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Kinetic Studies of a Novel Chemical Reaction in a Selective Separation of SO_2 from Refinery Cat-cracker Stack Flue Gas

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ABSTRACT

In the view of increased requirements on absorption rates and loading capacity for SO_2 gas absorption solvents, the main purpose of this study is evaluating the reaction kinetics of absorption of sulfur dioxide gas in a mixture of flue gas with amine solution without CO_2 gas being touched. In order to acquire this target, a selective amine-based absorber exploited and hence a high amount of SO_2 gas absorbed in the scrubbing process. The process was aimed to decrease the quantity of sulfur dioxide gas in cat-cracker exhaust. In this regard, the pilot plant was designed and set up in the Research Center of Petroleum University of Technology, containing both absorption and desorption stages. In this study, the effect of parameters which can improve considerably the kinetic reaction of the whole process investigated. We evaluated the process using different levels of pH, desorption temperature, concentration of inlet SO_2 gas, the absorber solution, and flow rate of the absorbing solution. We identified, thereafter, the optimum conditions for the absorption process and then reaction rate of sulfur dioxide gas absorption under optimum conditions. The results showed that an increase in temperature and the concentrations of inlet SO_2 gas resulted in a higher reaction rate. More than 99 %, the process efficiency achieved based on varieties of the parameters we showed that SO_2 gas was absorbed enough below standard level of 500 ppm from the inlet flue gas which is this success has much more merits for industrial sector purposes.

INTRODUCTION

Sulfur dioxide is one of the main pollution gases which is mostly released by power plants fossil-based, or chemical and petrochemical industries [1]. It should be added that such combustions as Furnace and chlorofluorocarbons (CFCs) has high possibility to produce proportion of SO_2 emissions from oil and gas industries [2]. Coal burning in power plants surely release SO_2 to the environment meanwhile most categories of coals include sulfur inside. Nowadays, the consequences of such toxic discharges might result in acid rain which has been a great concern [3]. In last decades, most of the developed countries have approved strong rules and procedures to limit SO_2 releases into the atmosphere [4].

implementing new techniques which present reformative absorbers with low construction costs has brought in new efficient process designs and conditions [5,6]. Scrubbers are commonly most appropriate equipment for gas absorption due to transfer mass occurrence between gas states and liquid in a counter current flow over numerous packing materials [7]. In this regards, packed towers are well-known to eliminate industrial gaseous

pollutants encountering present environmental standards [8]. Many advancement absorption techniques have been conducted between which scrubbing is probable to play a essential role in the development of SO_2 capturing process progresses [8–10]. Furthermore, packed scrubbers have mostly participated as manipulating equipment for elimination of flue gas pollutions [11]. Scholars are enthusiastic to examine more practical technique to achieve a permissible rate of SO_2 emission to the environment with consideration of global standard restrictions [12]. Amine scrubbers typically purify gaseous airstreams from pollutants [13,14]. Enormous wetted surface make wide mass transfer which is an affecting advantage of the pack scrubbers [15,16]. It is worth mentioning that corrosion resistance and low energy utilization are another merit which cause these packed columns appropriate [4].

In order to optimize process circumstances of SO_2 absorption in wet flue gas desulfurization, the proportion of operation's affecting parameters including solvent flow rate, SO_2 inflowing concentration, desorption temperature, and pH value are computed and set as variables. There is a need of doing experiments to estimate the best process conditions. The experimental design technique same as Taguchi provides an exact examination of process features. This technique has shown to be helpful when it is both reducing the number of experimentations and specifying controllable and uncontrollable parameters which can be considered as time efficient and dynamic [17–20]. SO_2 absorption performance was investigated by Taguchi's orthogonal array (OA) examination to determine suitable pH value for this pilot plant.

This study is focused on the reduction of SO_2 gas with a selective amino compound solvent. Leading the experiments on the pilot plant in the research center of Abadan Institute of Technology (AIT), the attained data are discussed in the article. It proved to be a strong solvent; it has brought innovations as SO_2 absorber in this research. The rest of appear is provided as follows. In the section 2, materials and methods are explained. Section 3, results and corresponding discussion are represented and conclusion are shown in section 4.

Theories (Reaction pathway)

Every experimenter needs proper planning to obtain adequate, relevant and reliable data so that one can infer the science behind the observed phenomenon. Therefore, such experimentation usually ends well before the number of experiments reaches a double digit. The procedure of experiment is based on a specific class of amine absorbents which optimality stabilities the ability to absorb and regenerate sulfur dioxide. In water solution, dissolved SO_2 undergoes reversible hydration and ionization to produce bisulfite and sulfite according to the following equations.

$$SO_2(g) \leftrightarrow SO_2(aq) \tag{1}$$

$$SO_2(aq) + H_2O(L) \leftrightarrow H_2SO_3(L) \tag{2}$$

$$H_2SO_3(L) \leftrightarrow HSO_3^- + H^+ \tag{3}$$

Adding amine, to the water increases the quantity of SO_2 dissolved. According to equation (5-4), the buffer drives the above equilibria to the right by reacting with the hydrogen ions to form ammonium salts. The overall reaction indicates that as the concentration of SO_2 in the feed gas increases, the equilibrium moves to the right, i.e. the quantity of SO_2 dissolved in the rich solvent increases. Thus, the scrubbing of more concentrated gas streams requires a less than proportional increase in solvent circulation rate. Since the gas volume, and therefore the gas side equipment, remains constant, a relatively small total cost increase is caused by an increase in feed SO_2 concentration.

$$NR1R2R3NR4R5 + H^+ \leftrightarrow NR1R2R3NR4R5H^+ \tag{4}$$
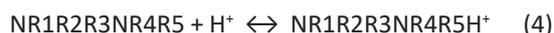
EXPERIMENTAL

The amine that was used is a selective absorber that absorbs SO2 selectively. CO2 gas, a competitor contamination with SO2 in flue gases, does not interfere with the absorption process. In the process, SO_2 is eliminated at ppm levels, while CO2 remains untouched. The physical properties of the absorbent are as the following: viscosity = 0.002 N.s/m^2, Absorption density = 1.0643 kg/l, and Boiling point = C. The applied absorbent consists of 30l of distilled water, 2.25 kg of sulfuric acid with 98% concentration, and 4 kg of an ionic-amine additive for selective absorption. Molecular weight of the defined additive was found 120 g/mole. The applied absorbent has a great capacity for absorption along with a long durability, as obtained in experimental tests that were done

in a 3month period during the experiments, no differences were exhibited in the absorbent efficiency. Another important feature of the solvent is that its flow rates as well as co.

concentration in the process are lower than the usual absorbers while the performance is the same, if not better. It needs not to be pure or anhydrous and in industrial scale performs perfectly up to the highest conventional environmental standards. Traditional selective removal processes such as CANSOLV use 20 % (wt/wt) of solvent; however, our solution contains only 8.16 % (wt/wt) of absorber. This reduction in solvent consumption is an innovative aspect that makes the system stronger and more effective. The cost of applied amine (5$ a pound) is lower than the amine absorbers used in conventional processes (12$ dollars a pound). The production of SO_3 during the process results in dilute salt solutions in the process.

The pilot plant was set up in the Research Center of Petroleum University of Technology. It was set so that no gas leakages existed. Via injecting the nitrogen gas to the whole pilot and letting it remain until reaching an appropriate pressure in both columns, all leakages were found with a foam test. They were all specified and blocked afterward.

The pilot plant is made up of a packed column containing metal grid packing. The construction of the plant was designed specifically to improve absorption. A temperature's range of 50–72^{0}C and an atmospheric pressure was looked after during the scrubbing process. This two steps process is performed in various ranges of pH and different flow rates of flue gas.

Prior to commencing the main experiments, the tank of the plant, with a capacity of 60l, was filled with distilled water (DW), as it contains no chlorine. The plant was run at high temperatures with DW to have it entirely cleaned for our real runs. At this level, the plant run was done five times with DW to insure of the actual experiments' correctness. Process flow diagram (PFD) of the SO_2 capturing is illustrated in Figure 1.

To prepare the absorber solution, 4 kg of the prepared amino compound absorber were added to 30l of water. The concentration of the solution was 87 g/l or 13 vol% with a pH of 11. Colle et al., (2005) discussed that the best amount of pH for SO_2 absorption in range of 5-6. Next, sulfuric acid was added to helps SO_2 absorption based on its features. Thus, 2.25l of sulfuric acid, with a concentration of 98 % were added to the absorber solution to reach the desired pH. The pH of the overall solution then reached 6.01.

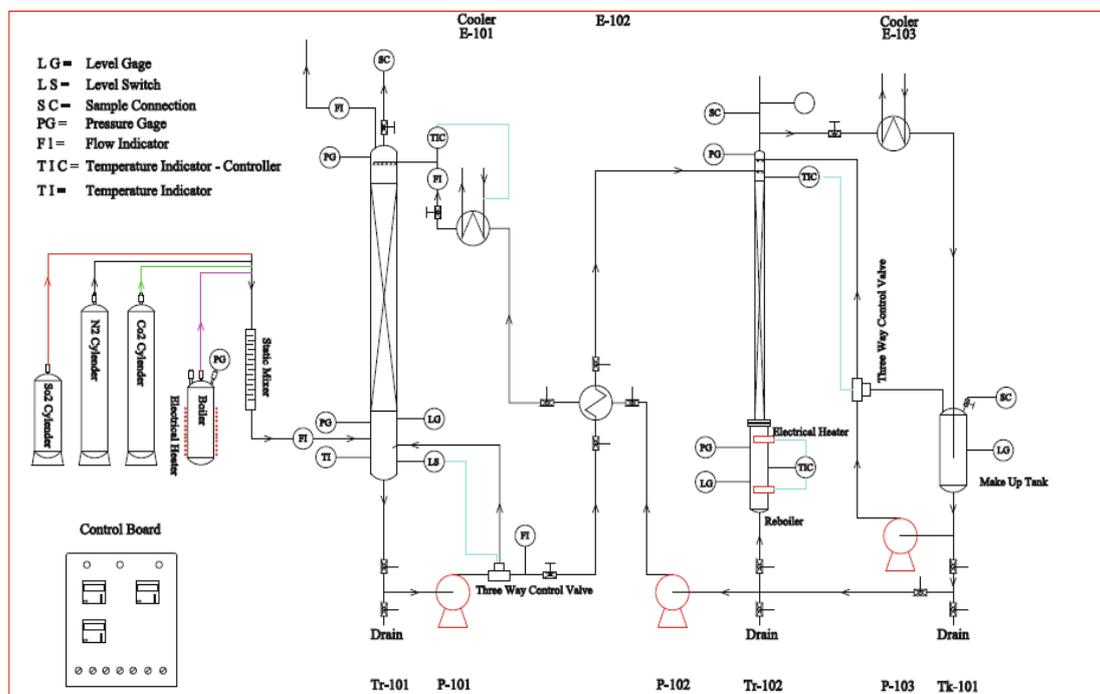A valve is supplied in the path between

Figure 1. Process flow diagram (PFD) of the SO_2 capturing pilot plant

deception tower and the storage tank, by opening which, desorption tower is filled with solvent. Two 2000 (w) elements are placed in desorption tower which heat up the solvent to the set point 120 °C. Each element is controlled by a thermo set which are placed on the controller box. When the temperature rose to 120 °C, the point set on the controller board, desorption tower pressure reaches 1.1 (bar). Desorption tower pressure can be adjusted by a valve placed in the solvent steam exit path. At the top of this tower a thermocouple is placed to measure the temperature and set the top temperature at a desire point. A pump is placed below the supply tank controlled by a thermo controller. This pump has two duties: 1) holds the top at a constant temperature by spraying sorbent to desorption tower, 2) provide compensatory absorber and keep the sorbet at a constant level. These two duties are done by a solenoid valve, which is commanded by the thermometer described above. As described before, pump 2 is placed between the absorption and desorption tower. This pump duty is to transmit the solvent from desorption tower to absorption tower. Because of the solvent temperature is about 120 °C, its temperature reduces in three ways; a cooler is designed after stripper tower whose flow can be set by a flow meter. Then solvent enters the sell & tube heat exchanger to reduce its temperature and increase the flow return to disruption temperature. Before pump 2 sprays solvent in the absorption tower on packing, flow passes through an adjustable flow meter, with division on flow meter as 0.4 m^3/hr.

After setting up the pilot plant, nitrogen is inserted into the pilot and all fitting was carefully checked in order not to have any leakage. The gas is inserted from the entrance at the bottom of the absorption tower. After the gage showed the required pressure, all fittings are tested by foam to check if there is a leakage.

Before the first experiment by using solvent, the storage tank filled with distilled water (because of having no Cl) up to 40 (lit) and the first experiment run was done at 120 °C to completely clear all paths of solvent to prevent any external event. The liquid flow passes a flow meter containing a setting knob with a precision of 25 ml/min before entering the absorption tower. After regulating the flow and passing the cooling water, the flow enters the absorption column and the absorber solution sprinkles on the packing.

RESULTS AND DISCUSSIONS

To obtain the optimum point, many parameters are influential, each of which should be considered in certain conditions. The parameters affecting SO_2 absorption studied in this paper include: the temperature of the desorption column, pH of the absorber solution, concentration of SO_2 in gas inlet of the pilot, and flow rate of the absorbing solution. The following levels were investigated in our study on the pilot:

• Desorption bottom Temperature (°C): 100, 110, and 120
• Absorbent pH: 2, 4, and 6
• Gas flow rate (m^3/hr): 2.5, 3, and 3.5
• Inlet SO_2 Concentration (ppm): 4500, 6500, and 8500

Considering all the above amounts we should have done $3^4 = 81$ experiments. To reduce the number of experiments we used Taguchi experimental design method. Therefore, 27 experiments were needed to gain optimum conditions of SO_2 absorption. Based on the number of parameters and levels, Taguchi method illustrates the number of experiments that should be done in specified conditions. For the designed

Table 1. The orthogonal array with design factors

Experiment Number	Absorbent PH	Inlet SO_2 Concentration (ppm)	Gas Flow Rate (m^3/hr)	Desorption Bottom Temperature (°C)
1	2	4500	2.5	100
2	2	6500	3	110
3	2	8500	3.5	120
4	4	4500	3	120
5	4	6500	3.5	100
6	4	8500	2.5	110
7	6	4500	3.5	110
8	6	6500	2.5	120
9	6	8500	3	100

experiment, we considered four parameters, three levels for each. Therefore, Taguchi method determines L9 array for this experiment. Table 1 illustrates orthogonal array with design factors.

Temperature: The selected temperature ranges above are chosen since desorption occurred in temperatures higher than the absorbent boiling point (107°C) which enhances the process of desorption: increasing temperature, increasing desorption. The maximum temperature of the desorption tower prevents the outlet trapping is 127 °C, furthermore, the references for selectivity of absorption by amine absorbent which are listed below include the mentioned range [22–24].

pH: Regarding the absorbent structure, the best absorption occurred in the mentioned range just as the pre-handled tests yielded that the absorption specifications bring about the best revival in this range. In addition, due to the presence of more than 80 % of water in the absorbent, the pH in the best SO_2 absorption falls between 2 and 4 which is resulted from the pH intimacy of solution of this gas in water [25].
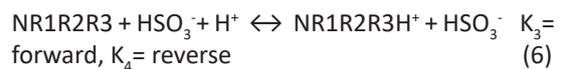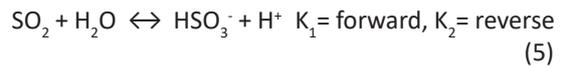
Flow rate: Gas to liquid ratio is usually almost 1,000 in industrial scale. In this study, an approach was made to this ratio using lower ratios to find the exact selection for best effectiveness of the process. Although lower numbers (100, 150, 400) are not quite industrial ratios, a higher solvent flow rate system guaranteed the absorption process.

Concentration: The input SO_2 was simulated on the basis of this gas concentration in catalytic cracker unit of Abadan oil refinery stack exhaust. Hence, the dealing range is about 6,000 ppm.

Kinetics between SO_2 and amine solution

The chemistry of SO_2 reactions in the liquid phase in amine solutions is complex and not fully understood. The absorption process in amine comprises several reactions.

The process reactions are illustrated as following:

$$SO_2 + H_2O \leftrightarrow HSO_3^- + H^+ \quad K_1= \text{forward}, K_2= \text{reverse} \tag{5}$$

$$NR1R2R3 + HSO_3^- + H^+ \leftrightarrow NR1R2R3H^+ + HSO_3^- \quad K_3= \text{forward}, K_4= \text{reverse} \tag{6}$$

amine-based absorber solution in injected to the column from the top in opposite side and the output of combustion enters at the packed absorption column from the lower part. Next, the output of the column is transferred to the desorption tower.

$$R_{SO2}= K_1C_{SO2} - K_2C_H^+ \times C_{HSO3}^- \tag{7}$$

From the first reaction (1), is produced and used in second one (2). Thus, is considered as the intermediate product based on it is not stable.

$$R_{CH}^+ = K_1C_{SO2}. -.(K_2C_H^+ \times C_{HSO3}^-) - (K_3{}_{NR123} \times C_{CH}^+ \times C_{HSO3}^-) + (K_4C_{NR123H}^+ \times C_{HSO3}^-) \tag{8}$$

The last term of mentioned correlation is omitted based on availability of reviving column for absorber. In addition, the outlet of the absorption column passes in the desorption

Table 2. Kinetic experimental setup

Operational conditions used for kinetic experiments	
Parameter	**Range**
SO_2 concentration in feed gas (ppm)	2500-8500
Temperature (K)	313-333
Gas flow rate ($\frac{m^3}{hr}$)	2.5
PH of absorbent	4

Table 3. Outlet concentration of SO_2 in various inlet concentration of SO_2 and constant PH and gas flow rate.

Temperature (k)	Outlet concentration of SO_2 (ppm)			
	2500ppm SO_2	4500ppm SO_2	6500ppm SO_2	8500ppm SO_2
313	1220	2300	3023	4100
323	960	1500	2035	3500
333	930	1400	1950	3100

Table 4. Initial rate of reaction in various inlet concentration of SO_2 and constant PH and gas flow rate.

Temperature (k)	Initial rate of reaction ($\frac{mol}{m^3.hr}$)			
	2500ppm SO_2	4500ppm SO_2	6500ppm SO_2	8500ppm SO_2
313	4.34	7.46	11.79	14.92
323	5.38	10.48	15.6	17.47
333	5.66	11.18	16.41	19.48

tower in which the absorber is revived. Therefore, reaction 2 is led forward to transform amine to NR1R2R3H+NR1R2R3 and next is attained by the reviving column.

According to the correlation (2), can be obtained as following:

$$C_{H+} = \frac{K_1 C_{SO_2}}{K_2 \times C_{HSO3-} + K_3 C_{NK123} \times C_{HSO3-}} \quad (9)$$

Substituting, correlation (6) in (4):

$$R_{SO_2} = K_1 C_{SO_2} + \frac{K_1 K_2 C_{SO_2}}{K_2 + K_3 C_{NR123}} \rightarrow R_{SO_2} = \frac{K_1 K_3 C_{SO_2} C_{NR123}}{K_2 + K_3 C_{NR123}} \quad (10)$$

after simplifying, the reaction rate for this mechanism is $r=k[SO_2]^n$

The reaction kinetics can be determined from the absorption rate measurement. It was used throughout this study to evaluate effect of operational parameters such as temperature and initial concentration of SO_2, as well as to determine the reaction kinetic. For this purpose, a simulated flue gas containing CO_2, N_2, O_2 and SO_2 was led into the column with a flow rate of 2.5 (m^3/hr). The process was terminated when the temperature and pH had stabilized at the optimum level. The reaction rate is given by where is the reactor volume. Molar flow rate is extracted from ideal gas law (PV=nRT) and then initial rate of reaction is calculated using r computation. as given in Table 2.

Results for concentration of SO_2 in sweet gas are shown in Table 3. when the PH of absorbent and gas flow rate are held constant.

As shown by Figure 2, 3, and 4 a first-order reaction with respect to the SO_2 concentration was obtained.

In general, temperature is an important parameter influencing reaction kinetics. For SO_2 absorption kinetics, the reaction rate constant normally is based on the Arrhenius expression:

$$K=K_0 e^{-Ea/RT} = Ln\,K = -\frac{Ea}{RT} + Ln\,K0 \quad (11)$$
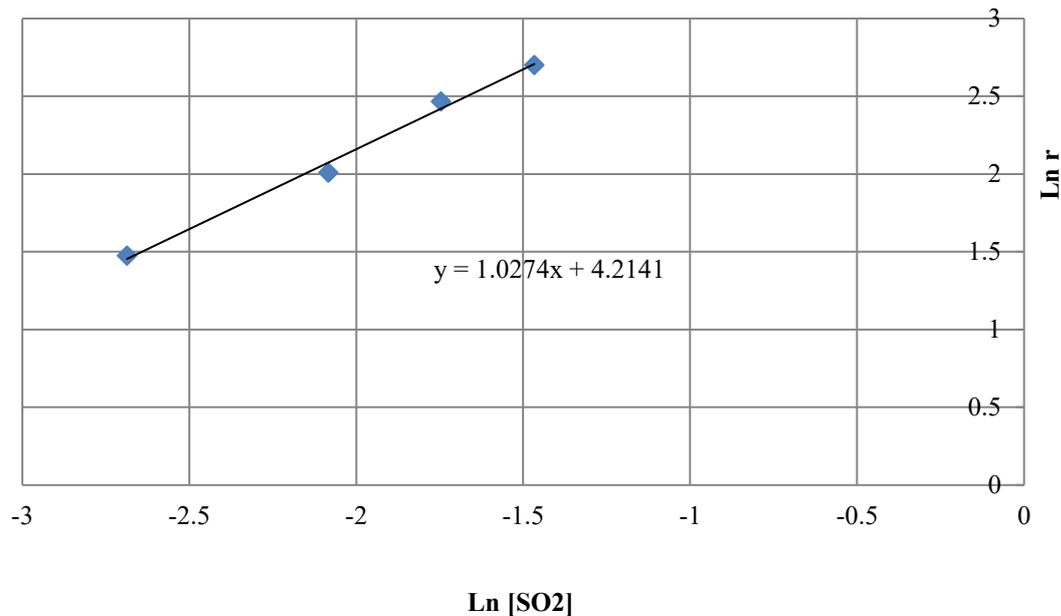
where: K= chemical reaction rate, K_0 = pre-

$$y = 1.0274x + 4.2141$$

Ln [SO2]

Figure 2. Ln r vs. Ln [SO_2] at 40 °C, gas flow rate of 2.5 m^3/hr and pH of 4

$$y = 0.9822x + 4.4282$$
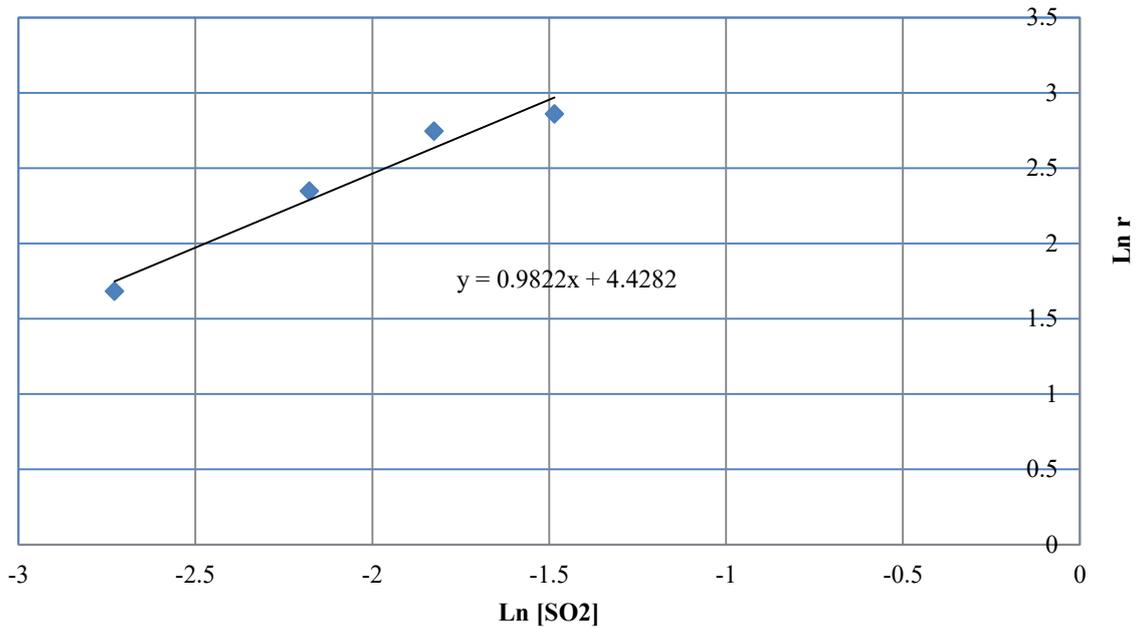
Figure 3. Ln r vs. Ln [SO$_2$] at 50 °C, gas flow rate of 2.5 m³/hr and pH of 4
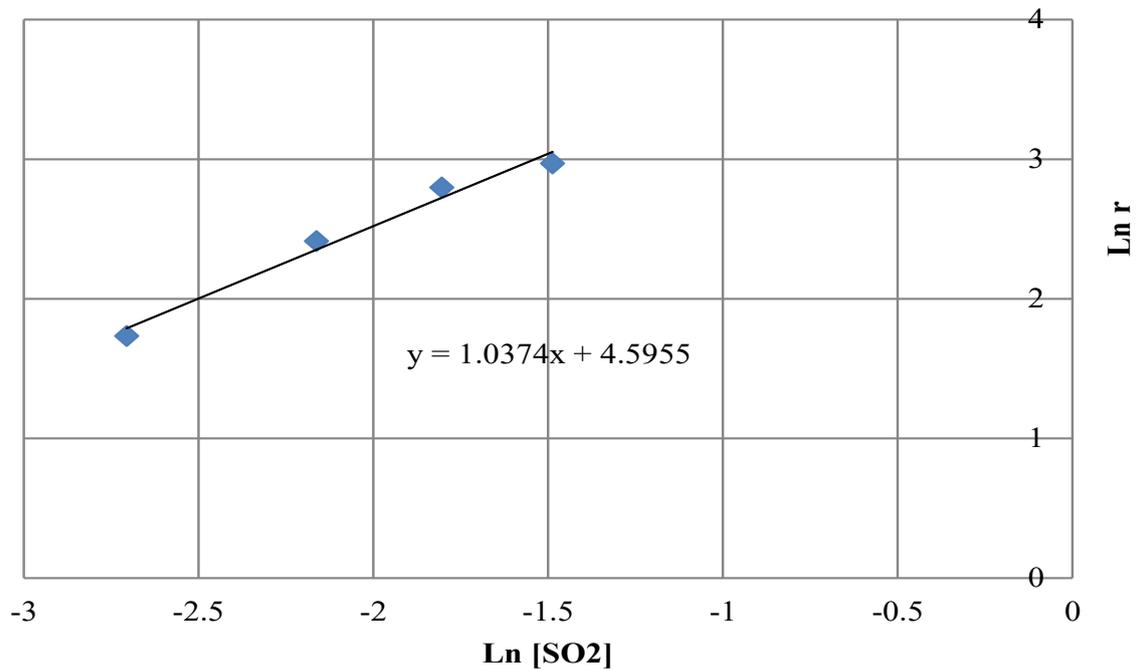

$$y = 1.0374x + 4.5955$$

Figure 4. Ln r vs. Ln [SO$_2$] at 50 °C, gas flow rate of 2.5 m³/hr and pH of 4

exponential constant, E_a = activation energy (J/mol), R = gas constant (8.314 J/mol.k),

T = temperature (K)

Parameters of the kinetic model such as k_0 and E_a is extracted by taking the best-fit straight line between ln K and 1/T. Values estimated for activation energy and pre-exponential constant are respectively 17.32 KJ and 5.1×10^4. From the figure 5. 9, an Arrhenius expression is defined as follows:
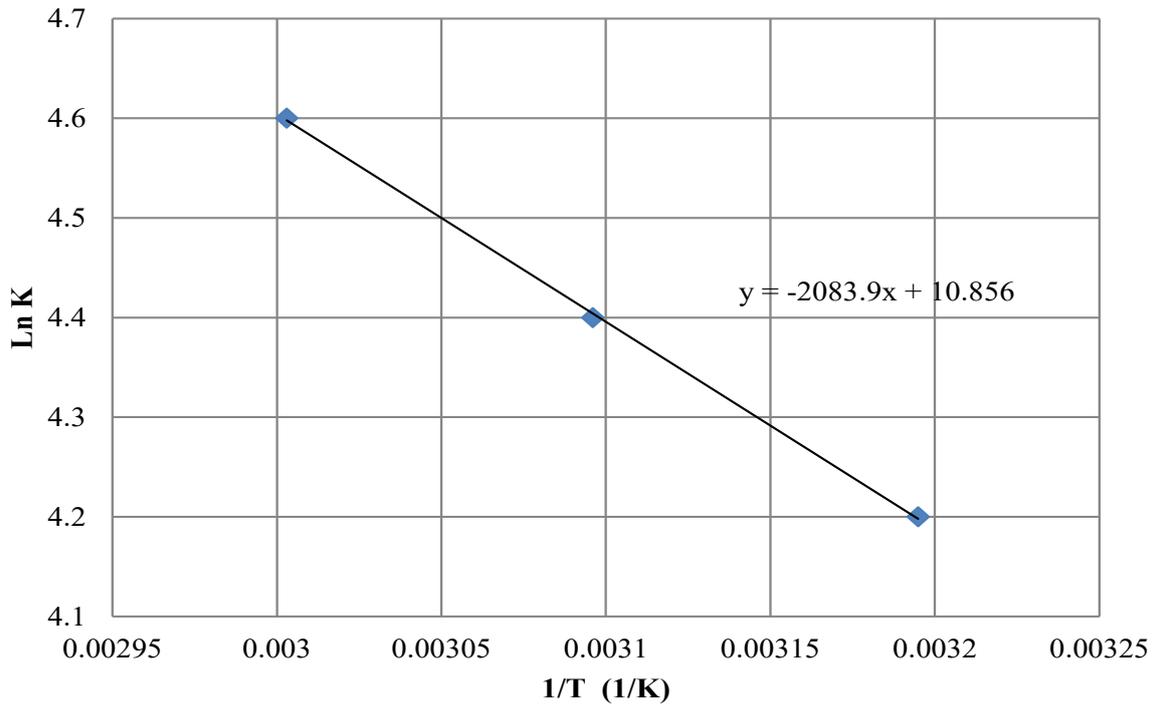
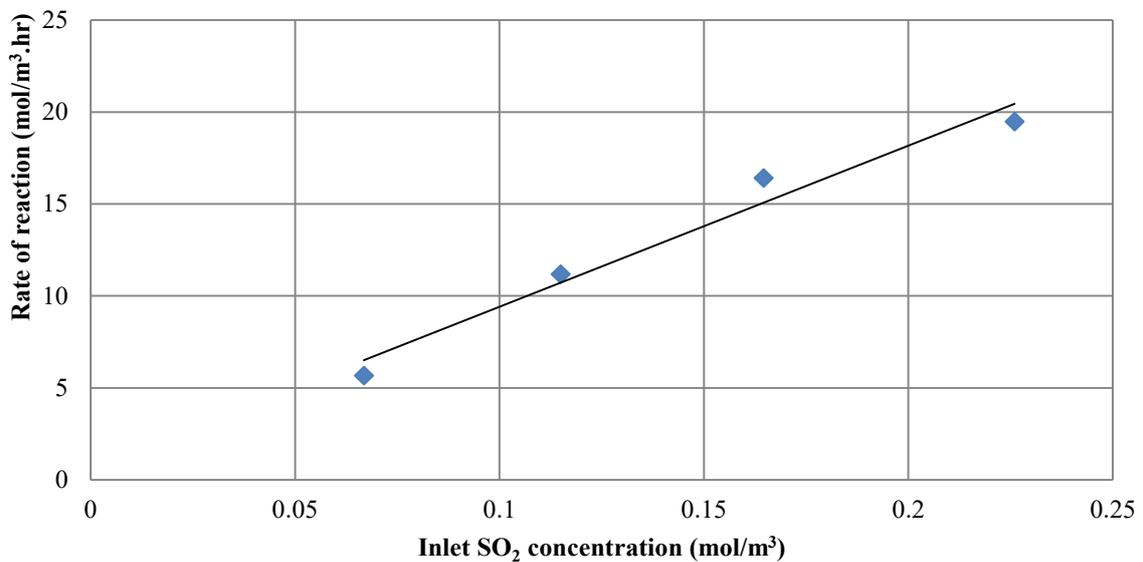Figure 5. Relationship between Ln K and 1/T

Figure 6. Effect of inlet SO$_2$ concentration on the rate of reaction

$$K= 5.1 \times 10^4 \exp\left(\frac{-2084}{T}\right) \quad (12)$$

Effect of SO$_2$ inlet concentration on SO$_2$ reaction rate

Experiments were carried out at 60 °C, the gas flow rate of 2.5 (m^3/hr) (and PH of absorbent was 4

to test the effect of SO$_2$ inlet concentration on SO$_2$ reaction rate. When the SO$_2$ inlet concentration was increased from 2500 ppm to 8500 ppm, the SO$_2$ reaction rate increased from 5.66 (mol/m^3.hr) to 19.48 (mol/m^3.hr) . The experimental results shown in Figure 6. indicate that the reaction
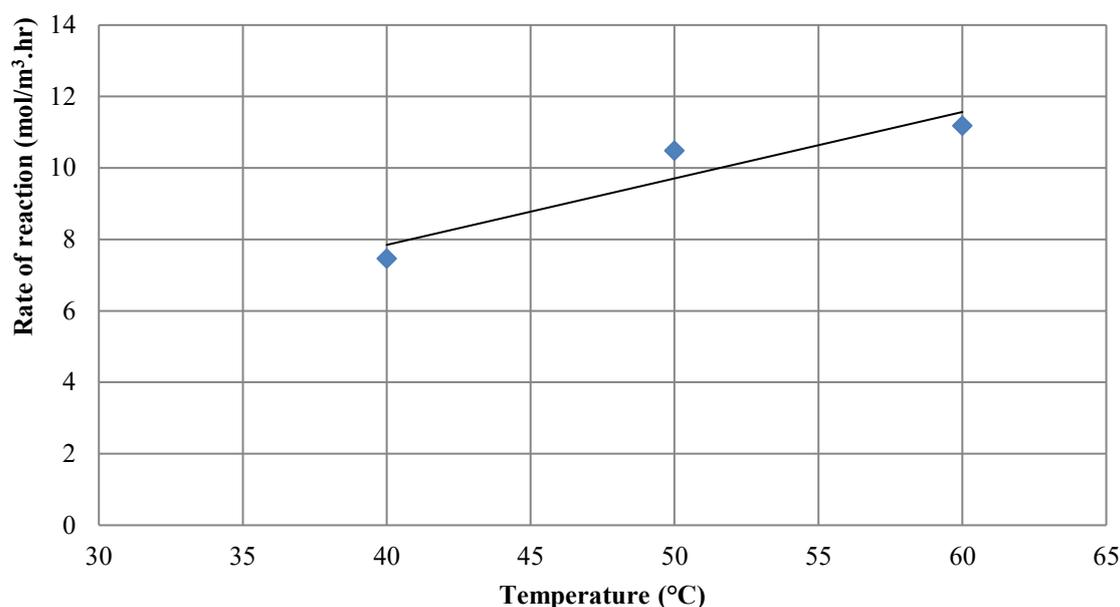
Figure 7. Effect of inlet SO_2 concentration on the rate of reaction

rate of SO_2 into amine solution increases with increasing SO_2 concentration in the inlet gas. The relationship between the reaction rate of SO_2 and its concentration in the inlet gas is nearly linear when the other parameters are held constant.

Effect of temperature on SO_2 reaction rate

The experimental results detecting the effect of temperature on SO_2 absorption rate were shown in Figure 7. During the runs, the SO_2 concentration was 4500, and the gas flow rate was 2.5 (. The reaction rate of SO_2 increases with temperature. The solubility of SO_2 in the liquid solution becomes lower with the increase of temperature. On the other hand, the diffusing capacity of SO_2 molecules in the liquid solution enhances by the increase of temperature, and the mass transfer of SO_2 in the solution increases with the increase of temperature. Moreover, the increasing of the temperature also accelerates the reaction due to the increasing quantity of activated molecular.

CONCLUSION

Design of equipment for gas absorption with chemical reactions requires adequate information on mass transfer characteristics as well as on intrinsic kinetics of the chemical reactions of the process. The conclusions can be drawn from the experimental results as follows:

The present investigation on absorption of

sulfur dioxide by amine solution reveals that the process kinetics is governed by a first order reaction. The pre-exponential constant was found 5.1×10^4, and the activation energy was calculated to be 17.32 KJ. We also showed that under these experimental conditions, the absorption rate of SO_2 into amine solution increases with the increasing SO_2 concentration in the inlet gas from 2500 ppm to 8500 ppm and with the increasing of temperature from 40°C to 60°C.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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