



Recovery of sulfur dioxide from gas mixture in packed bed column

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Abstract

Recovery of SO₂ from SO₂/Air mixture into aqueous sodium carbonate solution was performed using packed bed column in pilot scale. The aim of the study was to improve the recovery efficiency of this process, to find the proper operation conditions in the packed bed column, and to contribute to the application of this process in the industry. The SO₂-recovery efficiency was measured while the gas mixture rate, the inlet SO₂ concentration, sodium carbonate solution concentration, liquid temperature, and the liquid hold-up were changed according to experimental design. Computer program (Statgraphics/Experimental Design) was used to estimate the fitted linear model of SO₂-recovery efficiency (η) in terms of (G , C_{SO_2} , $C_{Na_2CO_3}$, T , and V_L), and the economic aspects of the process. The accuracy of η model is $\pm 2.38\%$. The linear model of η was adequate, and the operating parameters were significant, while the interactions were negligible.

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Keywords: SO₂ absorption; Aqueous sodium carbonate solution; Backed bed column.

1. Introduction

Thermal power plants are major sources of air pollutants, the large amount of SO₂ emitted from the combustion of coal leads to severe air pollution and results in great harm to people's living, production and health, and has to be controlled. Several desulfurization schemes, such as fuel pretreatment, concurrent burning and adsorption, and flue gas post treatment, have been proposed [1]. The flue gas desulphurization (FGD) is the main technology used, and a great number of FGD methods have been developed, such as dry-, semidry- and wet-processes, among which the wet-process is more widely applied, due to lower operating cost and more stable operation, and limestone- or lime-based scrubbing is used. However, many of these FGD methods are not regenerative and pollute the environment. The traditional SO₂ absorption process requires a large column packed with various packings [2] or spray tower [3], rotating-stream tray scrubber [4], *etc.* Since the mass transfer efficiency is poor, the size of columns is large, leading to high capital and operating costs. Therefore, researches have been focused on developing regenerative processes and equipment with process intensification for absorption of SO₂ [5-7]. SO₂-removal by absorption into sodium citrate buffer solution is generally considered as a fast, safe, green, and economical method with the advantages of non-toxic reagent with negligible losses, simple process, no fouling problem, and low oxidation of SO₂. In the process, SO₂ in flue gas is absorbed by aqueous sodium citrate solution, and dissolved SO₂ is subsequently recovered from the solution by steam

stripping or other regenerative method, or the resulting absorbent solution react with H₂S to obtain sulphur, which can be separated by flotation [8-10]. The desulfurization of SO₂ with absorption and desorption in sodium citrate buffer solution. SO₂ is a major constituent in air pollution. SO₂ which affects the environment by no. of ways like acid rain, corruptions and severe damage to the health. SO₂ causes a wide variety of health and environmental impacts because of the way it reacts with other substances in the air. Particularly sensitive groups include people with asthma who are active outdoors and children, the elderly, and people with heart or lung disease. Removal of SO₂ from gas mixtures by chemical absorption is one of the most important processes for environmental protection. A great number of gas desulphurization methods have been developed throughout the world [11,12] The most widespread processes are limestone-, or lime-based scrubbing [11-14], beside these, magnesium hydroxide [13, 14], sodium hydroxide [15] and organic solvents are also used as absorbents [16, 17].

A lot of research has been focused on regenerative processes [1, 8, 16-26]. The SO₂-removal by aqueous sodium carbonate solution received a considerable attention [18-28].

When sulfur dioxide in gas phase is absorbed into aqueous sodium carbonate solution, the following overall reaction between the dissolved sulfur dioxide and aqueous sodium carbonate solution may take place in the liquid phase [29], and can be expressed as:



Some FGD systems go a step further and oxidize the Na₂SO₃ to produce marketable Na₂SO₄ (Sodium Sulphate) [30, 31]:



The removal of sulfur dioxide from gas mixture by aqueous carbonate solutions is an important industrial absorption process for control of air pollution. Further, this chemical absorption process is of theoretical interest, since it is one in which the absorption is accompanied by a chemical reaction and the subsequent desorption of the volatile reaction product. However, there have been a few studies on the mechanism of chemical absorption of sulfur dioxide into aqueous carbonate solution [32].

2. Experimental work

2.1 Experimental apparatuses

The main equipment of the experimental apparatus as shown in Figure 1 is the packed bed column (1), and its heat exchanger (2), the size to gather of 1.5 m height and 0.10 m in diameter. The main complementary apparatus and pipe lines are as follows: Temperature gage (3), discharge point (4), digital pH- meter (5), compressed air in (6), sulfur dioxide gas in (7), air rotameter (8), sulfur dioxide rotameter (9), mixing chamber (10), gas mixture in (11), aqueous Na₂CO₃ solution in (12), condenser (13), liquid recycle to top of the column (14), cold water out from condenser (16), cold water in to condenser (17), gas mixture (18), droplet collector (19), SO₂ gas analyzer (20), gas mixture out to atmosphere (21), water to heat exchanger from thermostat (22), water from heat exchanger to thermostat (23), solid Na₂CO₃ (24), process water (25), mixing tank to prepare aqueous Na₂CO₃ solution (26), feeding pump of aqueous Na₂CO₃ solution to packed column (27), aqueous Na₂CO₃ solution feeding tank (28), thermostat water bath (29), (Na₂SO₄+Na₂SO₃, and CO₂) in solution from bottom of column (30), (Na₂SO₄+Na₂SO₃, and CO₂) solution tank (31) CO₂ gas out (32), discharge pump of (Na₂SO₄ + Na₂SO₃) solution (33) solution for further processing plant to oxidized Na₂SO₃ to Na₂SO₄, and producing dry powder Na₂SO₄ (34).

2.2 Operating parameters

The preliminary experiments were carried out to absorb of SO₂ gas from gas mixture into aqueous Na₂CO₃ solution in pilot scale packed column by using experimental apparatus as shown in Figure 1 to find the proper operating parameters could be used in this work. Operating variable parameters were as follows:

- * Gas mixture flow rate (*G*): 25-35 m³/h.
- * SO₂ gas inlet concentration (*C*_{SO₂}): 500-3500 ppm.
- * Aqueous Na₂CO₃ solution concentration (*C*_{Na₂CO₃}): 5-35 wt %.
- * Liquid temperature (*T*): 20-50 °C.
- * Liquid holdup in the column (*V*_L): 0.02-0.04 m³.

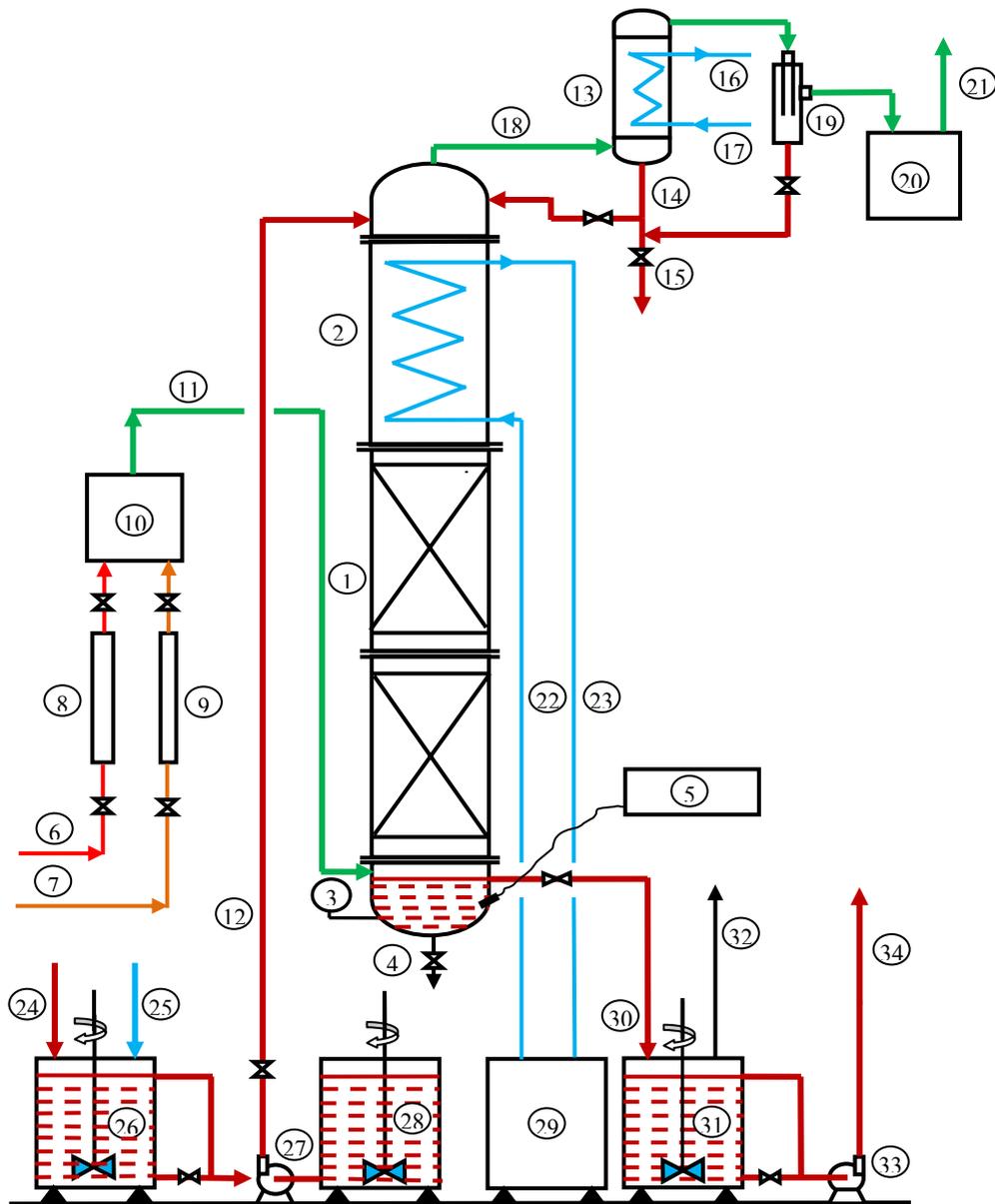


Figure 1. Schematic diagram of the experimental apparatus for SO_2 gas absorption from gas mixture into aqueous Na_2CO_3 solution in packed column.

2.3 Recovery of SO_2 from SO_2 /Air gas mixture

Experiments of absorption of SO_2 gas from SO_2 /Air gas mixture into aqueous Na_2CO_3 solution have been carried out using the mentioned various operating parameters by using experimental apparatus as shown in Figure 1, according to experimental design plan seen in Table 1. The gas mixture (11) enters the packed column (1) from lower part, while Na_2CO_3 solution from solution tank (28) by feeding pump (27) enters the upper part of the column. The heat exchanger (2) is maintain the desired liquid temperature constant in packed column (1) during the all experiments runs by circulation water [22, 23] from and to thermostat water bath (29) through the heat exchanger (2). Gas mixture from the top of column enters the condenser (13) to condense any liquid drops with it by cold water (17). The liquid (14) returns back to upper part of the column as recycle liquid or drain out from drain line (15). The gas from condenser pass through droplet collector (19), to separate any liquid drops in it. The SO_2 gas concentration in dry outlet gas mixture is measured by SO_2 -gas analyzer (20), then the gas mixture to atmosphere air with few traces of SO_2 gas (21). In the column, the liquid temperature measured by temperature gage (3), while the pH of the liquid is measured by digital pH-meter (5). The resultant reaction product (30) contains (Na_2SO_4 + little amount of Na_2SO_3 + CO_2) in solution from downer part of column sent to solution tank (31). The CO_2 gas (32) evolves to atmosphere air. In stirred tank (31) there

is (Na_2SO_4 and little amount of Na_2SO_3) solution, it is difficult to separate them from each other, for that reason transferred by the discharge pump (33) to further processing to oxidize Na_2SO_3 , and to producing dry powder of Na_2SO_4 .

Table 1. Experimental design plan for absorption of SO_2 into aqueous Na_2CO_3 solution.

Run No.	Gas mixture flow rate (G) (m^3/h)	SO_2 gas inlet concentration (C_{SO_2}) (ppm)	Na_2CO_3 solution concentration ($C_{\text{Na}_2\text{CO}_3}$) (wt %)	Liquid temperature (T) ($^\circ\text{C}$)	Liquid holdup in the column (V_L) (m^3)
1	35	500	5	50	0.04
2	35	500	5	20	0.04
3	25	3500	5	20	0.02
4	25	500	5	50	0.04
5	35	3500	5	20	0.02
6	35	3500	35	20	0.02
7	30	2000	20	35	0.03
8	35	500	35	50	0.04
9	35	3500	35	50	0.02
10	25	3500	35	20	0.04
11	30	2000	20	35	0.03
12	25	500	5	20	0.04
13	25	3500	35	50	0.02
14	35	3500	35	20	0.04
15	25	3500	35	50	0.04
16	25	500	35	50	0.02
17	25	500	35	20	0.02
18	35	500	35	20	0.04
19	35	3500	5	50	0.04
20	35	500	35	50	0.02
21	25	3500	5	20	0.04
22	25	3500	5	50	0.04
23	25	500	35	20	0.04
24	35	3500	35	50	0.04
25	35	3500	5	50	0.02
26	35	500	5	50	0.02
27	35	500	5	20	0.02
28	35	3500	5	20	0.04
29	25	3500	5	50	0.02
30	25	3500	35	20	0.02
31	25	500	35	50	0.04
32	25	500	5	50	0.02
33	30	2000	20	35	0.03
34	35	500	35	20	0.02
35	25	500	5	20	0.02

3. Results and discussion

The absorption of SO_2 gas from SO_2/Air gas mixture into aqueous sodium carbonate (Na_2CO_3) solution were carried out according to experimental design plan in Table 1 with the variation of gas mixture flow rate (G), SO_2 gas inlet concentration (C_{SO_2}), aqueous Na_2CO_3 solution concentration ($C_{\text{Na}_2\text{CO}_3}$), experimental liquid temperature (T) and liquid holdup in the column (V_L).

3.1 Definition of recovery efficiency

The SO_2 Recovery efficiency (η) was defined as [8, 33, 34]:

$$\eta = \frac{C_{SO_2,in} - C_{SO_2,out}}{C_{SO_2,in}} \times 100 \quad (3)$$

where, η = Recovery efficiency in (%), $C_{SO_2, in}$ = SO₂ gas inlet concentration in gas mixture in (ppm), $C_{SO_2, out}$ = SO₂ gas outlet concentration in gas mixture in (ppm).

Recovery efficiency (η) was calculated by using equation (3). The Recovery efficiency of SO₂ gas from gas mixture SO₂/Air was in the range of $\eta = 70.10 - 95.60$ %. The results of Recovery efficiency are summarized in Table 2.

Table 2. Data base of experimental design and results of SO₂ gas recovery from gas mixture (SO₂ /Air) into aqueous Na₂CO₃ solution.

Run No.	Gas mixture flow rate (G) (m ³ /h)	SO ₂ gas inlet concentration (C _{SO2}) (ppm)	Na ₂ CO ₃ solution concentration (C _{Na2CO3}) (wt %)	Liquid temperature (T) (°C)	Liquid holdup in the column (V _L) (m ³)	Recovery efficiency (η) (%)
1	35	500	5	50	0.04	70.90
2	35	500	5	20	0.04	73.30
3	25	3500	5	20	0.02	91.30
4	25	500	5	50	0.04	72.70
5	35	3500	5	20	0.02	86.40
6	35	3500	35	20	0.02	88.45
7	30	2000	20	35	0.03	85.35
8	35	500	35	50	0.04	75.76
9	35	3500	35	50	0.02	86.10
10	25	3500	35	20	0.04	95.60
11	30	2000	20	35	0.03	83.40
12	25	500	5	20	0.04	78.90
13	25	3500	35	50	0.02	93.30
14	35	3500	35	20	0.04	90.20
15	25	3500	35	50	0.04	91.10
16	25	500	35	50	0.02	83.85
17	25	500	35	20	0.02	83.10
18	35	500	35	20	0.04	80.60
19	35	3500	5	50	0.04	90.10
20	35	500	35	50	0.02	72.20
21	25	3500	5	20	0.04	93.50
22	25	3500	5	50	0.04	90.10
23	25	500	35	20	0.04	85.30
24	35	3500	35	50	0.04	89.80
25	35	3500	5	50	0.02	85.60
26	35	500	5	50	0.02	70.10
27	35	500	5	20	0.02	76.20
28	35	3500	5	20	0.04	89.20
29	25	3500	5	50	0.02	86.90
30	25	3500	35	20	0.02	95.10
31	25	500	35	50	0.04	84.80
32	25	500	5	50	0.02	70.30
33	30	2000	20	35	0.03	82.70
34	35	500	35	20	0.02	80.15
35	25	500	5	20	0.02	76.20
35	25	500	5	20	0.02	76.20

The effects of operating parameters on Recovery efficiency of SO₂ gas from SO₂/Air gas mixture could be seen as in the following Figures:

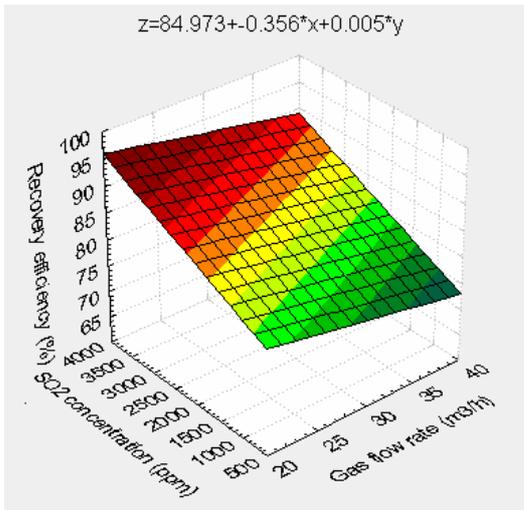


Figure 2. Effect of SO₂ gas concentration, and gas flow rate on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

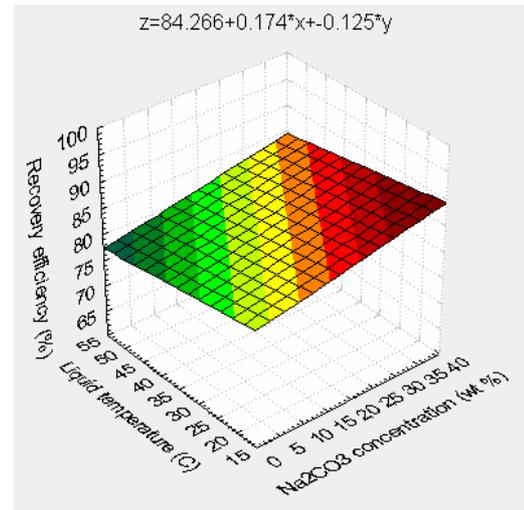


Figure 3. Effect of liquid temperature, and Na₂CO₃ concentration on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

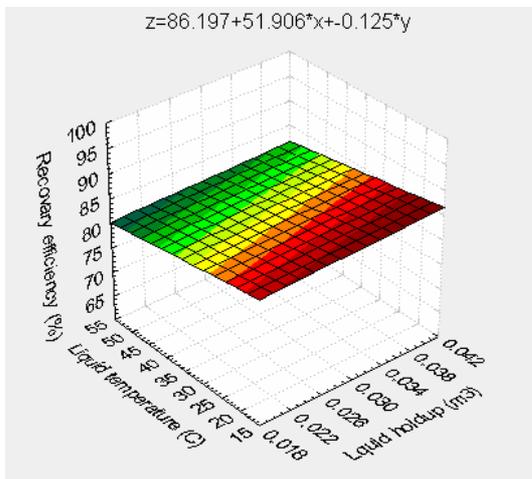


Figure 4. Effect of liquid temperature, and liquid holdup on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

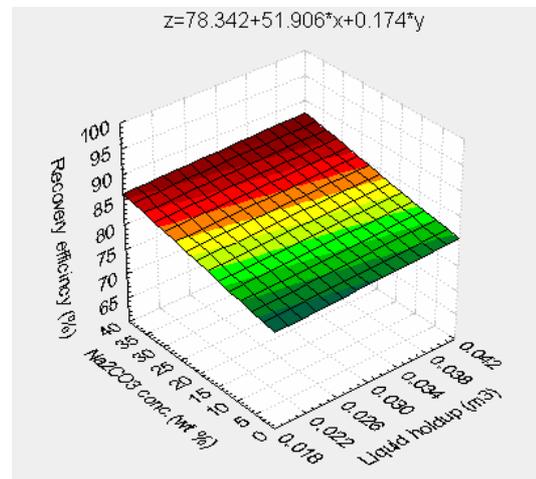


Figure 5. Effect of Na₂CO₃ concentration, and liquid holdup on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

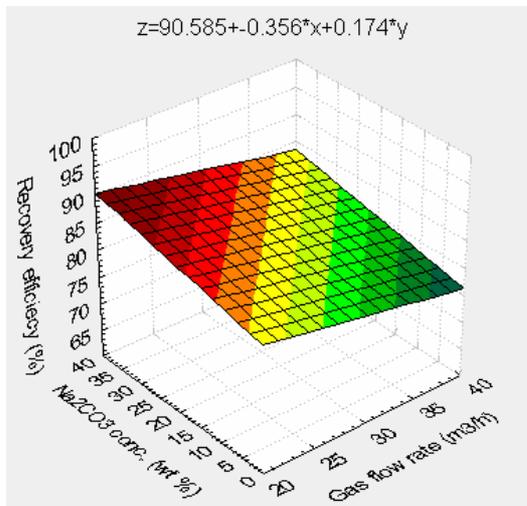


Figure 6. Effect of Na₂CO₃ concentration, and gas flow rate on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

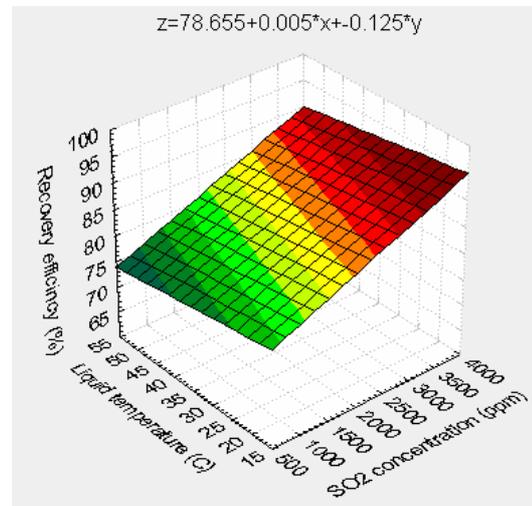


Figure 7. Effect of liquid temperature, and SO₂ concentration on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

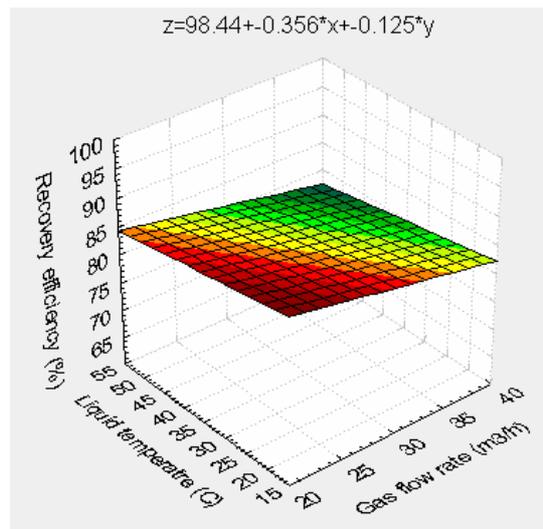


Figure 8. Effect of liquid temperature, and gas flow rate on Recovery efficiency of SO₂ gas from SO₂/Air mixture.

From Figures 2-8, the Recovery efficiency of SO₂ gas from SO₂/Air gas mixture (η) is increased with the increase in inlet SO₂ gas concentration ($C_{SO_2,in}$), aqueous Na₂CO₃ solution ($C_{Na_2CO_3}$), and liquid holdup (V_L), and decreased with an increase in gas mixture flow rate (G), and liquid temperature (T). The biggest effect of operating parameters on Recovery efficiency was G , and smallest one was (V_L).

3.2 Correlation model of Recovery efficiency

Computer program (Statgraphics/Experimental Design) was used to estimate the fitted linear model of Recovery efficiency (η) for SO₂ gas recovery from SO₂/air gas mixture into aqueous Na₂CO₃ solution in packed bed column in terms of operating parameters: G , C_{SO_2} , $C_{Na_2CO_3}$, T , and V_L . The output shows the results of fitting a multiple linear regression model to describe the relationship between Recovery efficiency and 5 independent variables.

The equation of the fitted model is:

$$\eta = 85.623 - 0.419G + 0.004C_{SO_2} + 0.154C_{Na_2CO_3} - 0.104T + 83.156V_L \quad (4)$$

Since the P-value in the ANOVA Table 3 is less than 0.05, there is a statistically significant relationship between the variables at the 95.0% confidence level.

Table 3. ANOVA.

Parameter	Estimate	Standard	T	P-value
		Error	Statistic	
Constant	85.6227	3.12356	27.4119	0.0000
Gas flow rate	- 0.418688	0.084302	- 4.96652	0.0000
Liquid holdup	83.1562	42.151	1.97282	0.0581
Liquid temperature	- 0.103937	0.0281007	- 3.69876	0.0009
Na ₂ CO ₃ concentration	0.153562	0.0281007	5.46473	0.0000
SO ₂ concentration	0.00434146	0.000281007	15.4497	0.0000

The R-Squared statistic indicates that the model as fitted explains 91.4654% of the variability in Recovery efficiency. The adjusted R-squared statistic, which is more suitable for comparing models with different numbers of independent variables, is 89.994%. The standard error of the estimate shows the standard deviation of the residuals to be 2.38442. This value can be used to construct prediction limits for new observations by selecting the Reports option from the text menu. The mean absolute error (MAE) of 1.72457 is the average value of the residuals. The Durbin-Watson (DW) statistic tests the residuals to determine if there is any significant correlation based on the order in which they occur in

your data file. Since the P-value is greater than 0.05, there is no indication of serial autocorrelation in the residuals at the 95.0% confidence level.

In determining whether the model can be simplified, notice that the highest P-value on the independent variables is 0.0581, belonging to Liquid holdup. Since the P-value is greater or equal to 0.05, that term is not statistically significant at the 95.0% or higher confidence level. Consequently, you should consider removing Liquid holdup from the model.

The validity range for the model in equation (4) is:

$20 \leq T \leq 50$ °C; $25 \leq G \leq 35$ m³/h; $0.02 \leq V_L \leq 0.04$ m³; $5 \leq C_{Na_2CO_3} \leq 35$ wt %; $500 \leq C_{SO_2} \leq 3500$ ppm.

The accuracy of the η model is ± 2.38 %.

The linear model in equation (4) is adequate, and the operating parameters were significant and were in ordered of $C_{SO_2} > C_{Na_2CO_3} > G > T > V_L$. The interactions of operating parameters were negligible.

The Pareto Chart of Recovery efficiency (η) of SO₂ gas from SO₂/Air gas mixture could be seen in Figure 9. The main effects of operating parameters on Recovery efficiency of SO₂ gas from gas mixer SO₂/Air is seen in Figure 10. The observed and predicted Recovery efficiency of SO₂ gas from SO₂/Air gas mixture represents in Figure 11.

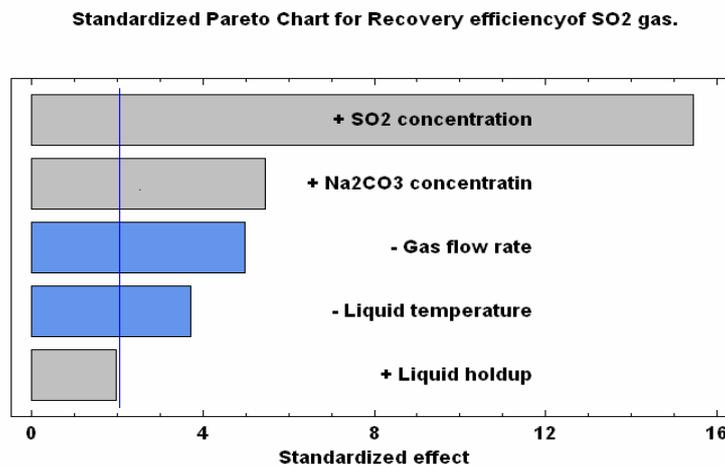


Figure 9. Effects of operating parameters on Recovery efficiency of SO₂ gas.

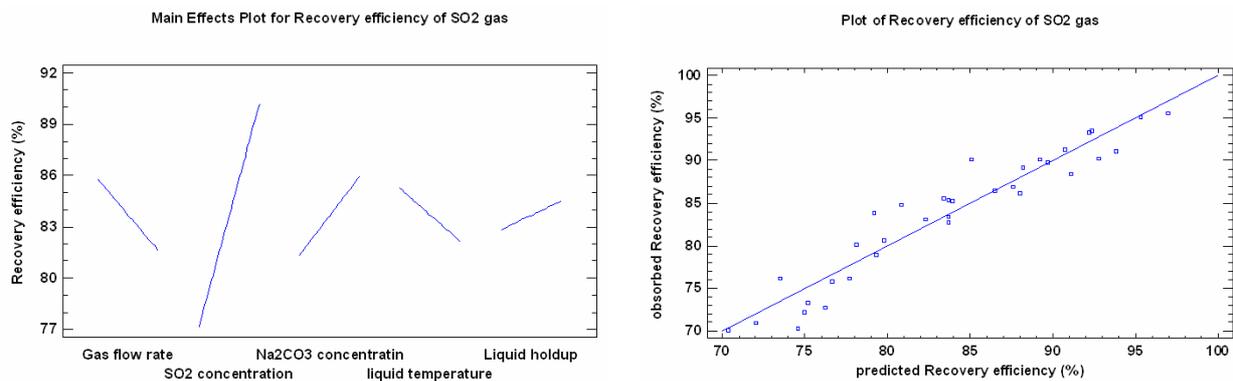


Figure 10. Main effects of operating parameters on Recovery efficiency of SO₂ gas.

Figure 11. Observed vs. predicted Recovery efficiency of SO₂ gas.

3.3 Optimal response

The optimum operating parameters for present work were obtained using the computer program to analyze the experimental results. The goal of optimizing was to maximize the Recovery efficiency (η) of SO₂ gas from SO₂/Air gas mixture. The results of optimizing were summarized in Table 4.

Table 4. Optimum operating parameters and optimum Recovery efficiency of SO₂ gas.

Operating parameters	Low value	High value	Optimum value
Gas mixture flow rate (m ³ /h):	25	35	25
SO ₂ gas inlet conc. (ppm):	500	3500	3500
Na ₂ CO ₃ solution conc. (wt %):	5	35	35
Liquid temperature (°C):	20	50	20
Liquid hold up in column (m ³):	0.02	0.04	0.04
Recovery efficiency of SO ₂ gas from SO ₂ /Air gas mixture (%):			96.97

4. Conclusions

- Recovery of SO₂ gas from SO₂/Air gas mixture into Na₂CO₃ solution was carried out in pilot scale packed bed column. The Recovery efficiency (η) of SO₂ gas was measured at various operating conditions (G , C_{SO_2} , $C_{Na_2CO_3}$, T , and V_L) according to experimental design. The measured Recovery efficiency was in the range of $\eta = 70.10 - 95.60$ %. The η could be improved and increases by increasing in the C_{SO_2} , $C_{Na_2CO_3}$, and V_L and with decreasing of G , and T .
- A computer program (Statgraphics/Experimental Design) was used to estimate the linear fitted model of Recovery efficiency (η) in terms of operating conditions (G , C_{SO_2} , $C_{Na_2CO_3}$, T , and V_L). The linear fitted model of η was adequate, and operating parameters were significant, while the interactions were negligible. The accuracy of Recovery efficiency model is ± 2.38 %.
- Using the same computer program the optimum operating conditions were obtained with values of $G = 25$ m³/h, $C_{SO_2} = 3500$ ppm, $C_{Na_2CO_3} = 35$ wt %, $T = 20$ °C, and $V_L = 0.04$ m³. The optimum Recovery efficiency (η) was in value of 96.97 %.
- On the base of results of measured Recovery efficiency, we conclude to scaling up the size of the pilot plant used in present work by 3-4 times to commercial size plant and using the optimum operating parameters obtained in future work.

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