

Correlations of kinetic parameter for CO₂ sorption using K₂CO₃/Al₂O₃ solid sorbent with various flow regimes/patterns

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Abstract — The kinetic parameters for carbon dioxide (CO₂) capture using a potassium carbonate supported on alumina (K₂CO₃/Al₂O₃) solid sorbent in fluidized bed/circulating fluidized bed were investigated. In this study, the experiment was divided into three parts: (i) varying five different flow regimes/patterns (fixed bed, multiple bubbling, slugging, turbulent and fast fluidization), (ii) varying five different sorption temperatures and (iii) varying four different water contents. The raw data for the kinetic parameter calculation was the breakthrough curve under given conditions. It was found that the selected deactivation kinetic model fitted well with all the experimental data. The correlation of kinetic parameters was observed. The reaction rate constants were highest at the turbulent fluidization flow regime. The values of reaction rate constants between 50 and 70 °C could be represented with the Arrhenius's form. In addition, the initial sorption and deactivation rate constants were increased and decreased with the increasing of water content, respectively, except at the highest water content.

Keywords: breakthrough curve; carbon dioxide; deactivation model; potassium carbonate; solid volume fraction

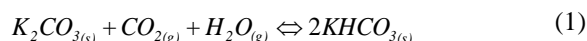
I. INTRODUCTION

Carbon dioxide (CO₂) is known as a significant source of global warming problem due to their ability to maintain the heat inside the earth atmosphere. Recently, the fluidization technology using dry solid sorbent has been considered as a solution for reducing CO₂ release. For conventional gas-solid flow, the flow regimes can be divided into five different patterns with the increasing of gas inlet velocity [1]. Each regime has its own distinct characteristic. Potassium carbonate (K₂CO₃) is frequently selected to use as a solid sorbent because it can easily regenerate using low temperature and economically employed when comparing to the other solid sorbents [2]. In the literature, a lot of research study is so far focused on two main problem areas: solid sorbent development and process system improvement. However, there are still many problems that need to be solved. The understanding of CO₂ sorption reaction rates is one of the important problems because it will help engineer and technologist to better design the reactor [3]. In addition, the information

about the effects of operation conditions such as adsorption temperature, water content, gas composition and gaseous impurities on CO₂ capture behavior was needed [4]. The deactivation model was successfully used to describe the decreasing in activity of alkali-metal carbonate during chemical reaction of various alkali-metal carbonates with CO₂ when comparing to the other model [5-7]. Thus, the deactivation model was selected to compute the kinetic parameters of carbonation reaction for potassium carbonate supported on alumina (K₂CO₃/Al₂O₃) in this study fluidized bed/circulating fluidized bed systems. Then, the effects of flow regime, sorption temperature and water content on the kinetic parameters were discussed. Besides, the correlations of kinetic parameters were explored.

II. THEORY

For the sorption reaction, anhydrous K₂CO₃/Al₂O₃ reacts with CO₂ and moisture (H₂O) to form potassium bicarbonate (KHCO₃) as shown by the following Eq.(1) [5]:



The sorption reaction equation is a heterogeneous gas-solid reaction. The mathematical analysis of this heterogeneous process must take into account the simultaneous influence of the chemical reaction and the heat and mass transfers for correctly predicting the reaction conversion as a function of time. In this study, to calculate the chemical kinetics of Eq. (1), the CO₂ breakthrough curve was employed using the deactivation model as shown below:

A. Breakthrough analysis with the deactivation model

For the deactivation model, the formation of a dense product layer over the solid reactant creates an additional diffusion resistance and is expected to cause a drop in the reaction rate. It then may cause significant changes in the pore structure, active surface area and activity per unit area of solid reactant with reaction extent. All of these changes cause a decrease in the activity of the solid reactant with time. In this model, the effects of all the

changes on the diminishing rate of carbonation are combined in a deactivation reaction rate term. To simulate the removal of CO₂ by solid sorbents, the following assumptions are made:

- (i) The carbonation reaction is operated under isothermal condition.
- (ii) The external mass-transfer limitation is neglected.
- (iii) The pseudo-steady state is assumed.

When the deactivation of the solid sorbent is assumed to be first-order with respect to the solid active sites and zero-order with respect to the concentration of CO₂, the solid activity then can be described as follows:

$$-\frac{da}{dt} = k_d C^m a^n \Rightarrow a = a_o \exp(-k_d t), (m=0, n=1) \quad (2)$$

With the above pseudo-steady state assumption, the isothermal species conservation equation for the CO₂ reactant gas is expressed as follows:

$$-Q_o \frac{dC}{dW} - k_o C a = 0 \quad (3)$$

Integrating Eq. (2), the following equation is obtained.

$$\int_{C_o}^C \frac{dC}{C} = \left(\frac{k_o a}{Q_o} \right) \int_0^W dW \Rightarrow \ln\left(\frac{C}{C_o}\right) = -\left(\frac{k_o a}{Q_o}\right) W \quad (4)$$

Where a is the activity of the solid reactant, t is the time, k_d is the deactivation rate constant, k_o is the initial sorption rate constant, Q_o is the gas flow rate, W is the weight of solid sorbent, C is the outlet concentration of CO₂ and C_o is the inlet concentration of CO₂.

Combining Eq. (2) and Eq. (4), Eq. (5) then can be obtained.

$$C = C_o \exp\left[-\frac{k_o W}{Q_o}\right] \exp(-k_d t) \quad (5)$$

Arranging Eq. (5), the following equation (Eq. (6)) is obtained:

$$\ln\left[\ln\left(\frac{C}{C_o}\right)\right] = \ln\left(\frac{k_o W}{Q_o}\right) - k_d t \quad (6)$$

Thus, if $\ln[\ln(C_o/C)]$ is plotted versus time, a straight line should be obtained with a slope equals to $-k_d$ and an intercept equals to $\ln(k_o W/Q_o)$, from which k_o then can be obtained.

To obtain analytical solution of Eq. (2) and Eq. (3) by taking $m = n = 1$, an iterative procedure is need to be applied. With this procedure, the Eq. (5) is substituted into Eq. (2), and the first correction for the activity is obtained by the integration of this equation. Then, the corrected activity expression is substituted into Eq. (3), and the integration of Eq. (3) gives the first corrected solution.

$$\frac{C}{C_o} = \exp\left[1 - \frac{\exp\left(\frac{k_o W}{Q_o}(1 - \exp(-k_d t))\right)}{1 - \exp(-k_d t)}\right] \exp(-k_d t) \quad (7)$$

The iterative procedure can be repeated for further improvement of the solution. In this procedure, higher-order terms in the series solutions of the integrals are neglected. The two parameters of k_o and k_d are then calculated from the concentration profiles.

III. METHODOLOGY

A. Solid sorbent preparation

The solid sorbent in this study was prepared by the impregnation of K₂CO₃ on porous alumina support (Al₂O₃). Five grams of support were added to an aqueous solution containing five grams of K₂CO₃ in 25 ml of de-ionized water. Then, it was mixed together in a shaker at room temperature for 24 hours. After mixing, the mixture was dried in vacuum oven at 105 °C and calcined in a furnace at 300 °C with the ramping rate of temperature of 3 °C/min. The amount of alkaline metal impregnated was determined by using a Varian spectra AA 6800 atomic absorption spectrophotometer. The physical properties of K₂CO₃ solid sorbent used in this study are summarized in Table 1.

Table 1. Prepared K₂CO₃/Al₂O₃ solid sorbent physical properties.

Solid property	Value
BET surface area (m ² /g)	80.92
Actual weight of K ₂ CO ₃ impregnated on Al ₂ O ₃ (wt%)	35
Solid sorbent diameter (d_p , μm)	150
Solid sorbent density (ρ_s , kg/m ³)	3,900

B. Fluidization apparatus

The CO₂ sorption tests were conducted in a lab-scale semi-circulating fluidized bed unit as shown in Fig. 1. The riser of lab-scale semi-circulating fluidized bed made from glass had inside diameter of 0.025 m and height of 0.80 m while the downcomer of lab-scale semi-circulating fluidized bed made from poly-chloride (PVC) had inside diameter of 0.050 m and height of 0.30 m. The ball valve in the transfer line was used to control the solid flow rate from downcomer to riser. Solid output storage was used to keep the solid sorbent entrained from the top of the riser. Ten pressure taps were installed along the height of the riser to measure the system pressure.

In this study, the solid sorbent flow pattern in fluidized bed/circulating fluidized bed were investigated by studying solid volume fraction (ϵ_s) profile along the height of the riser. The ϵ_s was calculated by processing the differential pressure or pressure drop, ΔP , measured at

successive pressure taps. When acceleration contribution and friction effects at the riser wall were discarded, the relationship $\Delta P = \rho_s(\varepsilon_s)gH$ then valid (where g and H being the gravity acceleration and distance between the two pressure taps, respectively [8,9]).

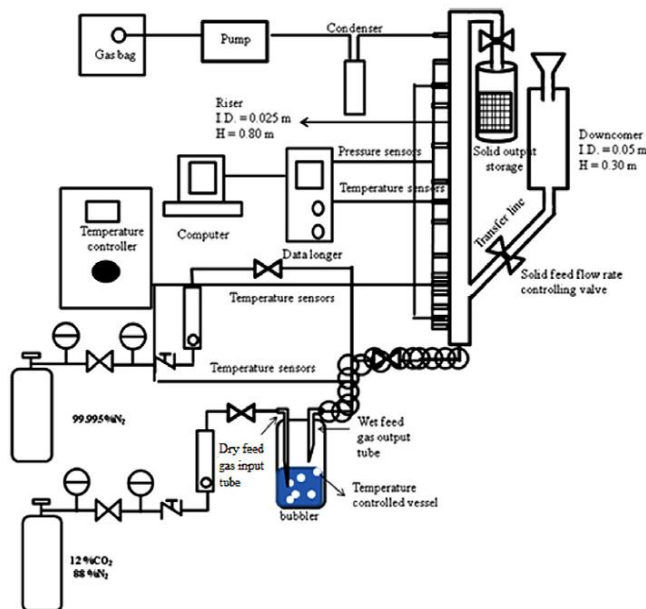


Figure 1. Schematic diagram of the semi-circulating fluidized bed system used in this study [1].

C. Flow regime/pattern characterization

In this study, wide range of gas velocities from 0.01 to 2.64 m/s was investigated. Five different flow regimes or patterns with the increasing of gas velocity were operated including fixed bed, multiple-bubbling fluidization, slugging fluidization, turbulent fluidization and fast fluidization. These include two different operating systems which are non-circulating and circulating. Non-circulating system is consisting of four different flow regimes or patterns: fixed bed, multiple-bubbling fluidization, slugging fluidization and turbulent fluidization. The circulating system can be operated with fast fluidization regime. These flow patterns were already verified in our previous study [10]. Table 2 presents the experimental operating conditions at different gas velocities.

D. CO₂ sorption procedure

For the CO₂ adsorption study, 60 and 300 grams of K₂CO₃/Al₂O₃ solid sorbent were placed into the riser for non-circulating and circulating systems, respectively (as defined in C. Flow regime/pattern characterization). The other experimental conditions of flow regimes or patterns in fluidized bed/circulating fluidized bed on CO₂ sorption is shown in Table 3.

Table 2. Experimental operating conditions of flow regimes/ patterns used in this study.

Fluidization system	Solid Loading (g)	Flow regime /pattern	Gas velocity (m/s)	Solid circulation rate (g/min)
Non-circulating	60	Fixed bed	0.01	-
		Multiple-bubbling	0.20	-
		Slugging	0.66	-
		Turbulent	1.02	-
Circulating	300	Fast fluidization	2.64	30

Table 3. Experimental operating conditions of CO₂ sorption in fluidized bed/circulating fluidized bed.

Condition	CO ₂ sorption
Sorption temperature (°C)	60
Pressure (atm)	1
Flow regime/pattern (-)	fixed bed, multiple-bubbling fluidizations, slugging fluidization, turbulent fluidization and fast fluidization
Gas velocity (m/s)	0.01-2.64
Volumetric flow rate (l/min)	1-30
Solid loading (g)	60 and 300
Gas composition (vol.%)	CO ₂ : 12, H ₂ O: 18.4, N ₂ : balance
Mole fraction of water vapor to CO ₂ (-)	1.50

IV. RESULTS AND DISCUSSION

A. Effect of various flow regimes/patterns

The effect of various flow regimes/patterns on CO₂ breakthrough curves of K₂CO₃/Al₂O₃ solid sorbent in fluidized bed/circulating fluidized bed at sorption temperature of 60 °C in 12 vol.% of CO₂ and 18.4 vol.% of H₂O is shown in Fig. 2. For the CO₂ capture capacity under fixed bed flow pattern, the CO₂ breakthrough curve was constant at the beginning stage then it gradually decreased with the increasing of sorption time. For multiple-bubbling flow regime, the CO₂ breakthrough curve was similar to the one with fixed bed flow pattern. However, the constant sorption time in multiple-bubbling flow regime was shorter than fixed bed flow pattern. This can be explained by the gas velocity in the multiple-bubbling flow regime is higher than that of fixed bed flow pattern. For slugging and turbulent flow regimes, the CO₂ breakthrough curve decreased sharply from the beginning stage with the increasing of sorption time because the residence time of gas flowing through the bed is not enough for the mass transfer of water vapor into solid sorbent surface. Though the residence time of gas in turbulent flow regime was shorter than slugging flow regime, the CO₂ sorption on solid sorbents under turbulent flow regime was better. This can be explained by the high contacting surface area of gas-solid sorbent

inside the turbulent flow regime. When the solid sorbents were operated in fast fluidization flow regime, they were elutriated out of the system by the fluidizing gas, recovered, and returned to the bottom of the riser. It took 8 minutes for all 300 grams of solid sorbent to pass through the riser. During first cycle or 0 to 8 minutes, the fresh solid sorbent adsorbed about 70 percent ($C_{AO}/C_A = 0.3$) of CO₂ in the feed gas since the operating gas velocity is extremely high. After 8 minutes, the sorption reaction of solid sorbents gradually decreased due to the returning of solid sorbents.

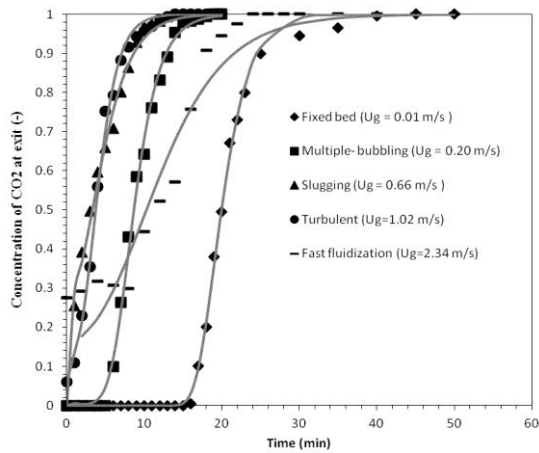


Figure 2. Effect of different flow regimes/patterns on breakthrough curve of CO₂ concentration in 12 vol% of CO₂ and 18.4 vol% of H₂O and sorption temperature of 60 °C.

To obtain kinetic parameters (k_0 and k_d), the regression fitting was performed using Eq. (7) with non-linear least square technique. Fig. 2 also displays the regression results of the experimental data by the deactivation model. The selected deactivation model could accurately predict the breakthrough behaviors for all the reaction of K₂CO₃/Al₂CO₃ and CO₂. Table 4 summarizes the reaction rate constant data for all flow regimes/patterns. The kinetic parameters were highly depending on the flow regime/pattern operated in the reactor. When considering simulated CO₂ breakthrough curve as shown in Fig. 2, the fixed bed flow pattern still gave the best sorption result due to the high sorption time. Even if the fixed bed flow pattern had the best result for CO₂ capture, the value of k_0 was very poor. The solid sorbent packing on each other causes the loss of active site of solid sorbent and low speed of reaction rate. In the case of multiple-bubbling flow regime, the value of k_0 in this regime showed that the gas-solid behavior or the higher gas-solid contacting surface area gave better k_0 . For slugging, turbulent and fast fluidization flow regimes, the solid sorbent could not remove all CO₂ in feed gas except only at early beginning stage. Considering the values of k_0 , the slugging and fast fluidization flow regimes showed similar k_0 value range with multiple-bubbling flow regime due to the large bubble formation and high system back-mixing,

respectively. The turbulent flow regime provided most promising value of k_0 because of both the suitable residence time and system back-mixing [11,12]. In addition, the value of k_d or deactivation rate constant had the same trend similar to the value of k_0 or initial sorption rate. For the fixed-bed and multiple-bubbling flow patterns, the k_d values were lower than k_0 values while the opposite results were observed for the other three flow regimes due to low system residence time. This implies the low solid sorbent deactivation in the last three flow regimes causing by the system hydrodynamics.

Table 4. Effect of different flow regimes/patterns on initial sorption rate and deactivation rate in 12 vol.% of CO₂ and 18.4 vol.% of H₂O and sorption temperature of 60 °C.

Flow pattern	W (g)	k_0 (m ³ /kg·min)	k_d (m ³ /kmol·min)	Averaged solid volume fraction (-)
Fixed bed	60	0.301	0.407	0.60
Multiple-bubbling	60	0.434	0.463	0.52
Slugging	60	0.446	0.390	0.29
Turbulent	60	1.005	0.600	0.15
Fast fluidization	300	0.432	0.180	0.09

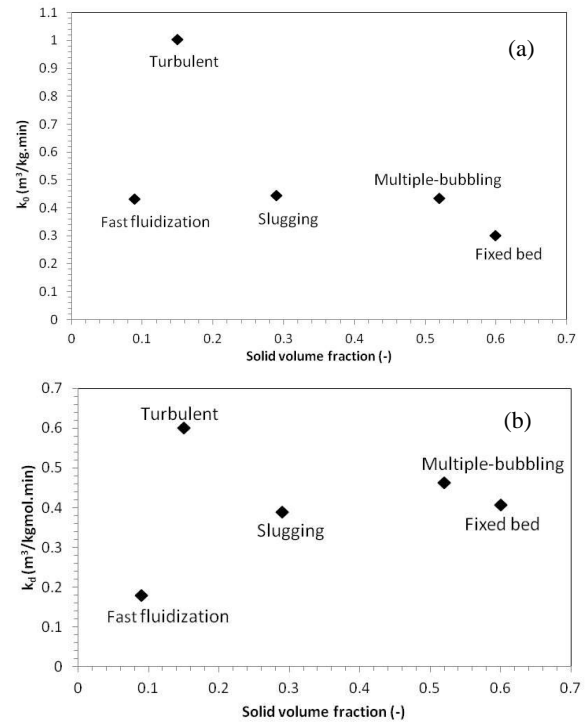


Figure 3. Effect of solid volume fraction on (a) initial sorption rate constant (k_0) and (b) deactivation rate constant (k_d).

The effect of solid volume fraction on the kinetic parameters was explored as shown in Fig. 3. From the figure, the highest kinetic parameters then can also be explained by the appropriate solid volume fraction inside the system.

B. Effect of sorption temperature

The effect of sorption temperature on CO₂ breakthrough curves using K₂CO₃/Al₂O₃ solid sorbent was investigated in a fixed bed flow pattern as shown in Fig. 4. In this study, five different sorption temperatures were used (50, 60, 70, 80 and 90 °C) at the CO₂ concentration of 12 vol.% and water concentration of 18.4 vol.%. From the figure, two trends of concentration of CO₂ at exit were observed. For temperature range of 50 to 60 °C, CO₂ capture capacity increased, while, for temperature range of 60 to 90 °C, CO₂ sorption decreased. Because the carbonation reaction is reversible and highly exothermic, the low CO₂ capture capacity at a lower and higher temperature is attributed to the backward reaction of Eq. (1).

The values of k_0 and k_d were then calculated using the same method in the previous section. As shown in Table 5, the k_0 increased with the temperature from 50 to 70 °C. When increasing the temperature to above 70 °C, the rate of reaction became unfavorable. The sorption temperature of 70 °C was the optimum condition. The overall trend of k_d was increased with the increasing of temperature except at sorption temperature of 50 °C. The smallest observed value was at 60 °C. The dense product layer is the reason for this phenomenon. The relationship between kinetic parameters and temperature (T) can be correlated using the Arrhenius form [13]. In this study, the data values of k_0 at 50, 60 and 70 °C and k_d at 50, 60, 70, 80 and 90 °C were correlated. The $\ln k_0$ vs T^{-1} and $\ln k_d$ vs T^{-1} plots are shown in Figure 5(a) and Figure 5(b), respectively. For initial sorption rate, the regression found the activation energy of 12.66 kJ/mol and frequency factor of 25.81 m³/kg.min, while, for the deactivation rate, the regression found the activation energy of 17.40 kJ/mol and frequency factor of 182.91 m³/kmol.min.

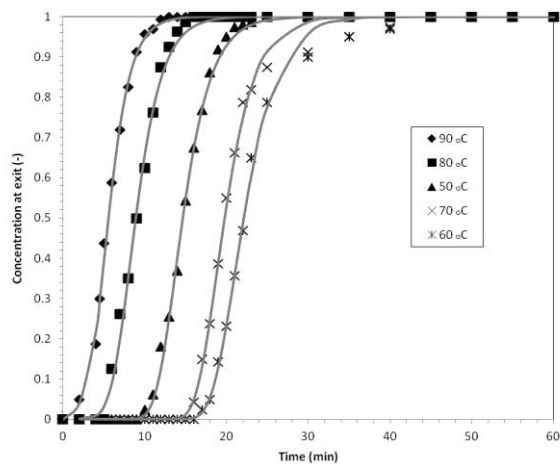


Figure 4. Effect of different sorption temperatures on breakthrough curve of CO₂ concentration in 12 vol.% of CO₂ and 18.4 vol.% of H₂O.

Table 5. Effect of different sorption temperatures on initial sorption rate and deactivation rate in 12 vol.% of CO₂ and 18.4 vol.% of H₂O and sorption temperature of 60 °C.

Sorption temperature (°C)	k_0 (m ³ /kg.min)	k_d (m ³ /kmol.min)
50	0.223	0.418
60	0.285	0.348
70	0.293	0.402
80	0.150	0.471
90	0.117	0.589

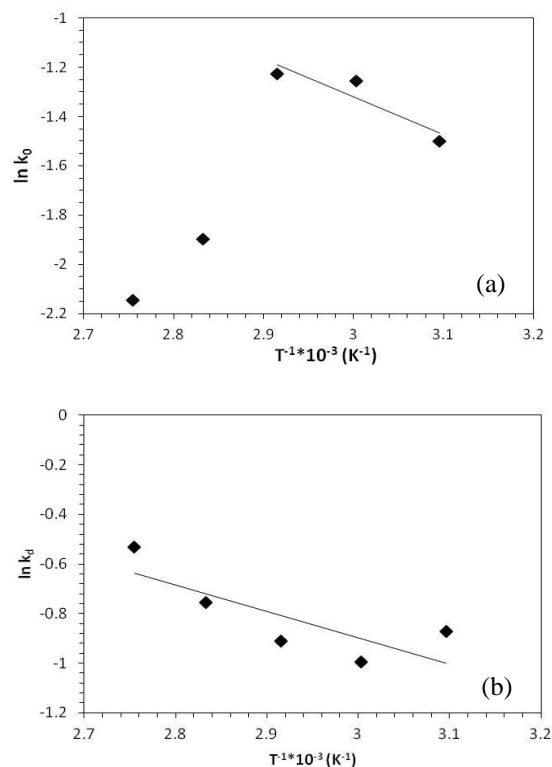


Figure 5. Determination of kinetic parameters in Arrhenius form of (a) initial sorption rate and (b) deactivation rate.

C. Effect of water content in feed gas

The effect of water content in feed gas on CO₂ breakthrough curves using K₂CO₃/Al₂O₃ solid sorbent was investigated in a fixed bed reactor. In this study, four different water contents were carried out as 7.0, 13.5, 18.4 and 22.5 vol.%. The measured values of the CO₂ breakthrough curves are plotted against the time in Fig. 6. Two trends of CO₂ concentration at exit could be observed. For 7.0 to 18.4 vol.% H₂O, the CO₂ sorption capacity increased with increasing water content. This result means that the sorption reaction conversion increases as the increasing of water content. For 18.4 to 22.5 vol.% H₂O, the CO₂ sorption capacity decreased with increasing water content. The exceptionally excess water content exhibits the proceeding of sorption reaction.

The evaluated values of k_o and k_d were reported in Table 6. The simulated curves using the computed kinetic parameters are also shown in Fig. 6. The values of k_o increased with the increasing of water content from 7.0 to 18.4 vol% H₂O. Stoichiometrically, one mole of K₂CO₃ can adsorb one mole of CO₂ and one mole of H₂O (Eq. (1)) and produce two moles of KHCO₃. The effect of water content then can be explained by the above stoichiometric reaction mechanism. However, some excess water content may assist the happening of the sorption reaction as stated by Seo et al. [4]. As the concentration was increased to 22.5 vol.% H₂O, the value of k_o decreased. It can be explained by the extremely surplus water content in the feed gas. This will then exhibit the rate of sorption reaction. In addition, all the values of k_d decreased with the increasing water content. From the previous literature, some scientist reported that excess water can decrease solid sorbent activation at the surface of the sorbent [14].

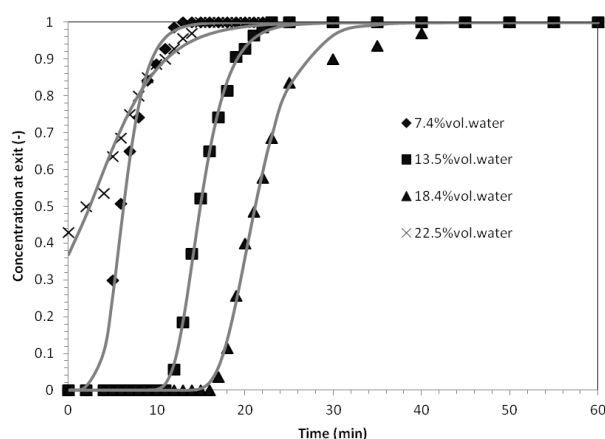


Figure 6. Effect of different water contents on breakthrough curve of CO₂ concentration in 12 vol% of CO₂ and sorption temperature of 60 °C.

Table 6. Effect of water contents on initial sorption rate and deactivation rate in 12 vol.% of CO₂ and sorption temperature of 60 °C.

Water content (vol.%)	Mole fraction of water vapor to CO ₂ (-)	k_o (m ³ /kg·min)	k_d (m ³ /kgmol·min)
7.0	0.58	0.135	0.602
13.5	1.08	0.250	0.455
18.4	1.50	0.261	0.355
22.5	1.88	0.038	0.249

V. CONCLUSION

The kinetic parameters for carbon dioxide capture using a potassium carbonate supported on alumina solid sorbent in fluidized/circulating fluidized bed had been successfully computed under different flow regimes/patterns using deactivation kinetic model. The obtained reaction rate constants was highest at moderate solid volume fraction value (or turbulent flow regime) due to

the system hydrodynamics including suitable gas-solid contacting area and residence time. The values of reaction rate constants between 50 and 70 °C could be represented with the Arrhenius's form. The initial sorption and deactivation rate constants were increased and decreased with the increasing of water content, respectively, except at the highest water content.

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