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## Experimental and theoretical study of fluidized bed for $SO_2$ recovery as sulfur from effluent gases from sulfur production unit

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### ABSTRACT

This paper studies the operating conditions for  $SO_2$  recovery in one fluidized bed reactor which is filled with aluminum oxide catalysts. The performance of the bed is analyzed experimentally and theoretically in this work. Sweet gas production is the major purpose of this process. Experiments are conducted for sour gases with different amounts of concentration. The influence of bed height, bed width, diameter of aluminum oxide particles, operating conditions such as temperature and pressure and also superficial velocity of inlet gas are measured and discussed. Likewise, comparison between obtained experimental data for bed height, temperature, sorbent aluminum content and the results of model is done. No relevant differences are observed between experimental data and theoretical model. Results show the increase in the amount of recovery with increasing sorbent aluminum content and bed height. The relation between recovery and operating temperature is linear, relatively. Finally, this paper provides clear insights into the effect of operating conditions of fluidized bed filled with aluminum oxide upon recovery. © 2014 Trade Science Inc. - INDIA

### KEYWORDS

Recovery;  
Experimental and theoretical  
study;  
Fluidized bed;  
Sulfur.

### INTRODUCTION

The use of multistage, fluidized beds of continuously recycled, coal-based metal oxidation reveals economically and technically attractive for both adsorption of stack gas  $SO_2$  and sequential conversion to elemental sulfur<sup>[1]</sup>. In literatures, conceptual design details and economic factors are surveyed for the treatment of power plant or oil refinery  $SO_2$  effluents with by-product sulfur recovery. The preliminary requirements for

removing  $SO_2$  from gas streams simulating boiler flue gas or incinerated Claus plant effluents have been assessed in continuous pilot plant. Continuous removal of the  $SO_2$  in a slipstream from an oil-fired power boiler was achieved with a total metal oxidation depth of 16 inches, which expands to about double on fluidization at special flue gas rates<sup>[2]</sup>. Natural gas or inexpensive hydrogen from coal is the basic reductant for conversion of the adsorbed sulfuric acid to sulfur<sup>[4]</sup>.

A dry fluidized activated carbon process is being

developed at the continuous pilot stage for recovery of  $SO_2$  from flue gases as elemental sulfur. The  $SO_2$  component is removed from the gases as sulfuric acid on metal oxidation by sorption, catalytic oxygenation and hydrolysis. This is accomplished in a fluidized bed sorbent cooled by water sprays at 150 to 3000 degree Fahrenheit. An important development is the direct conversion of sorbent sulfuric acid to elemental sulfur by reaction with internally produced hydrogen sulfide. Conceptual design compare favorably with published Figures on alternate measures to control  $SO_2$  emissions<sup>[5,6]</sup>.

The  $SO_2$  recovery from effluent gases of sulfur production unit depends on fluidized bed performance. In addition, conventional technologies require high level of energy. The superiority of this work is introducing a feasible and viable fluidized bed which is filled with aluminum oxide. So, in this paper, a proposed fluidized bed is considered as a main unit of  $SO_2$  recovery from effluent gases of sulfur production unit which is shown in Figure 1. The effluent gases from sulfur production unit should be passed through fluidized bed as the sulfur recovery unit to produce sweet gas and  $SO_2$  component. Performance of this fluidized bed is investigated experimentally and also theoretically.

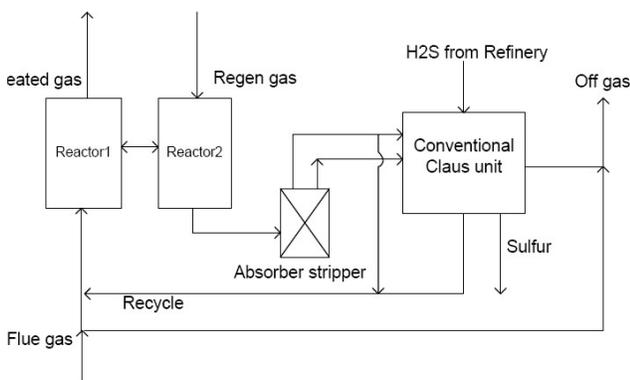


Figure 1 : A schematic of  $SO_2$  recovery process

## MATERIALS AND METHOD

### Pilot plant experiences

Pilot plant sets up to verify the mathematical model. Two fluidized bed reactors are applied for  $SO_2$  ad-

sorption process in this work. The reactor contains Aluminum oxide catalytic bed which has 0.07 m width and 0.2 m height. A Schematic of this bed is shown in Figure 2.

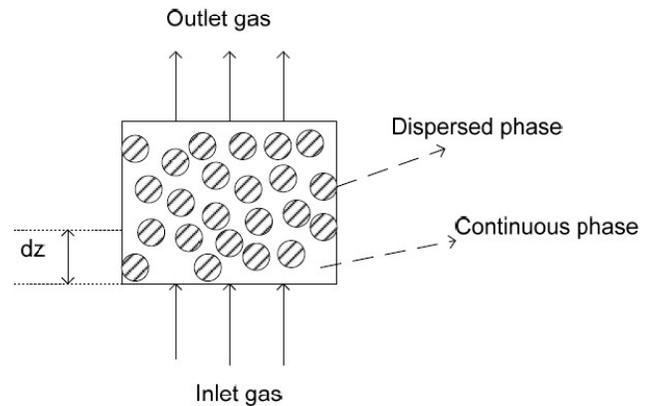


Figure 2 : Schematic of fluidized bed

The flue gas enters the reactor with a determined concentration of  $SO_2$ . The reacts with catalyst in reactor and the amount of  $SO_2$  concentration in gas decreases passing through the bed, gradually.

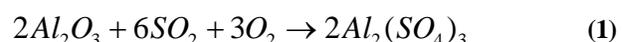
The concentration of  $SO_2$  in the feed gas stream and the outlet stream is measured by the standard method using literature. In addition, bed temperature, pressure and inlet gas velocity is measured in each experiment.

Catalysts adsorb 95 percentage of  $SO_2$  content of the gas stream approximately when the operating temperature of reactor is about 700 F to 800 F<sup>[7]</sup>.

Hydrogen, methane or carbon monoxide is used to activate the catalyst bed in regeneration process. So, adsorbed  $SO_2$  on catalysts is removed. The operating range of temperature in regeneration process is near the operating temperature in adsorption process and is about 750 F to 850 F.

### Process chemistry

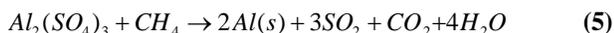
Gas stream enters the reactor which contains Aluminum catalytic bed. Aluminum reacts into Aluminum reacts with the oxygen which is in the gas stream into the Aluminum oxide. Then Aluminum oxide reacts with  $SO_2$  and  $O_2$  or with  $SO_3$  to produce aluminum sulfate according to reactions 1 and 2.



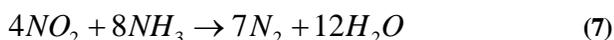
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Activation energy that is required for reactions 1 is 22 kJ/mol in reduction process Aluminum sulfate changes into the metal Aluminum and SO<sub>2</sub> releases. Mixture of Hydrogen and Carbon monoxide or light hydrocarbons can be used instead of hydrogen in reduction process. Related reactions 3, 4 and 5 show reduction process of bed.



Also, Aluminum oxide and Aluminum sulfate vanishes nitrogen oxide compounds selectively in fluidized bed as catalysts. Therefore, NH<sub>4</sub> is injected into the flue gas stream before entering the fluidized bed and then reacts with Nitrogen oxide compounds in fluidized bed in contact with catalysts as are shown in reactions 6 and 7.



### MATHEMATICAL MODEL

A mathematical model is developed to obtain the concentration profile in the fluidized bed reactor through the time. Two-phase theory is used to describe the adsorption process of SO<sub>2</sub> in the fluidized bed. Two distinct phases, one dispersed phase and one continuous phase, are recognized from the bed based on this theory<sup>[8]</sup>.

The theory is expressed as following assumptions:

All gas in excess of that is required just to fluidize the bed passes through the bed in the bubble form.

Continuous phase is at minimum fluidization conditions so, the gas relative velocity and solid remains unchanged.

The gas passing through the dispersed phase is close to plug flow regime.

In this predicted model, the fluidized bed is represented by n-compartments in a series, and then material balances are written with the mentioned assumptions for each compartment.

Following equations 8, 9, 10, 11 and 12 show model parameters associated with the proposed two phase model.

The amount of gas exchanges between phases<sup>[9]</sup>.

$$Q = 0.75U_{mf}\pi D_b^2 \quad (8)$$

$$\dot{m} = 0.75\rho_g U_{mf}\pi D_b^2 \quad (9)$$

The minimum fluidization velocity<sup>[10]</sup>.

$$U_{mf} = \frac{\mu}{d_p \rho_g} \left\{ \left[ 33.72 + 0.0408 \frac{d_p^3 \rho_g (\rho_p - \rho_g) g}{\mu^2} \right]^{0.5} - 33.7 \right\} \quad (10)$$

Bubble diameter at height of H<sup>[11]</sup>.

$$d_b = 1.4\rho_p d_p \left( \frac{U}{U_{mf}} \right) L + d_{bo} \quad (11)$$

The diameter of bubble at the bed entrance point<sup>[12]</sup>.

$$d_{bo} = \frac{\left( \frac{6G}{\pi} \right)^{0.4}}{g^{0.2}} \quad (12)$$

Where  $G$  is defined as Equation 13:

$$G = \frac{U - U_{mf}}{n} \quad (13)$$

Equation number 14 calculates gas diffusion mass transfer coefficient from bubble to the continuous phase<sup>[13]</sup>.

$$K_G = 0.975 D_{AB}^{0.5} \left( \frac{g}{d_b} \right)^{1/4} \quad (14)$$

### Mass balance in the dispersed phase

The material balance is written below according to the Figure 2. Also, as shown the Figure 3 illustrate the proposed plant. There are two main mechanisms used in the equations. The bulk mass transfer because of the fluid velocity and the molecular mass transfer because of the SO<sub>2</sub> concentration gradient between phases are occurred. Therefore, equation 15 and 16 show the mass balance in the dispersed phase.

$$\dot{m}_{bdis} \Big|_z - \dot{m}_{bdis} \Big|_{z+\Delta z} - \dot{m}_{mdis} = m_{acc} \quad (15)$$

$$A(U - U_{mf})C_d \Big|_z - A(U - U_{mf})C_d \Big|_{z+\Delta z} - N(Q + K_G A_b)(C_d - C_c) = V_d \frac{\partial C_d}{\partial t} \quad (16)$$

### Mass balance in the continuous phase

Equation 17 and 18 show the material balance in

the continuous phase. Where  $r$  is a symbol of the reaction rate and is determined according to Equation 1 which is a first-order reaction rate formula. The rate of reaction depends on the  $\text{SO}_2$  concentration.

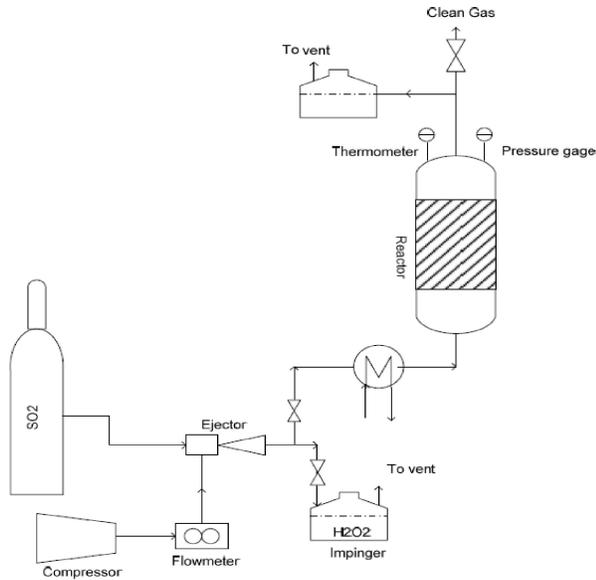


Figure 3: Schematic of proposed pilot plant

$$\dot{m}_{bcn.} \Big|_z - \dot{m}_{bcn.} \Big|_{z+\Delta z} - \dot{m}_{mcon.} - \dot{m}_r = m_{acc.} \quad (17)$$

$$AU_{mf} C_c \Big|_z - AU_{mf} C_c \Big|_{z+\Delta z} - NQ(C_c - C_d) - r = V_c \frac{\partial C_c}{\partial t} \quad (18)$$

The unreacted core model justifies this dependency. According to this model, the reaction occurs on the outer layer of catalyst initially and then continues in the other inner layers. So, the whole catalyst will be deactivated finally.

Regeneration process should occur to active the catalyst. It is assumed that the catalyst dimensions remain unchanged during the reaction, so the rate of reaction is expressed as Equation 19. The rate of solid-gas reaction is determined by two diffusion resistances which one is between the catalyst layers and the other is in chemical reaction. So,  $K$  is defined as Equation 20. Where,  $K_s$  the constant rate of the first order reaction is expressed as Equation 21.

$$r = kA_c C_c \quad (19)$$

$$k = \frac{1}{\frac{r_p(r_p - r_u)}{r_u D_{eff.}} + \frac{r_p^2}{r_u^2 k_s}} \quad (20)$$

$$k_s = k_{s0} \exp\left(\frac{-E_a}{RT}\right) \quad (21)$$

The amount of frequency factor varies with different amounts of aluminum oxide content in the catalyst. Variation of diameter for the unreacted layer of the catalyst with time is expressed as Equation 22<sup>[14]</sup>.

$$\frac{1}{3r_p D_{eff.}} r_c^3 - \frac{1}{2D_{eff.}} r_c^2 - \frac{1}{k_s} r_c + \frac{1}{6D_{eff.}} r_p^2 + \frac{1}{k_s} r_p - \frac{C_A}{\rho_p} t = 0 \quad (22)$$

The  $\text{SO}_2$  concentration is found in each phase through each compartment by solving Equation 16 and 18 simultaneously considering boundary conditions and initial condition which are defined in Equation 23.

$$C(t=0) = C_0, C(z=0) = C_{in} \quad (23)$$

Final concentration of the released gas is calculated according to the Equation 24.

$$C_g = FC_d + (1-F)C_c \quad (24)$$

So, the concentration profile of is found by solving the mentioned equations through the bed considering the time.

## RESULT AND DISCUSSION

Comparison between experimental results and theoretical values is shown in figures in this part. The proposed pilot plant sets up with 0.07 m in wide and 0.20 m in height includes Aluminum Oxide catalyst particles with 0.003 in diameter. Reaction occurs at temperature of 250 °C and pressure of 111.458 kPa. Experiments are held in the mentioned pilot plant to investigate the effect of different operating conditions on the amount of removed sulfur dioxide. In addition, the effect of feed velocity and some bed specifications such as width and height of catalytic bed and diameter of catalytic particles are surveyed. The performance of the fluidized bed in  $\text{SO}_2$  removal is presented as  $\text{SO}_2$  removal percentage in this section and is defined as Equation 25.

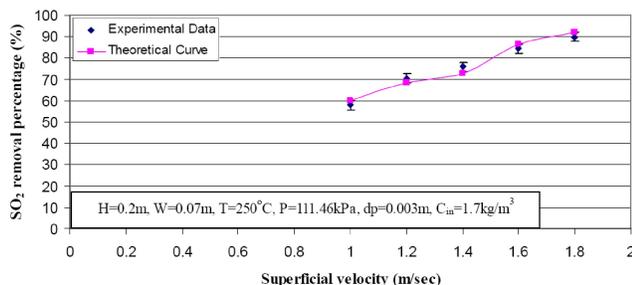
$$\text{SO}_2 \text{ removal percentage} = \frac{\text{Amount of SO}_2 \text{ in the feed} - \text{Final amount of SO}_2}{\text{Amount of SO}_2 \text{ in the feed}} \times 100\% \quad (25)$$

### The Effect of superficial velocity

If the feed superficial velocity increases, the percentage of  $\text{SO}_2$  which is removed will be increased.

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Figure 4 shows this relation. Velocity changes from 1  $m/s$  to 1.8  $m/s$  and augments the percentage of SO<sub>2</sub> removed from the feed gas from 50 % to 90 %. Increasing the feed flow rate causes the augmentation in superficial velocity and somehow makes higher turbulent flow in the catalytic bed. So, decreases the mass transfer diffusion resistance from the gas flow to the catalyst and the level of SO<sub>2</sub> removal become more than frequent, finally.

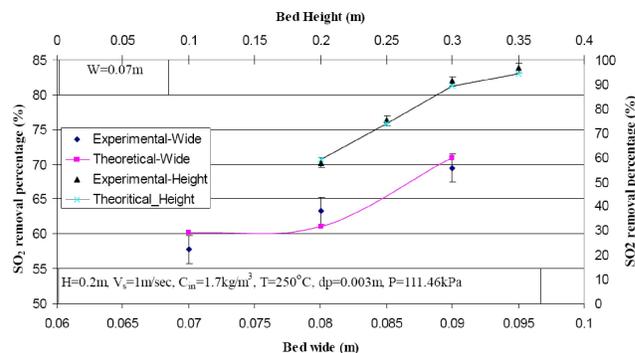


**Figure 4 : The amount of removed SO<sub>2</sub> versus the feed superficial velocity**

### The Effect of height of catalytic bed and width of catalytic bed

The effect of different heights of catalytic bed on the amount of removed SO<sub>2</sub> is shown in Figure 5. According to the results of experiments, if the bed height augments 0.15  $m$  then SO<sub>2</sub> removal will increase from 55 % to about 98 %. Since the mass transfer surface area is increased by height of bed so makes better process performance and the amount of SO<sub>2</sub> removal is increased. Residence time increasing and finally the higher contact time between gas and solid phase is concluded from increasing in height. Also, it is obtained that no sever pressure drop is obtained by increasing the height of bed in the experimental ranges.

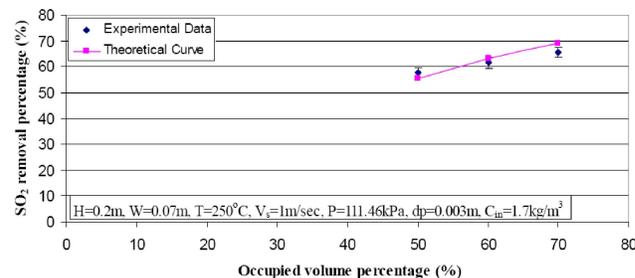
Experiments are held in three different width of bed as 0.07  $m$ , 0.08 and 0.09. The amount of SO<sub>2</sub> removal percentage increases from about 57% to 63% and 69%, respectively. Increasing in the cross section width of the reactor increases the mass transfer surface area, so the rate of SO<sub>2</sub> removal increases and Figure 5 shows these values.



**Figure 5 : The amount of removed SO<sub>2</sub> versus the height and width of bed.**

### The effect of occupied volume

Results show the contact area increases when the occupied volume percentage is increased and Figure 6 proves this fact. The volume fraction of the bed which is occupied by the catalyst particles is named as occupied volume.

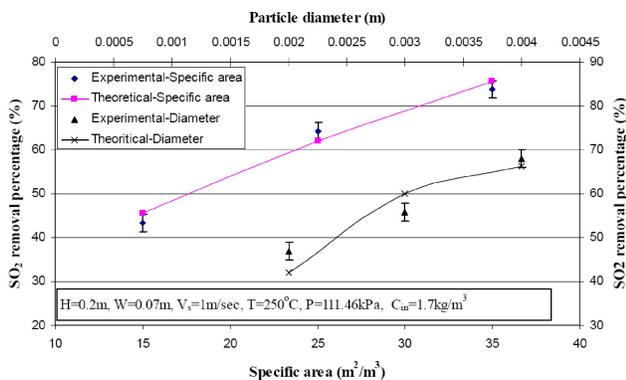


**Figure 6 : The amount of removed SO<sub>2</sub> versus the occupied volume of bed.**

### The effect of diameter of catalyst and specific area

Figure 7 shows the positive effect of larger diameter of catalyst particles on the performance of the SO<sub>2</sub> removal process. Catalysts with three different diameters as 0.002, 0.003 and 0.004  $m$  are used in this experiments. Although the convective mass transfer surface area seems to be decreased by somehow larger particles but the diffusion mass transfer surface areas inside the catalyst are increased. So, the performance of the process is improved in this range of particle diameter.

Also, the effect of bed specific surface area on the performance of the SO<sub>2</sub> removal process is obtained in the Figure 7. This Figure indicates on this fact that, increasing in mass transfer surface area of catalyst increases the rate of SO<sub>2</sub> adsorption in the bed.

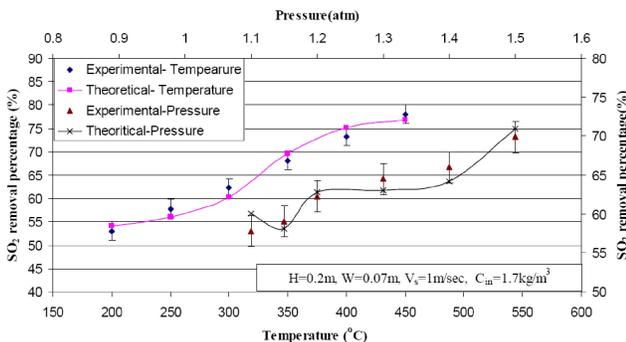


**Figure 7 :** The amount of removed SO<sub>2</sub> versus the diameter of particles and the bed specific surface area.

**The effect of temperature and pressure**

Temperature is one of the key operating parameters in the catalytic process and determines the chemical reaction direction. The effect of operating temperature is considered in this part.

The higher temperature makes higher reaction rate between catalyst and SO<sub>2</sub> content in air. Increasing temperature from 200°C to 450°C precedes the adsorption process of SO<sub>2</sub> from about 52% to about 79% respectively. Also, temperature may increase the amount of SO<sub>2</sub> removal percentage at a constant slope according to the plot in the Figure 8.



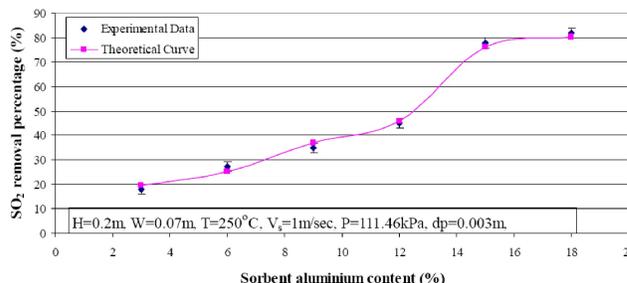
**Figure 8 :** The amount of removed SO<sub>2</sub> versus the operating temperature and pressure.

As shown in Figure 8, the increase in operating pressure increases the amount of SO<sub>2</sub> removal percentage. This may be since of the direct effect of pressure on the diffusion mass transfer coefficient in the gas stream.

**The effect of sorbent Aluminum content**

Figure 9 shows the effect of amount of sorbent Alu-

minum on the operating efficiency. The amount of SO<sub>2</sub> removed from the polluted gas is increased by increasing the amount of Aluminum in the catalyst. Also, theoretical results show the same inclination between process efficiency and the sorbent contents. This may be because of the dependency of the amount of reaction constant on the amount of the Aluminum sorbent. According to the results the proper amount of sorbent in the catalyst is between 12 to 15 percentages. The largest augmentation in the curve is obtained in this range of sorbent Aluminum content with the highest slope. The percentage of SO<sub>2</sub> removal shows seldom increase at higher amounts of catalyst because of the increase in the rate of erosion in Aluminum Oxide catalyst.



**Figure 9 :** The amount of removed SO<sub>2</sub> versus the amount of sorbent content

**CONCLUSION**

Pilot plant fluidized bed reactors with Aluminum catalyst are proposed to investigate the SO<sub>2</sub> removing process from the gas stream. Also, mathematical modeling is done by related mass transfer equations. The effects of changing in the amounts of key operating parameters on the process efficiency are surveyed.

Also, the increase in operating temperature, pressure, sorbent Aluminum content, diameter of catalyst, occupied volume of bed, width of bed, height of bed, superficial velocity and specific surface area shows direct effect and increases the efficiency of the SO<sub>2</sub> removal process.

According to the results, increasing the height of bed just about 0.15 m causes about 0.39% pickup in the efficiency of SO<sub>2</sub> removal process and the value of SO<sub>2</sub> removal percentage reaches to 99%. Increas-

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ing the feed superficial velocity from  $1 \text{ m/s}$  to  $1.8 \text{ m/s}$  shows about 30% enhancement in the value of  $SO_2$  removal percentage from 60% to 90%. Also, increasing in sorbent Aluminum content increases the process efficiency to about 80% value of removal percentage and the best range of amount of sorbent Aluminum is found between 12% and 15% to prevent the catalyst friability. Operating pressure augmentation from 110 to 150  $kPa$  increases the process operating efficiency slightly, from 60%  $SO_2$  removal to 70%.

All mentioned parameters which are considered in this paper, affect the mass transfer surface area, mass transfer contact time or the turbulences of mass transfer media. In addition, the effect of temperature on the mass transfer coefficient and sorbent content on the reaction rate constant is shown by the theoretical and experimental results. According to the achieved plots, the average deviation value between theoretical and experimental results is 7.2%. So, the mathematical model is reliable due to the experimental data.

### NOMENCLATURE

$A$  Bed cross section,  $m^2$   
 $A_c$  Particle surface area in the continuous phase,  $m^2$   
 $C_c$  Gas concentration in continuous phase,  $kg/m^3$   
 $C_c$  Gas concentration in continuous phase,  $kg/m^3$   
 $C_d$  Gas concentration in dispersed phase,  $kg/m^3$   
 $C_{in}$  Inlet concentration of  $SO_2$  into the bed,  $kg/m^3$   
 $C_{in}$  Inlet concentration of  $SO_2$  into the bed,  $kg/m^3$   
 $C_g$  Gas outlet concentration,  $kg/m^3$   
 $C_0$  Initial concentration of  $SO_2$ ,  $kg/m^3$ ,

$D_{AB}$   $SO_2$  diffusion coefficient in gas,  $m^2/sec$   
 $D_{eff}$  Gas effective diffusion coefficient in continuous phase,  $m^2/sec$   
 $g$  Gravitational acceleration,  $m/sec^2$   
 $k$  Overall reaction constant,  $m/sec$   
 $\kappa_g$  Gas mass transfer coefficient from dispersed phase to continuous phase,  $m/sec$   
 $k_s$  Reaction constant,  $m/sec$   
 $k_{s_0}$  Frequency factor,  $m/sec$   
 $m_{acc}$  Accumulated mass of  $SO_2$  in the control volume,  $kg/sec$   
 $\dot{m}_{dis}$  Amount of mass flow rate of dispersed phase transferred by velocity,  $kg/sec$   
 $\dot{m}_{dis}$  Dispersed phase molecular amount of mass flow rate,  $kg/sec$   
 $\dot{m}_{bcon}$  Amount of mass flow rate of continuous phase transferred by velocity,  $kg/sec$   
 $\dot{m}_{mcon}$  Continuous phase molecular amount of mass flow rate,  $kg/sec$   
 $\dot{m}_r$  Mass flow disappeared by reaction,  $kg/sec$   
 $n$  Number of orifices in the gas distributor  
 $N$  Number of bubbles  
 $Q$  Gas volumetric flow rate exchanged between phases,  $m^3/sec$

- $r$  Reaction rate,  $\text{kg}/\text{sec}$
- $r_u$  Diameter of unreacted core,  $m$
- $r_p$ , Radius of particle  $m$
- $R$  Universal gas constant,  $\text{kJ}/\text{kg.K}$
- $t$  Time, sec
- $T$  Absolute temperature,  $K$
- $U$  Actual gas velocity,  $m/\text{sec}$
- $U_{mf}$  Gas minimum fluidization velocity,  $m/\text{sec}$
- $V_c$  Continuous volume fraction in the control volume
- $V_d$  Dispersed volume fraction in the control volume
- $z$  Vertical direction,  $m$
- Italic**
- $\rho_g$  Gas density,  $\text{kg}/\text{m}^3$
- $\rho_p$  Particle density,  $\text{kg}/\text{m}^3$
- $\mu$  Gas viscosity,  $\text{kg}/\text{m.s}$

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