	0.75	92.1	93.2	13.9	14.1
28	30	14	25	1	92.5	92.0	10.9	13.6
29	20	20	154	0.75	98.9	99.7	20.0	23.2
30	50	14	111	1	79.6	78.6	3.7	4.7
31	20	8	68	1.25	89.2	88.8	8.4	9.2

B. Analysis of Variance (ANOVA)

ANOVA for the CO_2 capture efficiency from the central composite design are presented in Table III. The statistical significance was evaluated using the P-value of the model, factors and interactions. In this analysis, the model is considered to be significant since the corresponding P-value is less than 0.05. The temperature (X_1), buffer concentration (X_2), salinity (X_3) and gas flow rate (X_4) are significant factors on CO_2 capture since they have P-values less than 0.05. The predicted model of CO_2 capture (7) was obtained by the following second-order polynomial function.

$$CO_2\ capture\ efficiency\ \% = 60.55 - 0.328X_1 + 3.118X_2 + 0.0833X_3 + 33.44X_4 - 0.01844X_1^2 - 0.10020X_2^2 - 0.000300X_3^2 - 20.55X_4^2 + 0.03632X_1 \times X_2 + 0.307X_1 \times X_4 \quad (7)$$

TABLE III
ANOVA FOR CO_2 CAPTURE EFFICIENCY VERSUS TEMPERATURE (X_1), BUFFER CONCENTRATION (X_2), SALINITY (X_3) AND GAS FLOW RATE (X_4)

Source	Degree of freedom	Standard error	F-value	P-value
Model	14	1853.63	95.80	0.000[a]
X_1	1	561.15	406.02	0.000[a]
X_2	1	577.42	417.80	0.000[a]
X_3	1	11.66	8.44	0.010[a]
X_4	1	30.09	21.77	0.000[a]
X_1^2	1	97.19	70.32	0.000[a]
X_2^2	1	372.06	269.21	0.000[a]
X_3^2	1	8.81	6.37	0.023[a]
X_4^2	1	47.16	34.12	0.000[a]
X_1X_2	1	76.00	54.99	0.000[a]
X_1X_3	1	2.91	2.11	0.166
X_1X_4	1	9.42	6.82	0.019[a]
X_2X_3	1	1.89	1.37	0.259
X_2X_4	1	5.71	4.13	0.059
X_3X_4	1	0.05	0.03	0.857

[a] significant model, factor or interaction at p-value < 0.05.

The coefficients of determination (R^2) of the regression equation was 0.99, indicating that the polynomial can adequately describe the relationship between the factors, interactions and responses. The p-value of lack-of-fit is more than 0.05, which implies that the fit is significant. The model adequacy was further verified by analyzing the residuals for both responses. The analysis shows that all the residuals fell within the range of -1 to + 1, and they are randomly distributed around zero, in addition, there was no noticeable trend or pattern in the variation in the residuals which indicates a good agreement between the model predictions and the experimental values. RSM results of the sodium removal is shown in Table IV. The results indicate the model is significant (P-value < 0.05). The effect of temperature (X_1), buffer concentration (X_2), salinity (X_3) and gas flow rate (X_4) are significant (P-value < 0.05). The lack-of-fit implies that the fit is significant (P-value > 0.05). The coefficients of determination (R^2) of the regression equation was 0.95. The model adequacy was further verified by plotting the normal probability and residual plots for the response. The residuals analysis indicates an excellent agreement between the model predictions and the experimental values. The predicted model of sodium removal (8) was obtained by the following second-order polynomial function:

$$Sodium\ removal\ \% = 18.6 - 0.855X_1 + 3.338X_2 - 0.0091X_3 - 7.7X_4 - 0.0250X_2^2 - 0.03774X_1 \times X_2 + 0.00258X_1 \times X_3 - 0.00604X_2 \times X_3 \quad (8)$$

C. Model Validation

Two random experiments were performed to further validate the mathematical models obtained by CCD. Experimental conditions of temperature (X_1), buffer concentration (X_2), salinity (X_3) and gas flow rate (X_4) were chosen randomly from the ranges of Table I. The model predictions and experimental results of the CO_2 capture and sodium removal for these validation experiments are presented in Table V. The CO_2 capture and sodium removal models show excellent agreement with the experimental data at the 95% Confidence Interval (95% CI). Figs. 1 (a) and (b) show the actual and predicted values for CO_2 capture efficiency and sodium removal, respectively. The results prove that the developed models can successfully predict both responses using the modified Solvay method.

TABLE IV
ANOVA FOR SODIUM REMOVAL VERSUS TEMPERATURE (X_1), BUFFER CONCENTRATION (X_2), SALINITY (X_3) AND GAS FLOW RATE (X_4)

Source	Degree of freedom	Standard error	F-value	P-value
Model	14	1417.73	23.58	0.000[a]
X_1	1	635.09	147.85	0.000[a]
X_2	1	225.33	52.46	0.000[a]
X_3	1	30.85	7.18	0.016[a]
X_4	1	135.39	31.52	0.000[a]
X_1^2	1	4.70	1.09	0.311
X_2^2	1	23.21	5.40	0.034[a]
X_3^2	1	10.24	2.38	0.142
X_4^2	1	6.65	1.55	0.231
X_1X_2	1	82.04	19.10	0.000[a]
X_1X_3	1	19.74	4.59	0.048[a]
X_1X_4	1	8.96	2.08	0.168
X_2X_3	1	38.91	9.06	0.008[a]
X_2X_4	1	2.30	0.54	0.475
X_3X_4	1	7.97	1.85	0.192

[a] significant model, factor or interaction at p-value < 0.05.

TABLE V
THE PREDICTED AND EXPERIMENTAL RESULTS OF CO_2 CAPTURE AND SODIUM REMOVAL FOR THE CCD AT 95% CONFIDENCE INTERVAL

X_1 T °C	X_2 B g/L	X_3 S g/L	X_4 F L/min	Results	CO_2 capture efficiency %	Sodium removal %
				Exp.	91.7	19.4
10.0	5.0	150.0	0.5	Pred.	88.3	15.4
				95% CI	83.3- 93.4	6.5- 24.3
				Exp.	96.4	16.4
30.0	15.0	75.0	1.0	Pred.	95.7	15.3
				95% CI	94.8- 96.8	13.6- 16.9

D. Model Optimization

Response surface optimizer has been used to find the optimal set of experimental parameters in the considered range that maximize the CO_2 capture efficiency and sodium removal. The optimum CO_2 capture efficiency was found to be at a temperature of 17 °C, a gas flow rate of 0.76 L/min, a buffer concentration of 1.6% and water salinity of 124 g NaCl/L as shown in Fig. 2. The optimum conditions were tested experimentally using desalination reject brine with salinity of 71,700 mg/L. As shown in Table VI, the CO_2 capture efficiency in 180 min was 99% and the maximum sodium removal was 35%. The experimental and predicted values are very close and within the 95% Confidence Interval; which indicate that the model is fit and can predict the performance of the process at different conditions.

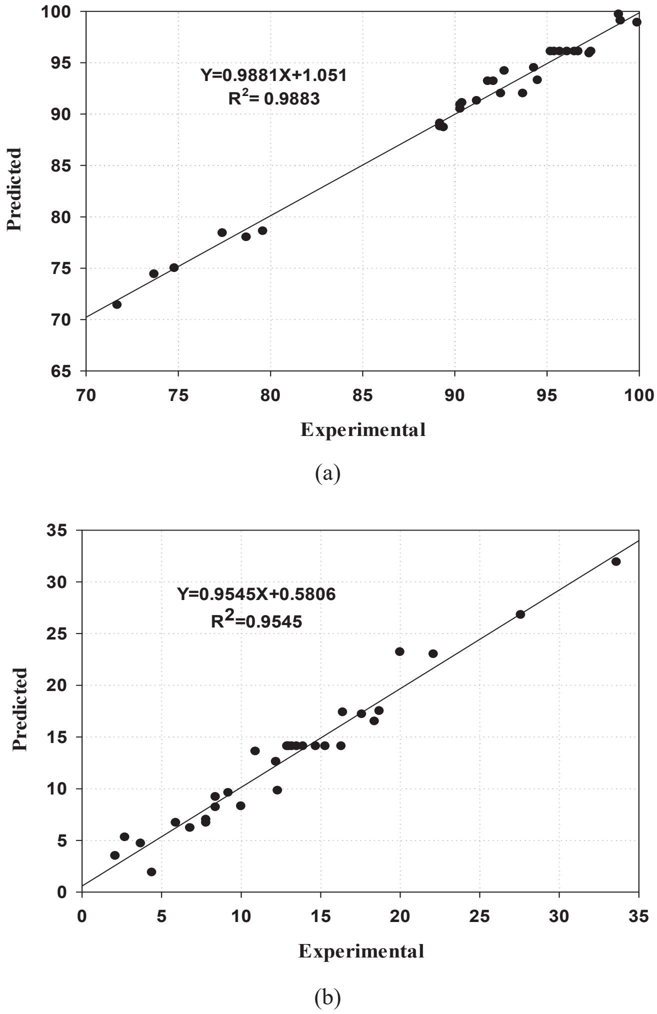


Fig. 1 The CCD predicted and experimental data for (a) the CO_2 capture efficiency and (b) the sodium removal

TABLE VI
PREDICTED AND EXPERIMENTAL CO_2 CAPTURE AND SODIUM REMOVAL AT THE OPTIMUM CONDITIONS USING REAL REJECT BRINE

X_1 T °C	X_2 B g/L	X_3 S g/L	X_4 F L/min	Results	CO_2 capture efficiency%	Sodium removal %
				Exp.	99.3	35.2
17	16	71.7	0.75	Pred.	100	31.0
				95% CI	98.5-100	25.6-36.5

E. Effect of Temperature and Buffer Concertation

Fig. 3 shows the two dimensional (2D) contour plots of the effects of temperature (X_1) and buffer concentration (X_2) on CO_2 capture efficiency and sodium removal while keeping the salinity (X_4) and gas flow rate (X_4) constant. It is clear that CO_2 capture efficiency increased from 79.6 to 99.9% with a decreased temperature of 50-10 °C at buffer concentration of 14 g/L as shown in Fig. 3 (a), and sodium removal increased from 3.7 to 27.6% with the same decreased temperature as shown in Fig. 3 (b); this can be explained by the optimum temperature range of forward Solvay's reactions, where optimum temperature is about room temperature [14], while reversible reactions come about temperature range of 38–60 ºC [15]. The CO_2 capture efficiency increased from 71.7 to 97.4% with an increased buffer concentration of 2–14 g/L at temperature of 30 ºC, and sodium removal increased from 18.4 to 33.6% with an increased buffer concentration of 8-20 g/L. For the chemical reaction, when carbon dioxide dissolves in brine, it forms carbonic acid (H_2CO_3), which dissociates into bicarbonate ions at high pH level [16] and, consequently the CO_2 capture efficiency and sodium removal will increase. However, the effect of buffer concentration on pH level is proportional to certain limit as shown in Fig. 3 (a); increasing buffer concentration more than 20g/L does not seem to add much to the CO_2 capture efficiency.

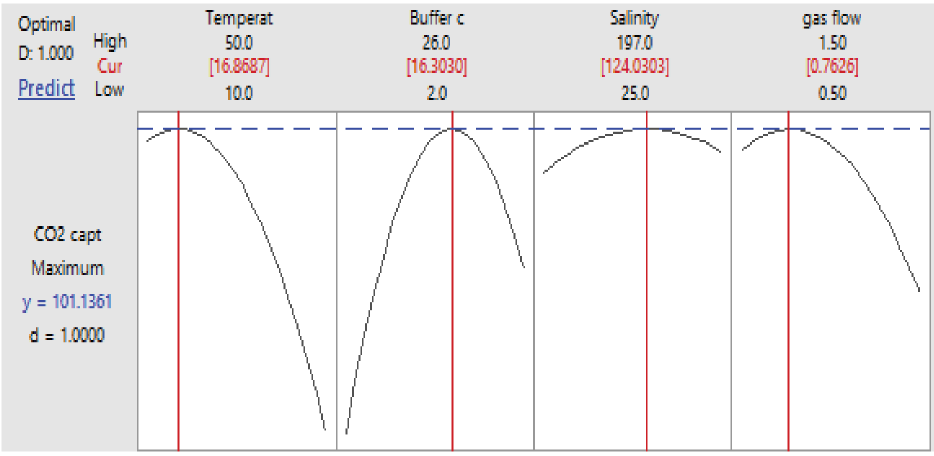
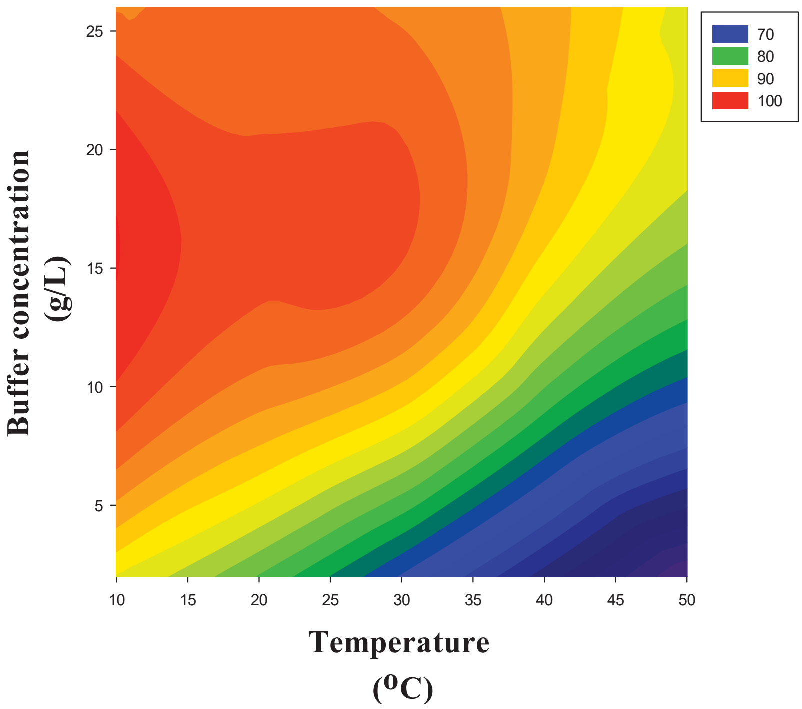
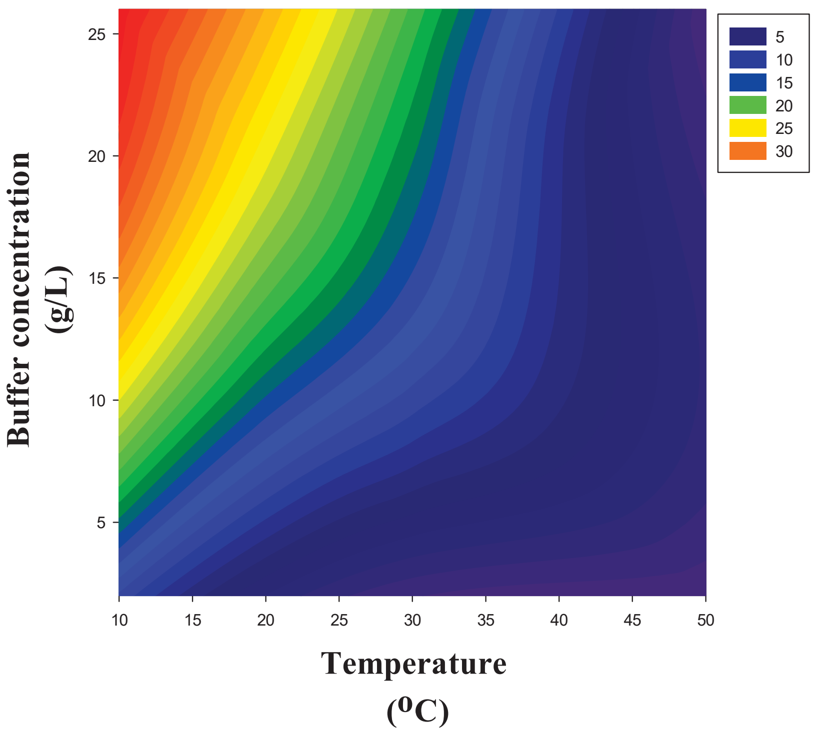


Fig. 2 The optimum temperature (X_1), buffer concentration (X_2), salinity (X_3) and gas flow rate (X_4) to have maximum CO_2 capture efficiency

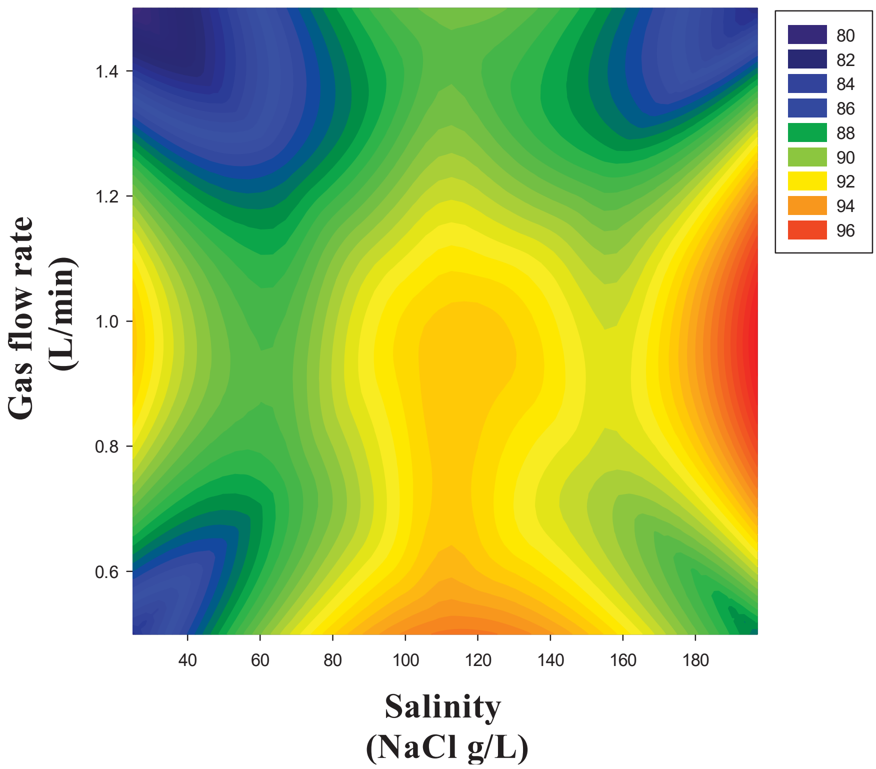


(a)

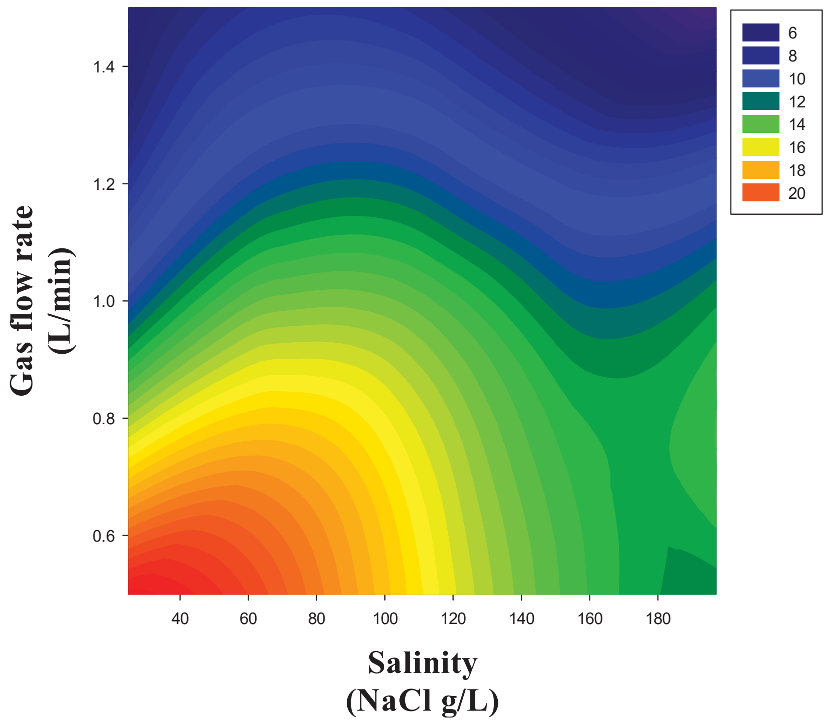


(b)

Fig. 3 Interaction effect of temperature and buffer concentration on (a) CO_2 capture efficiency and (b) sodium removal



(a)



(b)

Fig. 4 Interaction effect of salinity (X_3) and gas flow rate (X_4) on (a) CO_2 capture efficiency and (b) sodium removal

F. Effect of Salinity and Gas Flow Rate

Two dimensional (2D) contour plots shown in Figs. 4 (a) and (b) visualize the interaction effects of salinity (X_3) and gas flow rate (X_4) on CO_2 capture efficiency and sodium removal, respectively. It should be noted that when the response surface for the effect of two factors was plotted the other factors were constant. Fig. 4 (a) shows that there is an increase in the CO_2 capture efficiency with increased gas flow rate up to 0.8 to 1 L/min. This increase can be explained by increasing the reaction rate according to the increase in CO_2 loading to the reactor; however, increasing the gas flow rate more than 1 L/min, decreases the CO_2 capture efficiency due to the decrease in the gas residence time in the reactor, which accordingly decline the reaction [17]. The CO_2 capture efficiency increases with increasing salinity up to 120 g/L; where increasing the salinity increases the reaction rate and accordingly, increases the CO_2 capture efficiency. However, at higher salinity (more than 130 g NaCl), a decline in CO_2 capture efficiency occurred; this inhibitory response can be explained by declining the pH level, which accordingly decreases the reaction rate. Fig. 4 (b) shows the effects of salinity (X_3) and gas flow rate (X_4) on sodium removal. Sodium removal increases significantly as the gas flow rate decreases and decreases slightly as the salinity increases more than 130g NaCl/L; as discussed earlier, increasing the gas residence time increases the contact time between reactants, while decreasing pH level hinders the reaction.

IV. Conclusion

The CO_2 capture and sodium removal can be adequately described by second order polynomial models developed using composite central designs. Model validation using experimental data demonstrated that there is an excellent agreement between model predictions and experimental data. A temperature of 17 ºC, buffer concentration of 1.6%, salinity of 124 g/L and gas flow rate of 0.76 L/min maximized CO_2 capture efficiency to 99%. A 35% sodium removal has been achieved using real reject brine at optimum temperature, buffer concentration and gas flow rate.

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