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Sulfur Dioxide Removal in Coal Slurry Reactor

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Abstract

The objective of this work was to study the feasibility of using coal slurry for the removal of SO_2 from simulated flue gas stream (air, SO_2). The effect of gas rate, temperature, and initial SO_2 concentration on the overall removal efficiency was investigated at wet and dry bed conditions. The results indicated that the optimum gas rate was 60 ℓ/min. The SO_2 removal efficiency was highly temperature sensitive, and increases with increasing the bed temperature especially at wet bed conditions and decreases with increasing SO_2 initial concentration. A mathematical model for the desulfurization process was proposed based on the material balance for gaseous and solid phase streams. The model was found to give a very good description of the experimental data with 95% confidence level.

Key words: SO_2 Removal, Coal Slurry Reactor, FGD, Modeling

الخلاصة

يهدف البحث الى دراسة جدوى أستخدام الفحم المنشط في وسط ثُلاثي الأطوار لازالة ثنائي اوكسيد الكبريت من مجرى غازي محاكي لمجرى غازات مدخنة(هواء,ثنائي اوكسيد الكبريت). لقد تم دراسة تاثير كل من نسبة تدفق الغاز, درجة الحرارة وتركيز الغاز الابتدائي على كفاءة الازالة الكلية في حالة الحشوه الرطبه و الجافه. النتائج اشارت الى ان معدل تدفق الغاز المثالي هو 60 لتر/دقيقة. كفاءة ازالة ثنائي اوكسيد الكبريت تتاثر كثيرا بدرجة الحرارة حيث تزداد بزيادة درجة الحراره خصوصا في حالة الحشوة الرطبة و تقل بزياده تركيز غاز ثنائي اوكسيد الكبريت الأبتدائي. تم اعداد موديل رياضي يصف عملية الأزاله. الموديل الرياضي المقترح أُسس على موازنة الماده لكل من الغاز والصلب. الموديل أعطى وصف جيد جداً للنتائج العمليه وبمقدار ثقة 95%.

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1- Introduction:

Adsorption of sulfur dioxide on carbonaceous materials has been extensively studied. Lizzio and DeBarr (1) showed that the reaction of SO_2 with carbon (C) in the presence of O_2 (in air) and H_2O involves a series of reactions that leads to the formation of sulfuric acid as the final product. The rate-determining step in the overall process is the oxidation of SO_2 to SO_3. In optimizing the SO_2 removal capabilities of carbon, most studies only assume a given mechanism for SO_2 adsorption and conversion to H_2SO_4 to be operable. Cho (2) reported that activated carbon which contains the catalysis of ferric/ferrous ions for the reaction between SO_2 and O_2 has been known for many years. The oxidation reactions occur by three routes. First, SO_2 serves as a reducing reagent of ferric ion. Second, SO_2 together with O_2 serves as an oxidizing reagent of ferrous ion to ferric ion. Third, ferric ion catalyzes the oxidation reaction of SO_2.. Bagreev *et al.* (3) showed that the normalized capacity of the activated carbon adsorbent, especially at higher temperature , is much larger than that of the activated carbon at lower temperature. The significantly higher activity of the surfaces of the adsorbents carbonized at higher temperature result from the combined effect of de-hydroxylation of inorganic phase oxides and their solid state reactions promoted at high temperature and reducing conditions and with increasing temperature new reactive oxides are formed, e.g. $FeSO_4$ and/or $CaSO_3$ are well-developed compared to the starting material. Cho and Miller (4) studied the overall removal of sulfur dioxide flue gas with coal scrubbing; the objective of the study was to determine the effects of temperature, oxygen concentration and SO_2 concentration on SO_2 removal from simulated flue gas streams and on leaching rate of coal pyrite. Bagreev and Bandosz (5) show for the removal of SO_2 using coal slurry the importance of the role of water in the formation of sulfuric acid in pore system. Liu et al. (6) used waste semi-coke as the raw material to prepare catalysts of industrial scale size for SO_2 removal. The effect of water content in the flue gas, reaction temperature, and space velocity on the de-sulfurizing property was investigated. Space velocity exhibited an optimal value of 830 h^{-1}.

The present study focuses on the modeling of the de-sulfurization process in coal slurry reactor.

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2- Experimental Work:

The experiments were carried out in a laboratory scale apparatus which is shown schematically in Figure (2). The test section consists of a cylindrical column of (7.5) cm inside diameter and (50) cm height filled to a height of 24 cm with (400) g activated carbon (dp=1-3 mm). The chemical analysis and the physical properties of the activated carbon used in this study were given in tables (1and 2), respectively. The chemical analysis was determined using atomic absorption, and the physical properties were obtained using surface area analyzer. A (T-type) thermocouple was used to measure the temperature of the activated carbon bed. The thermocouple was connected to an on/off controller to adjust the bed temperature. A heater of 225 watt, wrapped around the bed, was used to heat the bed to the required temperature. The heater was connected to a variac that regulates the electrical current supplied to the heater.

Table (1): Chemical Analysis of Activated Carbon

Material	wt %	Material	wt %
C	92.46	Mg^{+2}	3.25
Ca^{+2}	2.75	Mn^{+2}	0.116
Co^{+2}	0.075	Pb^{+2}	0.512
Cu^{+2}	0.15	PO_4^{-3}	0.018
Fe^{+2}	0.0696	SO_4^{-2}	0.018
Fe^{+3}	0.325	Zn^{+2}	0.025

Table (2): Physical Properties of Activated Carbon

Surface area	1122.13 (m^2 / g)
Bulk density	0.4423 (g /cm^3)
Porosity	0.48972 (-)

• ***Experimental Procedure:***

Four hundreds grams of (1-3) mm particles diameter of activated carbon were introduced into the test section. A slurry of activated carbon was prepared by immersing the activated carbon bed with 2 liter of water. Sulfur Dioxide gas was synthesized (7) by dropping the desired concentration of sulfuric acid into the SO_2 generation flask that contains the aqueous sodium sulfite solution according to the chemical reaction:

$$Na_2SO_3 + H_2SO_4 \Rightarrow SO_2 + Na_2SO_4 + H_2O \quad .....(1)$$

After adjusting the bed temperature to the desired value, the air stream at a desired flow rate was then passed through this container to carry the SO_2 to the test section. The initial concentration of SO_2 was measured at the beginning of each run. In order to measure the concentration of SO_2 in the effluent gas stream, (10) ml of the iodine sample was taken from the absorption trap, titrated with standard sodium thiosulfate solution (0.1) N, in the presence of starch indicator.

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This step was repeated every 5 minutes till the end of experimental run time (1 hr). After each experimental run the bed was washed with fresh water until the concentration of SO_2 gas in the air stream is free from SO_2.Each experimental run were repeated for 3-5 times to validate the experimental results. The removal efficiency of SO_2 was calculated as the ratio of SO_2 concentration that was adsorbed by activated carbon to the initial concentration of SO_2 gas fed to the bed.

$$\eta\,(\%) = \frac{C_0 - C_{so_2}}{C_0} * 100 \qquad .....(2)$$

3- Model Development

The following assumptions were used to formulate the mathematical model: constant porosity, spherical solid particle, dispersion radial and axial directions are neglected (i.e. plug flow), and isothermal process.

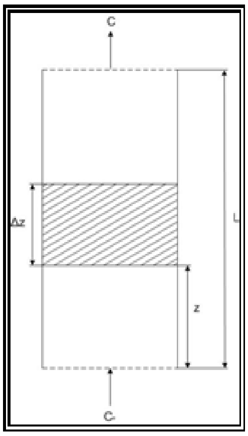

Figure (1) Mathematical Representation of the Model

- For The Gas Phase

Referring to Figure (1) a mass balance is performed for the gas phase:-

$$\begin{bmatrix}\text{molar rate}\\ \text{In}\end{bmatrix} - \begin{bmatrix}\text{molar rate}\\ \text{out}\end{bmatrix} - \begin{bmatrix}\text{molar rate of}\\ \text{disapperance}\end{bmatrix} = \begin{bmatrix}\text{molar rate of}\\ \text{accumulation}\end{bmatrix} \qquad .....(3)$$

$$F_{so_2}\Big|_z - F_{so_2}\Big|_{z+\Delta z} - a\, r_{so_2}\,(A_R\,\Delta z) = \varepsilon\,\rho\,(A_R\,\Delta z)\frac{\partial C_{so_2}}{\partial t} \qquad .....(4)$$

Dividing both sides by $(A_R\,\Delta z)$ and taking the lim it as $\Delta z \to 0$

$$\lim_{\Delta z \to 0} \frac{F_{so_2}\Big|_z - F_{so_2}\Big|_{z+\Delta z}}{A_R\,\Delta z} - a\, r_{so_2} = \varepsilon\,\rho\,\frac{\partial C_{so_2}}{\partial t} \qquad .....(5)$$

$$-\frac{\partial F_{so_2}}{A_R\,\partial z} - a\, r_{so_2} = \varepsilon\,\rho\,\frac{\partial C_{so_2}}{\partial t}$$

$$\frac{F_{so_2}}{A_R} = u\, C_{so_2} \qquad \left(\frac{mol}{m^2\,s} = \frac{m}{s}\cdot\frac{mol}{m^3}\right) \qquad .....(6)$$

$$-u_z\,\frac{\partial C_{so_2}}{\partial z} - a\, r_{so_2} = \varepsilon\,\rho\,\frac{\partial C_{so_2}}{\partial t}$$

Transfer the differential equation into dimensionless form:

$$-\frac{C_o\,u_z}{L}\,\frac{\partial \dfrac{C_{so_2}}{C_o}}{\partial \dfrac{z}{L}} - a\, r_{so_2} = \varepsilon\,\rho\,C_o\,\frac{\partial \dfrac{C_{so_2}}{C_o}}{\partial t} \qquad .....(7)$$

$$Z = \frac{z}{L}$$

$$Y = \frac{C_{so_2}}{C_o}$$

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$$n = C_o\, u_z\, A_R \qquad \left(\frac{mol}{s} = \frac{mol}{m^3}.\frac{m}{s}.m^2\right) \qquad .....(8)$$

$$-\frac{n}{A_R\, L}\frac{\partial Y}{\partial Z} - a\, r_{so_2} = \varepsilon\, \rho\, C_o \frac{\partial Y}{\partial t} \qquad .....(9)$$

$$a = \frac{S_e\, w}{V_R}$$

Rearranging equation (9) gives:

$$\frac{n}{L\, A_R}\frac{\partial Y}{\partial Z} + \frac{S_e\, w}{V_R} r_{so_2} + \varepsilon\, \rho\, C_o \frac{\partial Y}{\partial t} = 0 \qquad .....(10)$$

- For the Solid Phase:-

Referring to Figure (1) a mass balance is performed for the solid phase:-

$$\begin{bmatrix}\text{molar rate}\\ \text{In}\end{bmatrix} - \begin{bmatrix}\text{molar rate}\\ \text{out}\end{bmatrix} - \begin{bmatrix}\text{molar rate of}\\ \text{disapperance}\end{bmatrix} = \begin{bmatrix}\text{molar rate of}\\ \text{accumulation}\end{bmatrix} \qquad .....(3)$$

$$-(-\, r_{so_2})\, v_s\, S_e\, M = \frac{\partial \dfrac{(C_o - C_{so_2})}{C_o}}{\partial t} \qquad .....(11)$$

$$\frac{(C_o - C_{so_2})}{C_o} = X \text{ (Sorbent Conversion)}$$

Rearranging equation (4 – 3) gives :

$$\frac{\partial X}{\partial t} = r_{so_2}\, v_s\, S_e\, M \qquad .....(12)$$

To solve equations (10) and (12) the following initial boundary conditions are used:

$$a - \quad \text{at } t = 0\ \&\ Z \rangle 0 \Rightarrow Y = 0, X = 0$$

$$b - \quad \text{at } Z = 0\ \&\ t \rangle 0 \Rightarrow Y = 1$$

In order to solve the differential equations (10) and (12) a rate expression of the desulfurization reaction, SO_2 was assumed to react with water molecules in the presence of the sorbent to form H_2SO_3 according to the reaction:

$$SO_2 + H_2O + \text{Sorbent} \Rightarrow H_2SO_3 - \text{Sorbent} \qquad .....(13)$$

The rate of reaction (r_{SO2}) for the desulfurization reaction over the sorbent can be expressed as a product of a temperature dependent rate constant k (T) and the concentration of the reactants (36) as shown:

$$r_{so_2} = \frac{dX}{dt} = k(T)\,[SO_2]^{\alpha}\,[H_2O]^{\beta} \qquad .....(14)$$

Where (α) is the order of the reaction with respect to SO_2 and (β) is the order of the reaction with respect to H_2O. Assuming that the concentration of H_2O is in excess as compared to SO_2, Eq. (14) can be simplified to:

$$\frac{dX}{dt} = k(T)\, C_o\, (1 - X)^{\alpha}\, (RH)^{\beta} \qquad .....(15)$$

Where (C_o) is the initial concentration of SO_2 and (RH) is the relative humidity of the feed gas.

The temperature dependent rate constant in Eq. (15) is taken as the global reaction rate constant which obeys the Arrhenius Law (12), k, given by:

$$k(T) = k = A_f \exp\left(-\frac{E}{RT}\right) \qquad .....(16$$

Where (E) is the activation energy of the desulfurization reaction, (A_f) is the frequency or pre-exponential factor of the desulfurization reaction and (R) is the universal gas constant. The rate expression for the desulfurization reaction can be written as:

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$$r_{so_2} = A_f \exp\left(-\frac{E}{RT}\right) C_o \,(1-X)^{\alpha}\,(RH)^{\beta} \qquad .....(17)$$

With the inclusion of effectiveness factor (ξ) in the rate expression, it is written as:

$$r_{so_2} = \xi\, A_f \exp\left(-\frac{E}{RT}\right) C_o \,(1-X)^{\alpha}\,(RH)^{\beta} \qquad .....(18)$$

The effectiveness factor is taken as a constant equal to 0.9. In order to obtain the values of (A_f, α, E and β) Eq. (18) was substituted into Eq. (10) and Eq. (12), yielding Eqs.(19) and Eq. (20).

For the gas phase:-

$$\frac{n}{L\,A_R}\frac{\partial Y}{\partial Z} + \varepsilon\,\rho\,C_o\frac{\partial Y}{\partial t} + \frac{S_e\,w}{V_R}\left[\xi\, A_f \exp\left(-\frac{E}{RT}\right) C_o \,(1-X)^{\alpha}\,(RH)^{\beta}\right] = 0 \qquad .....(19)$$

For the solid phase:-

$$\frac{\partial X}{\partial t} = v_s\, S_e\, M\left[\xi\, A_f \exp\left(-\frac{E}{RT}\right) C_o \,(1-X)^{\alpha}\,(RH)^{\beta}\right] \qquad .....(20)$$

The ordinary differential equation was solved numerically. The values of (A_f,α, E and β) were then obtained by least-square fitting of the solved ordinary differential equation to the experimental data.

A computer program has been developed in Matlab V7.0 to perform the numerical solution formulated previously. The values of A_f, α, E and β were found to be 1.07, 2.2, 25 kJ/mol and 3.1 respectively.

4- Results & Discussion

Figure (3) clarify that an optimal overall removal efficiency can be obtained at gas rate of 60 l/min. Beyond this value the overall removal efficiency decreases. These results are in agreement with the work of Liu et al. (6) who obtained a similar behavior for the change of space velocity with SO_2 removal efficiency. This may be attributed to that the kinetic behavior varied with gas rate and the desulfurizing property was controlled by diffusion at gas rates less than 60 l/min., and controlled by adsorption or catalytic reaction at gas rates higher than 60 l/min.

Figure (4) shows the change of the overall removal efficiency of SO_2 with temperature for different initial SO_2 concentrations, and constant flue gas flow rate of 20 l/min in wet bed conditions. The trend of the results indicate that the overall removal efficiency is independent of SO_2 initial concentration at low temperatures (T=30 and 40) ^{0}C and increases at higher temperatures. The significantly higher activity of the surfaces of the adsorbents carbonized at higher temperature result from the combined effect of dehydroxylation of inorganic phase oxides and their solid state reactions promoted at high temperature and reducing conditions and with increasing temperature new reactive oxides formed, e.g. $FeSO_3$ and/or $CaSO_3$ are well-developed compared to the starting material.

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Figure (5) shows the effect of temperature on the overall removal efficiency of SO_2 for constant flue gas flow rate of 20 l/min in dry bed conditions. The trend of the results shows a similar pattern as that obtained in wet bed where at low temperatures the effect of the initial concentration of SO_2 is minimal and increases as the temperature increases. In dry beds, the absence of water precludes the formation of sulfuric acid in the pore system. However, it is possible that at higher temperatures SO_2 forms sulfites through reactions with oxides to form $FeSO_4$ and/or $CaSO_3$ that are not formed at lower temperatures.

Figures (6) to (9) show the effect of initial concentrations of SO_2 for an hour interval on the effluent concentration of SO_2 at constant flow rate 20 l/min, and various temperatures in wet and dry bed conditions.

The results indicate that at any certain time the effluent concentration of SO_2 increases as the initial concentrations of SO_2 increases. This is due to the decrease in the activity of activated carbon as a direct consequence of exposing a fixed amount of the sorbent to the increasing concentration of SO_2.

Figures (10) to (13) show the change of effluent concentration with time for wet and dry bed conditions for an hour interval at constant flue gas rate of 20 l/min, and different operating conditions.

The trends of the results indicate a lower value for the effluent gas concentration in the wet beds as compared to those in dry beds for the same experimental conditions. Moreover the difference in the concentration of SO_2 in the effluent gas between wet and dry bed conditions at any time becomes higher as the temperature of the bed increased. Bagreev and Bandoz [(5)] explained that for high temperature, volume decreases in micropores, which indicates the gradual filling of these pores with the products of surface reactions (assuming that SO_2 is oxidized to H_2SO_4 of density 1.83 g/cm^3 in wet bed and suggests that the acid is deposited at entrances to the pores).Therefore in the presence of water , the removal of SO_2 is much larger than that for dry bed.

Figure (14) shows the break-through life of 200 g of the activated carbon at flue gas flow rate of 60 l/min, initial concentration of SO_2 of 500 ppm and a temperature of 80 ^{0}C in wet bed. From the curve obtained, it can be seen that the effluent concentration of SO_2 increases with time until a time of about 600 min beyond which the concentration of the activated carbon bed with time [(10)].

Figure (15) shows the simulated vs. experimental curves at two different experimental conditions. It can be seen that the model gives very good predications for the experimental data. The confidence level of the model

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was 95% using the goodness of fit test (11).

5- Conclusions:

The conclusions withdrawn from the present study may be summarized as follows: The removal efficiency of SO_2 increased on increasing the flue gas flow rate till 60 l/min and then decreases slowly. The removal efficiency depends on the reaction temperature to a large extent. The results indicate an increase in the SO_2 removal efficiency of about 3.6% on increasing the temperature by 10 ^{O}C in the range of (30-60) ^{O}C and an increases of about 1.7% in the range of (60-80) ^{O}C, all for 1 hr and C_0=500 ppm in wet bed.

A reduction in the SO_2 removal efficiency was observed on increasing the SO_2 initial concentration in the flue gas stream. The removal efficiency of SO_2 was higher under wet conditions than that under dry conditions. The mathematical model proposed was found to provide a good description of the desulfurization reaction under conditions prevailing in the flue gas desulfurization process at higher temperature.

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Nomenclature

Symbol	Definition	Unit
A_R	Transversal bed section	m^2
a	Specific area of the bed	m^2/m^3
A_f	Frequency factor	s^{-1}
C_0	Initial conc.of SO_2	ppm
C_{SO2}	Effluent conc.of SO_2	ppm
C	SO_2 conc.	ppm
dp	Slurry particle diameter	mm
E	Activation energy	$kJ.mol^{-1}$
k	Global reaction rate constant	s^{-1}
k (T)	rate constant	s^{-1}
L	Bed length	m
M	Molecular weight of activated carbon	$g.\ mol^{-1}$
n	Molar flow rate of SO_2	$mol.s^{-1}$
Q	Flow rate of SO_2	l/min
R	Universal gas constant	$J.\ mol^{-1}.K^{-1}$
RH	Relative humidity	[%]
r_{SO2}	Reaction rate	$mol.m^{-2}.s^{-1}$
S_e	Specific surface area	$m^2.g^{-1}$
T	Temperature	^0C
t	Time	s or min
u	Axial superficial velocity of gas	$m.s^{-1}$
v_s	Stoichiometric coefficient	[-]
V_{STP}	Volume of gas at standard conditions	ℓ
V_R	Volume of the reaction bed	m^3
w	Sorbent weight in the bed	g
X	Sorbent conversion	[-]
Y	Dimensionless SO_2 concentration (C/C_0)	[-]
Z	Dimensionless length position	[-]
α	Order of the desulfurization reaction with respect to SO_2	[-]
β	Order of the desulfurisationn reaction with respect to H_2O	[-]
ρ	Molar density of solid particles	[mol.m^{-3}l.m^{-3}]
η	Removal efficiency of SO_2	[%]
ε	Bed porosity	[-]

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η	Removal efficiency of SO_2	[%]
ξ	Effectiveness factor	[-]

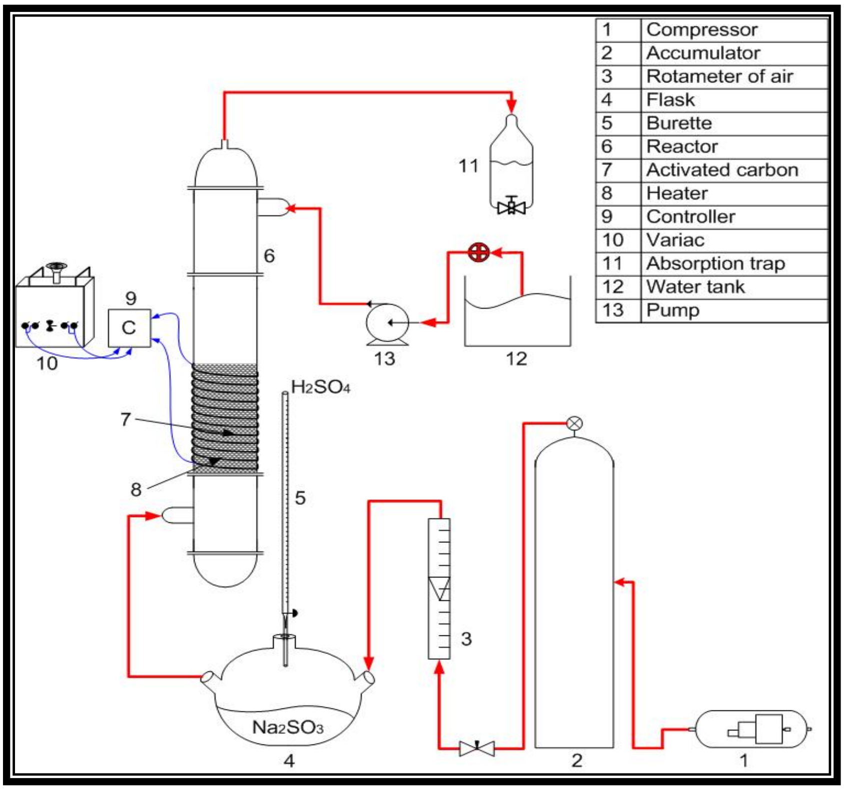

Figure (2) Schematic Diagram of the Experimental Set-up

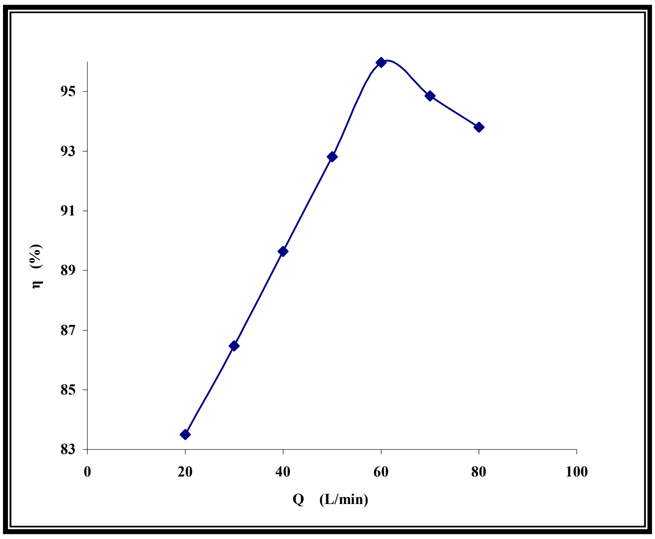

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Figure (3) The effect of gas flow rate on the overall removal efficiency of SO_2 in wet bed

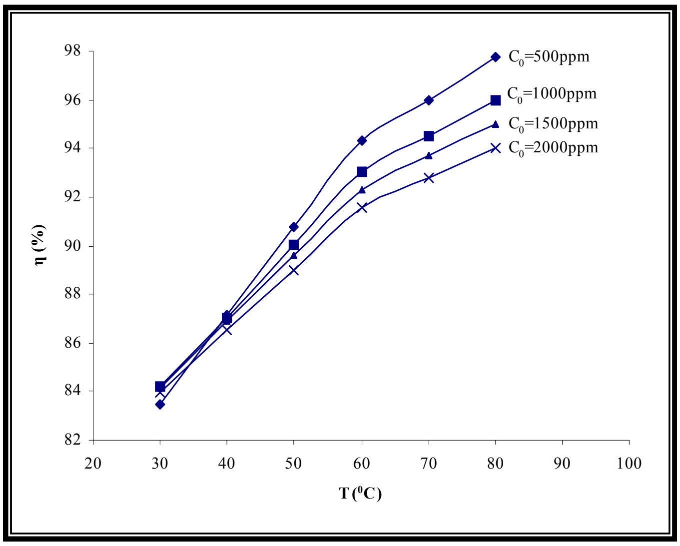

Figure (4) The effect of temperature on the overall removal efficiency of sulfur dioxide at Q=20 l/min in wet bed

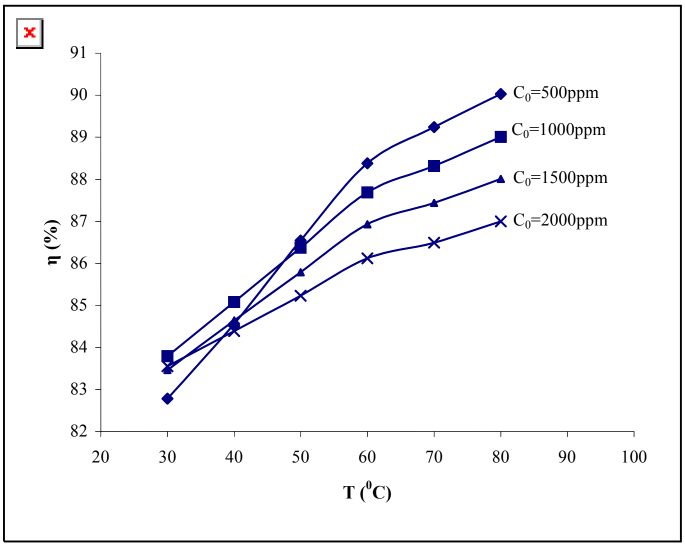

Figure (5) The effect of temperature on the overall removal efficiency of sulfur dioxide at Q=20 l/min in dry bed

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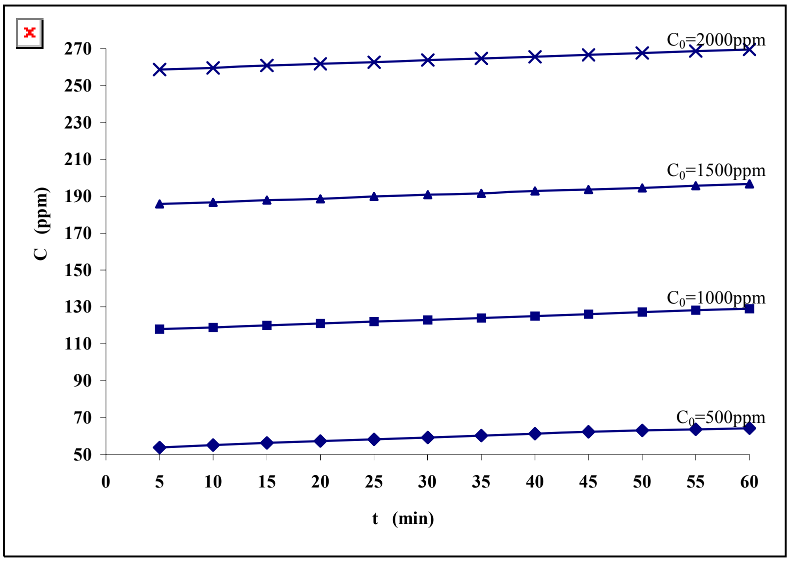

Figure (6) The effect of SO_2 initial concentration on the effluent concentration of SO_2 ($T= 40\ ^0C$) in wet bed condition

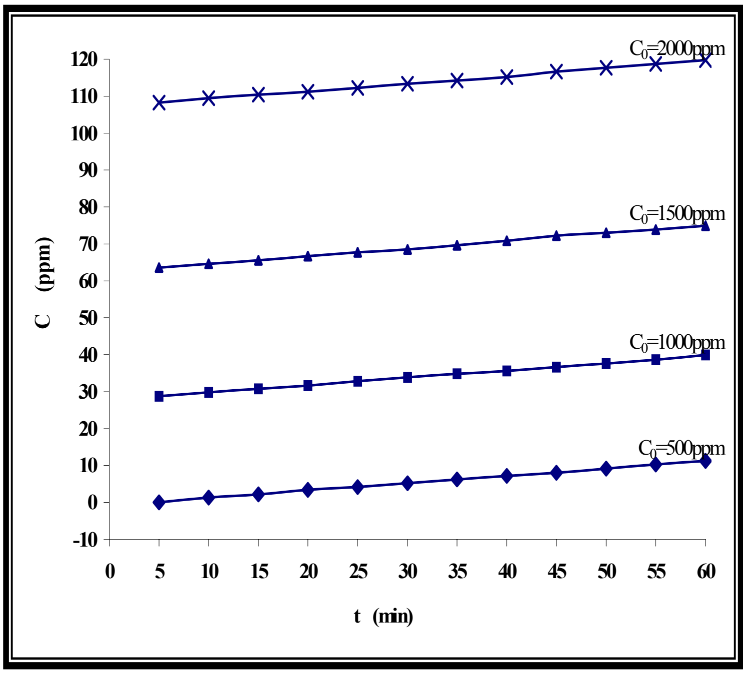

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Figure (7) The effect of SO_2 initial concentration on the effluent concentration of SO_2 ($T= 80\ ^0C$) in wet bed condition

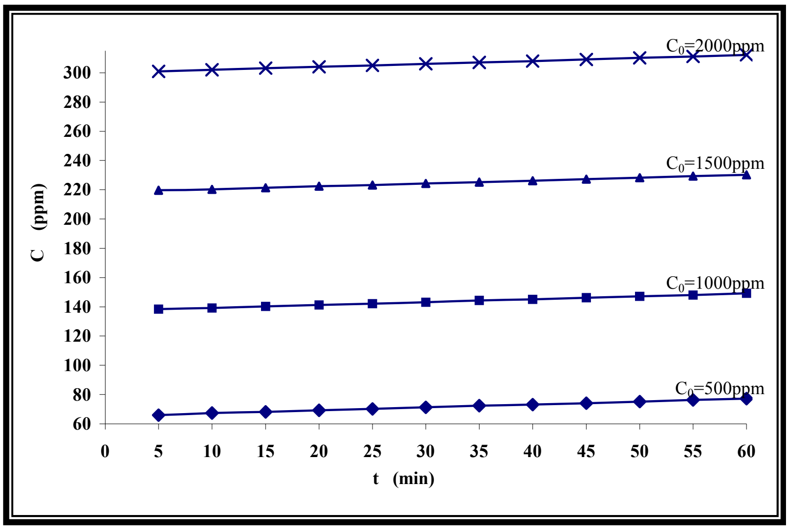

Figure (8) The effect of SO_2 initial concentration on the effluent concentration of SO_2 ($T= 40\ ^0C$) in dry bed condition

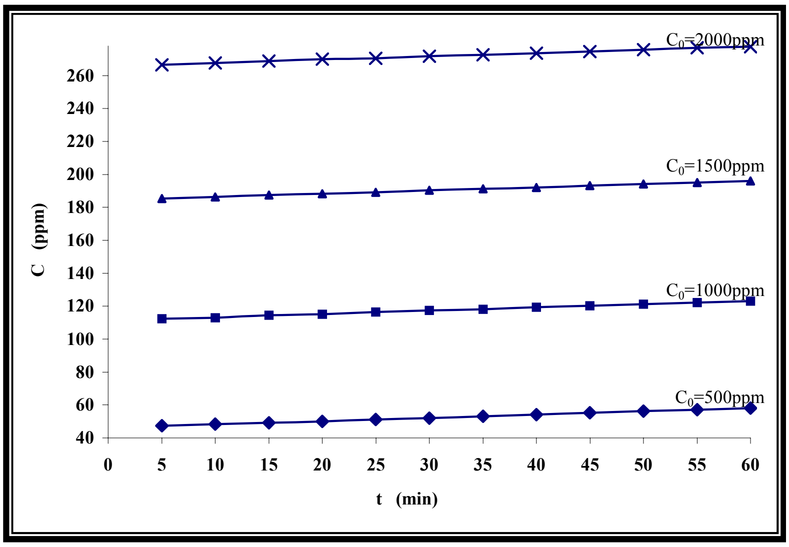

Figure (9) The effect of SO_2 initial concentration on the effluent concentration of SO_2 ($T= 80\ ^0C$)

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in dry bed condition

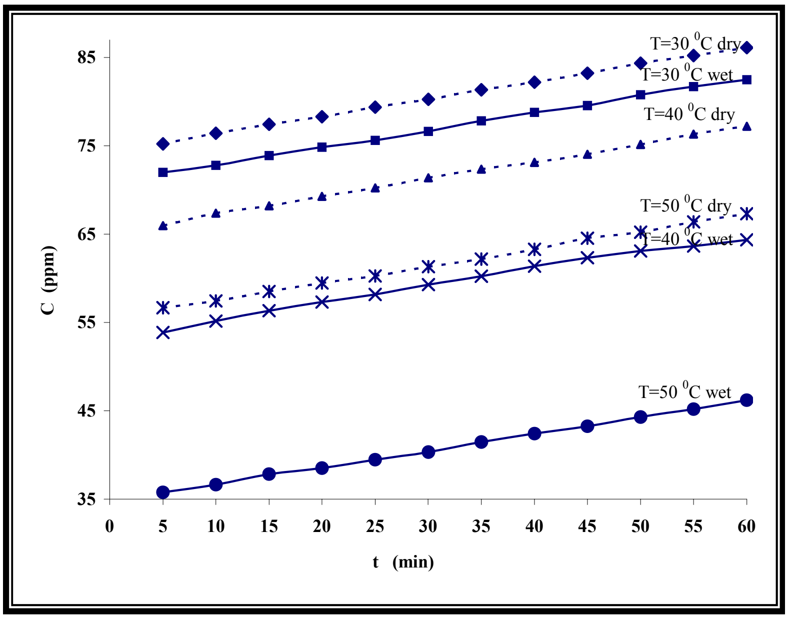


Figure (10) The effect of wet and dry conditions on the effluent concentration of SO_2 (T=30, 40& 50 ^{0}C), C_0=500ppm

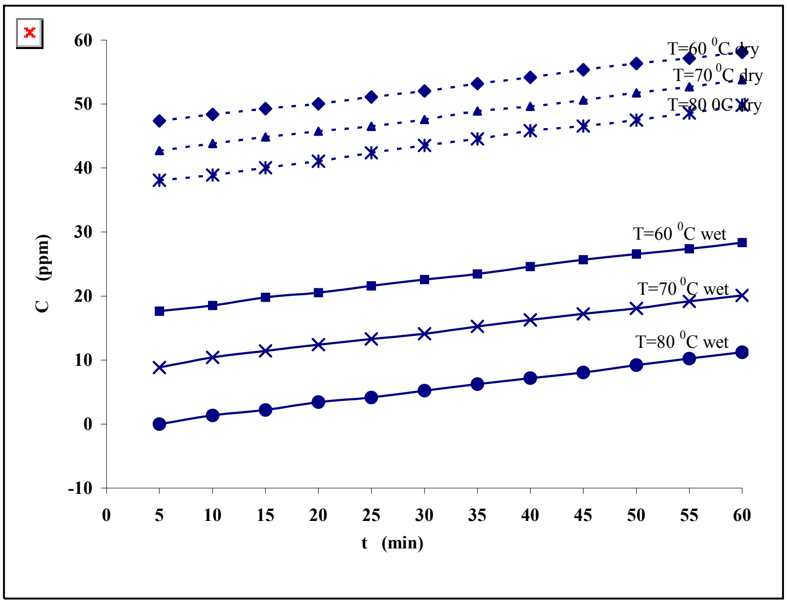


Figure (11) The effect of wet and dry conditions on the effluent concentration of SO_2 (T=60,70 & 80) ^oC,C_0=500ppm

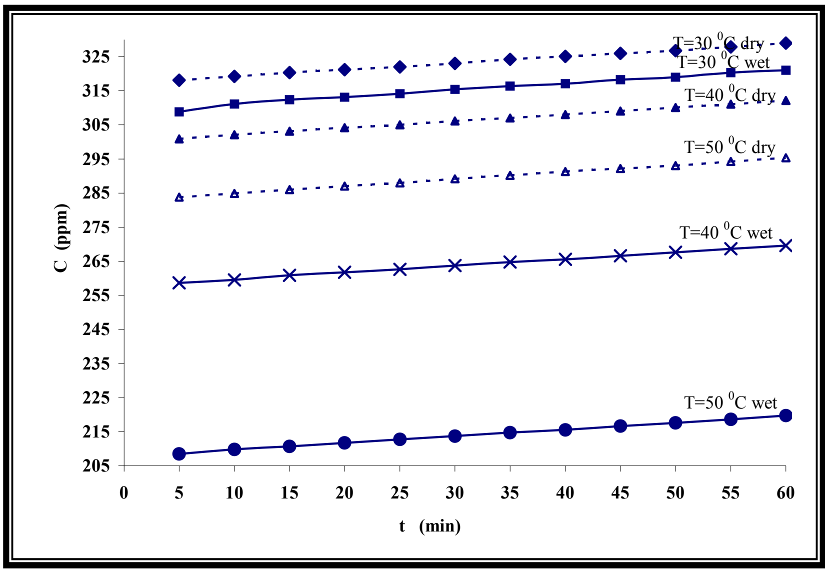
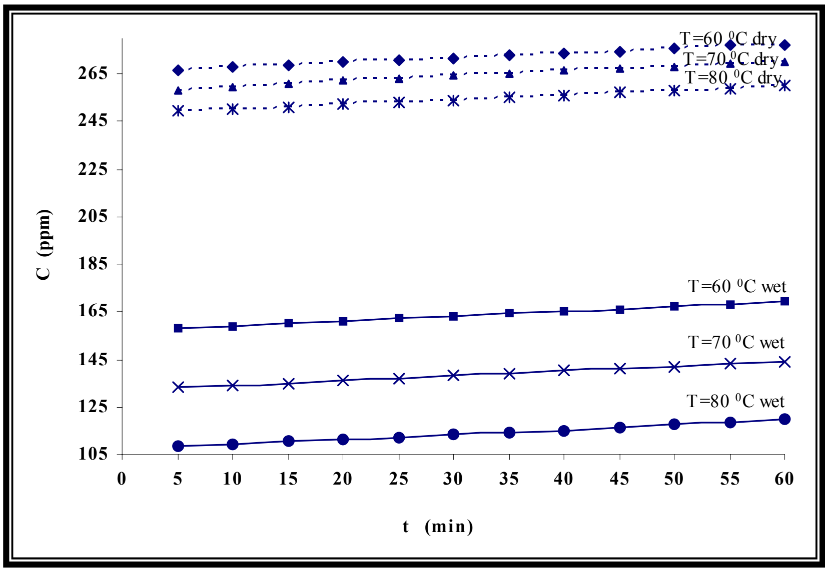


Figure (12) The effect of wet and dry conditions on the effluent concentration of SO_2 (T=30, 40& 50 ^0C), C_0=2000ppm



* Dept. Chemical Eng., Univ. of Tech.

Figure (13) The effect of wet and dry conditions on the effluent concentration of SO_2 ($T=60,70$ & $80\ ^0C$), $C_0=2000ppm$

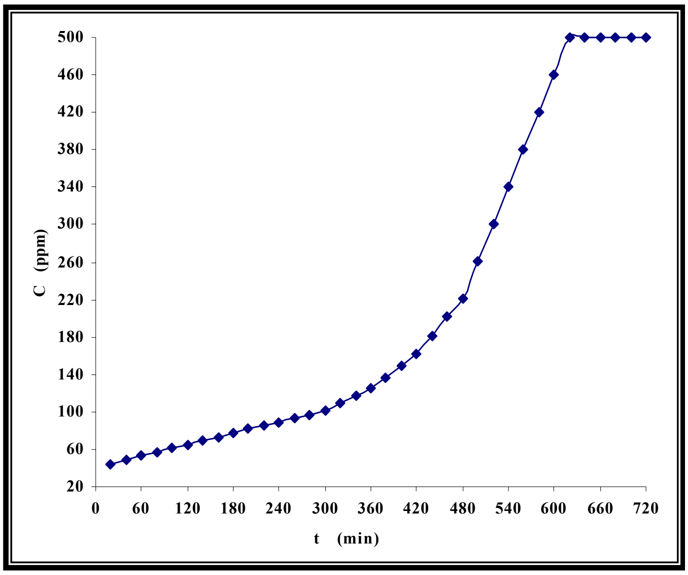
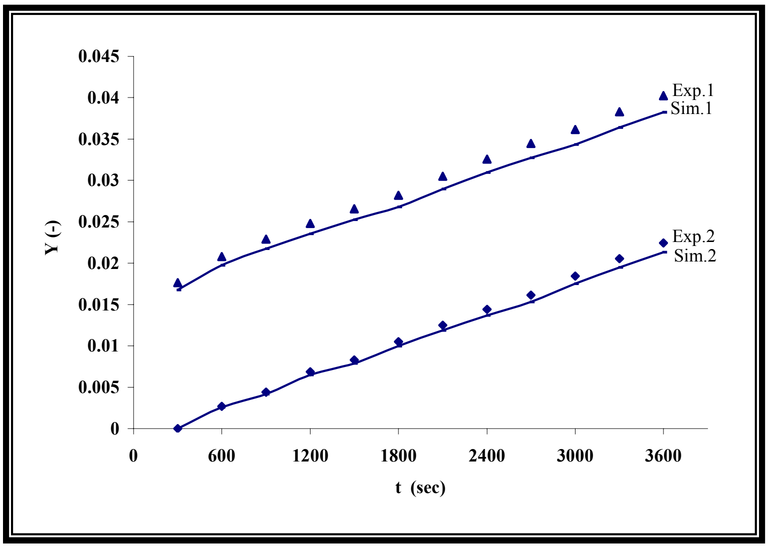


Figure (14) The breakthrough life of 200g of activated carbon at $Q=60$ l/min, $C_o=500$ ppm and $T=80\ ^0C$ in wet bed



* Dept. Chemical Eng., Univ. of Tech.

Figure (15) Comparison between simulated and experimental results for:
Condition1: C_0=500ppm, T=70 ^{0}C in wet bed
Condition2: C_0=500ppm, T=80 ^{0}C in wet bed

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