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Simulation on the Breakthrough Curve During CO_2 Adsorption from Biogas in a Fixed Bed Column

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Abstract. Separation of CO_2 gas from the biogas can be accomplished by adsorption process. An adsorbent has a maximum capacity, so the adsorbent will eventually be saturated at a certain time. Therefore, it is necessary to simulate the adsorption mass transfer to produce a breakthrough curve. The breakthrough curve from the simulation of adsorption mass transfer was validated with the experimental data. The research was conducted using variations in temperature and pressure. The mass transfer simulation was solved using the finite difference method. The adsorbent used in this research was 13X zeolite and the biogas was obtained from cow dung waste. Convergent curves can be obtained in numerical simulations as breakthrough curves. This research shows that adsorption should occur at low temperatures and high pressure. Column height and flow velocity also influence the breakthrough time. The comparison of the simulated breakthrough time with experimental data is not much different with R^2 0.9969. The striking difference is in the adsorption zone with average relative error (ARE) values ranging from 9.57% to 20.49%. From the results of entropy, enthalpy, and Gibbs free energy calculations, the biogas adsorption research on the 13x zeolite column is an exothermic and spontaneous process.

Keywords: Adsorption, Biogas, Breakthrough Curve, Finite Element, Mass Transfer

INTRODUCTION

Biogas is a form of renewable energy currently derived from the anaerobic decomposition of organic matter. The main components of biogas are methane gas (CH_4), around 65%; carbon dioxide gas (CO_2), about 40%; and other gases such as nitrogen (N_2) 1% and hydrogen sulfide (H_2S), around 24 ppm (Ullah Khan *et al.*, 2017). The gas component in biogas used as an energy source is methane. The presence of significant CO_2 gas in biogas will reduce the energy value of biogas so that the use of biogas as an energy source is only for purposes that require low energy. Therefore,

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the quality of biogas needs to be improved so that it can be widely used for energy purposes (Kapoor *et al.*, 2019)

One technique to separate CO_2 gas from biogas is adsorption. Adsorption is a mass transfer phenomenon from the gas to the solid phase. Adsorption is carried out on a vertical fixed bed column filled with adsorbent and pressurized gas is flowed into the column so that gas separation occurs (Andriani *et al.*, 2014). As long as the size of CO_2 gas is smaller than other components of biogas gas, more CO_2 gas will be adsorbed than other biogas gas components (Khayyun and Mseer, 2019). CH_4, CO_2, O_2, and N_2 molecular size are 4.0, 2.8, 2.8, and 3.0 Å, respectively, at standard conditions (Yang *et al.*, 2014). Solids that can adsorb are called adsorbents. The adsorbent has a site or pore where the adsorbate or gas can stick. The number of sites on the adsorbent is limited, so the adsorbent will be saturated or unable to adsorb gases (Sahoo and Prelot, 2020).

In the adsorption process, the time when the adsorbent cannot adsorb gas or the saturation time is very important to find first so that the quality of the adsorption results is as desired. One way to determine the saturation time is by simulating the adsorption mass transfer (Kim *et al.*, 2015). There are three zones in the gas adsorption mass transfer in the column: clean zone, the adsorption zone, and the saturated zone (El-Naas and Alhaija, 2013). Figure 1 shows three zones on the adsorption column during the adsorption process.

At the beginning of time, a lot of CO_2 gas will be adsorbed to the adsorbent at the bottom while the adsorbent at the top of the column is still in the clean zone. In the middle of time, the adsorbent at the bottom of the column will be saturated because the adsorbent can no longer adsorb CO_2 gas. In contrast, the adsorbent in the middle of the column in the adsorption zone or adsorbent still has a site that can be occupied by CO_2. At the end of time, all adsorbents in the column will be saturated, or the entire adsorbent site has been filled with CO_2 gas so that the composition of CO_2 gas entering the column is the same as that leaving the fixed bed column.

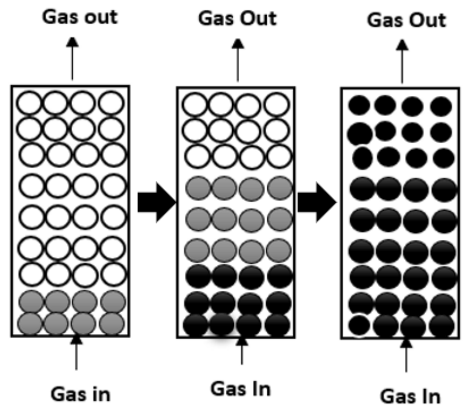


Fig. 1: Three adsorption zones on the column; white is the clean zone, gray is the adsorption zone, and black is the saturated zone

Mass transfer occurs when the gas is in the fixed bed column, including mass transfer by axial dispersion, convection flow or advection, and adsorption. Axial dispersion depends on the value of the molecular diffusivity between the two gases, the Schmidt number, and the Reynolds number. Advection depends on interstitial velocity. The amount of gas adsorbed depends on the adsorption rate constant. The adsorption rate constant was obtained from the adsorption kinetics model. The adsorption kinetics model often used is the linear driving force (LDF) or the pseudo first order (PFO) model. The obtained linear regression method obtained the adsorption rate constant (Shafeeyan *et al.*, 2014).

The adsorption mass transfer simulation will produce a breakthrough curve. The breakthrough curve is the ratio of the gas concentration leaving the column with the gas concentration entering the column time; this curve is in the shape of an "S" and shows three zones of the adsorption process at the outlet fix bed column (Siqueira *et al.*, 2017). The breakthrough curve also provides information about the breakthrough time, when the concentration of the adsorbate coming out of the column outlet increases.

The novelty of this research is that it enriches information on the effectiveness of CO_2 separation in biogas through the adsorption process with modeling and simulation so that the CH_4 purity level becomes higher. So, it can enrich information on biogas purification technology, for example, pressure swing adsorption.

This research conducts simulations and experiments on the adsorption of CO_2 gas on a fixed bed column with a certain pressure and temperature. This research compares experimental data from previous research (Al Kindi *et al.*, 2023) with simulations of breakthrough curves for CO_2 gas adsorption on biogas onto 13x zeolite in columns. Studies on adsorption thermodynamics are included in this research.

METHOD

Simulation

The simulation model was developed based on the gas mass balance equation for the bulk flow in a fixed column, as given by Equation (1) (Kim *et al.*, 2015; Siqueira *et al.*, 2017).

$$D_{ax}\frac{\delta^2 C_i}{\delta z^2} - \frac{(1-\varepsilon)}{\varepsilon}\rho_s\frac{\delta q_t}{\delta t} - \frac{\delta(uC_i)}{\delta z} = \frac{\delta C_t}{\delta t} \quad (1)$$

The equation is to be solved using boundary condition, as in Equations (2) and (3), and initial condition, as in Equations (4) and (5).

$$D_{ax}\left(\frac{C_i}{z}\right)\bigg|_{z=0} = -u(C_{x=0-} - C_{x=0+}) \quad (2)$$

$$D_{ax}\left(\frac{C_i}{z}\right)\bigg|_{z=L} = 0 \quad (3)$$

$$C_{i(x,t=0)} = 0 \quad (4)$$

$$q_{CO2(x,t=0)} = 0 \quad (5)$$

Where D_{ax} is the mass axial dispersion coefficient (cm^2/s), which is obtained from Equations (6), (7) and (8), C_i is the CO_2 concentration in the gas phase (mg/mL), ε is the bed porosity which is obtained by dividing the total volume of zeolite 13X by the volume of the column, ρ_s is the density of adsorbent (g/mL), u is the interstitial velocity (m/s) which is obtained from Equation (9), dan C_t is CO_2 concentration coming out of the column (mg/mL), D_m is the molecular diffusivity (cm^2/s), Re is the Reynold number, Sc is the Schmidt number, ρ is the density of the gas (kg/m^3), d_p is the diameter of zeolite (m), μ is the viscosity of the gas, Q is the volume rate (L/s), and A is the cross section flow area. Equation (2) is a parabolic form of diffusion-advection with two variables, namely distance (δ_z) and time (δ_t) and can be solved using an explicit finite difference scheme and supporting equation.

$$\frac{\varepsilon D_{ax}}{D_m} = 20 + 0.5ReSc \quad (6)$$

$$Re = \frac{\rho g \varepsilon u d_p}{\mu};\ Sc = \frac{\mu}{\rho g D_m} \quad (7)$$

$$u = \frac{Q}{A.\varepsilon} \quad (8)$$

The amount of CO_2 gas adsorbed each time depends on the value of the adsorption rate constant from the adsorption kinetics model. This study uses a linear flying force (LDF) model (Equation 9) as the adsorption kinetics model or pseudo-first order (Unuabonah *et al.*, 2018).

$$\frac{dq_t}{dt} = k(q_e - q_t) \tag{9}$$

Where k is the adsorption rate constant (1/s), and q_e is the equilibrium adsorption capacity (g of CO_2/g adsorbent). Equation (9) is first converted to linear form Equation (10), and the constant k is obtained by multiplying the slope of the linear line by 2.303, and the value of qe is obtained from the experiment. Validation of adsorption breakthrough curve simulation with experimental data using average relative error (ARE) is described in Equation (11).

$$log(q_e - q_t) = log(q_e) - \left(\frac{k}{2.303}\right)t \tag{10}$$

$$ARE = \frac{100}{N}\sum_{i=1}^{n}\left|\frac{qe_{eks} - qe_{sim}}{qe_{eks}}\right| \tag{11}$$

Experiment

A schematic diagram of the experimental setup is shown in Figure 2. The experimental setup provided an adsorption fixed bed column unit, a gas cooling unit, and a gas cleaning unit. The fix bed adsorption column was made of steel columns with 0.3 m height and 0.03 m diameter, and a cartridge heater of 400 W. Zeolite 13X was used as an activated adsorbent and replaced for every experimental run. Zeolite 13X inside the adsorption column was heated by a cartridge heater, and the PID and SSR controls kept the temperature constant. The working pressure in the adsorption column was 2.2, 2.6, and 3 bar, and the temperature variation was 30°C, 50 °C, and 100°C. Other tools that support this research include a flow meter, hose, pressure gauge, gas analyzer (portable infrared syngas analyzer gas board-3100P), gas vessel, biogas balloon, temperature controller, and globe valve.

Data collection of CO_2 gas from the fixed bed column is carried out continuously, and the biogas flow rate is 4 L/min. The amount of gas adsorbed by zeolite at any time (qt) was calculated using Equation (12). The biogas sampled came from a cow dung digester and were then stored in a biogas balloon.

$$q_t = \frac{C_o - C_t}{m_s} \tag{12}$$

Adsorption Thermodynamics

The study of adsorption thermodynamics is useful for understanding the nature of adsorption, which is obtained by calculating thermodynamic parameters such as enthalpy (ΔH), entropy (ΔS) and Gibbs free energy (ΔG). The negative enthalpy value of the adsorption process can explain that adsorption is exothermic (releases heat), and the large value can determine the adsorption type (Rashidi *et al.*, 2013). The value of the Gibbs free energy can explain spontaneous or non-spontaneous adsorption processes (Azizian *et al.*, 2018a). The enthalpy and entropy values can be obtained using Equation (13), and the Gibbs free energy is found using Van't Hoff equation (14).

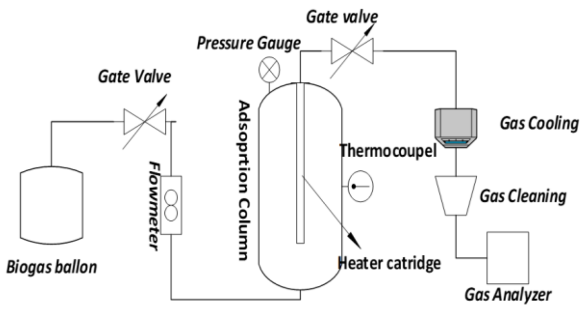


Fig. 2: Scematic diagram of experimental biogas adsorption system

$$\ln K_{eq} = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (13)$$

$$\Delta G = -R.T.Ln\, K_{eq} \quad (14)$$

where K_{eq} is the adsorption equilibrium constant, T is temperature, and R is the gas constant.

RESULTS AND DISCUSSION

Experimental Data

Gas flows into the column with the gate valve closed until the experimental pressure was reached (2.2 bar, 2.6 bar, and 30 bar). Next, the gate valve was opened slowly, so that the pressure in the column was maintained. The gas that comes out of the column was biogas, which has a higher CH_4 value than before entering the column. Next, the CO_2 content was read by a gas analyzer in real time for 3 minutes. Figure 3 shows the CO_2 gas breakthrough curve experimentally at 30°C at various pressures. From this figure, a higher adsorption pressure leads to longer breakthrough times. This is because it takes longer for the CO2 concentration front to reach the bed outlet.

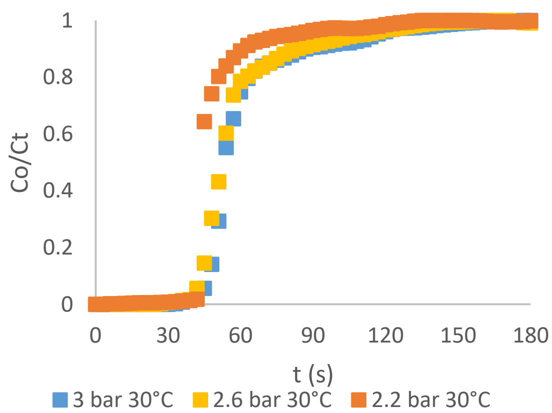


Fig. 3: Breakthrough curve from the experiment at with pressure variation

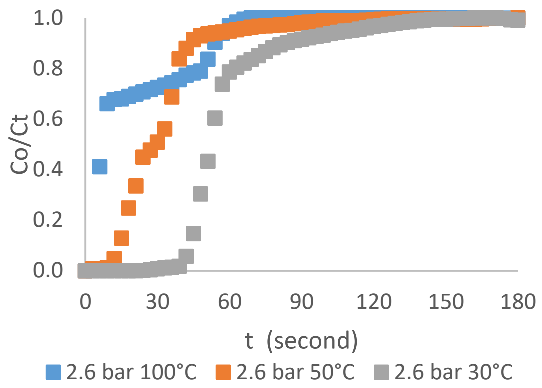


Fig. 4: Breakthrough curve from the experiment with temperature variation

Figure 4 shows the CO_2 gas breakthrough curve from the experimental results at a pressure of 2.6 bar at various temperatures. Gas flows into the column with the gate valve closed until the experimental pressure was reached (2.6 bar). Next, the gate valve was opened slowly with the pressure in the column maintained constant and the temperature maintained at 25°C, 50 °C, and 100 °C. Next, the CO_2 content was read by a gas analyzer in real time for 3 minutes. From the figure, it can be concluded that the higher the temperature, the faster the zeolite will saturate. High temperatures will damage the adsorption process because the pores of the 13X zeolite will shrink when given a high temperature so that only a small amount of CO_2 gas is adsorbed, and the rest leaves the column without adsorption. Breakthrough time percentage change in the 50°C, and 100°C breakthrough temperatures decreased by around 75% and 85%, respectively.

Simulation and Validation Data

Table 1 shows the parameters used in the simulation at a temperature of 30°C. The interstitial velocity was obtained from the calculation of Equation (8). Column porosity is obtained by dividing the column volume by the total zeolite volume. Molecular diffusion is the diffusion between CO_2 gas and air. The simulation can converge because it meets the conditions. The reaction rate constant's value is obtained by using a linear driving force model (LDF) or pseudo first order from experimental data.

The simulation of the adsorption mass balance on the fixed bed column was started by discretizing Equation (2) to the form of Equation (15) so that it could be solved using a finite difference with an explicit scheme.

$$C_z^{t+1} = C_z^t - \frac{u\Delta t}{\Delta z}(C_z^t - C_{z-1}^t) + \frac{D_{ax}\Delta t}{\Delta z^2}\left(C_{z+1}^t + C_{z-1}^t - 2 * C_z^t\right) - \frac{(1-\varepsilon)}{\varepsilon}\rho_s\frac{\Delta q_t}{\Delta t} \quad (15)$$

The simulation will converge if the von Neumann stability is fulfilled in Equations (16), (17), and (18).

$$R = \frac{u\Delta t}{\Delta Z} \quad (16)$$

$$S = \frac{D_{ax}\Delta t}{\Delta Z^2} \quad (17)$$

$$1 - R - 2S \geq 0 \quad (18)$$

Table 1. Simulation parameters

Parameter	Value	Unit
Volume rate	4	L/min
Bed length (z)	0.3	m
Bed radius	0.03	m
Superficial velocity	0.023	m/s
Interstitial velocity (u)	0.049	m/s
Volume of zeolite 13X	0.39	L
Bed porosity (ε)	0.479	
molecular diffusivity CO_2-air (D_m)	$1.6*10^{-5}$	m^2/s
Gas viscosity (µ)	$1.47*10^{-5}$	Pa.s
density of gas (ρg)	1.977	kg/m^3
Adsorben density (ρ_s)	0.27	g/cm^3
Diameter of zeolite	0.0014	m
Schmidt number (Sc)	0.465	
Reynold number (Re)	4.442	
Mass axial dispersion coefficient (D_{ax})	0.422	cm^2/s
Δt	0.3	s
Δz	3	cm
R	0.657	
S	0.018	
Von Neumann stability	0.132	

Figure 5 shows the amount of CO_2 that is adsorbed over time (q_t) from the experimental result (Al Kindi *et al.*, 2023). The amount of CO_2 adsorbed is affected by temperature and pressure. The higher the temperature, the smaller the amount of CO_2 that is adsorbed, while the higher the pressure, the greater the amount of CO_2 that is adsorbed.

The experimental q_e value was obtained when there was no significant change in q_t. The adsorption rate constant (k) value is obtained from the slope of the linear curve log (q_e-q_t) *vs* t in Figure 6. The adsorption rate constant is the product of the slope by 2.303. Table 2 shows the values of q_e and k for each adsorption treatment used to simulate the breakthrough curve.

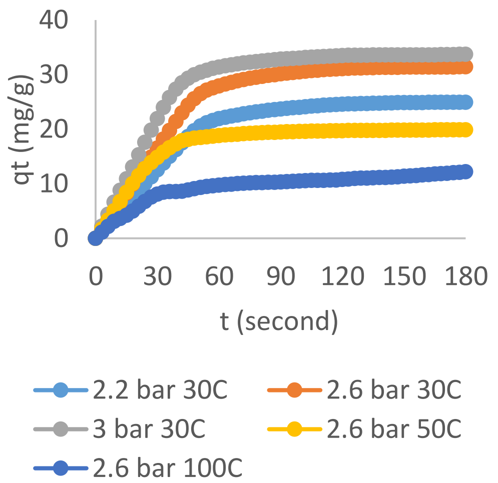
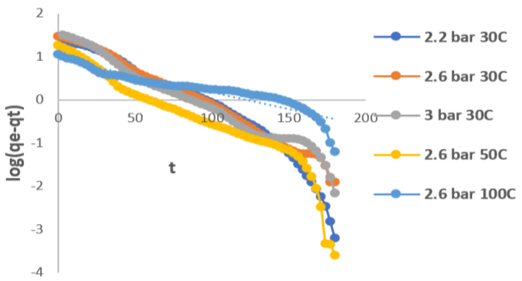


Fig. 5: Adsorption curve



y = -0.0204x + 1.7403 (2.2 bar 30°C)
y = -0.0179x + 1.659 (2.6 bar 30°C)
y = -0.0172x + 1.5076 (3 bar 30°C)
y = -0.0176x + 1.2186 (2.6 bar 50°C)
y = -0.0073x + 0.9413 (3 bar 100°C)

Fig. 6: Linear curve log (q_e-q_t) *vs t*

Table 2. Value of q_e and k

Adsorption treatment	q_e (g of CO_2/g)	k
2.2 bar; 30°C	24.91	0.047
2.6 bar; 30°C	31.43	0.041
3 bar; 30°C	33.7	0.04
2.6 bar; 50°C	19.88	0.04
2.6 bar; 100°C	12.32	0.017

Figure 7 compares the simulation of the breakthrough curve and the experimental data. There is no significant difference in breakthrough time (BT) between the simulation results and experimental data with an R^2 value of 0,9969. However, there are differences in the shape of the adsorption zone or mass transfer zone (MTZ), which is calculated by average relative error (ARE). Validation of the adsorption breakthrough curve simulation with experimental data and breakthrough time if the purity level of CH_4 coming out of the column is 90% is presented in Table 3.

Auta *et al.* (2015) used activated carbon to adsorb gas at varying temperatures of 30°C, 40°C, and 50°C. It was found that the greater the gas temperature, the faster the breakthrough time.

Gibbs Free Energy, Enthalpy, and Entropy

Table 4 shows the energy, entropy, and Gibbs free energy values of CO_2 adsorption in biogas on zeolite 13X. A negative value of the ΔH energy means that adsorption is an exothermic phenomenon or releases heat. When CO_2 gas enters the zeolite pores, the energy possessed by CO_2 is reduced so that it releases heat. The small adsorption ΔH value explains that adsorption is a physical process or adsorption of CO_2 gas onto the zeolite due to Van der Waals forces.

Table 3. Value of ARE

Adsorption treatment	BT_{sim} (second)	BT_{exp} (second)	ARE (%)
2.2 bar; 30°C	42	40	9.57
2.6 bar; 30°C	43	41	6.04
3 bar; 30°C	45	44	20.49
2.6 bar; 50°C	22	24	15.32
2.6 bar; 100°C	6	8	10,02

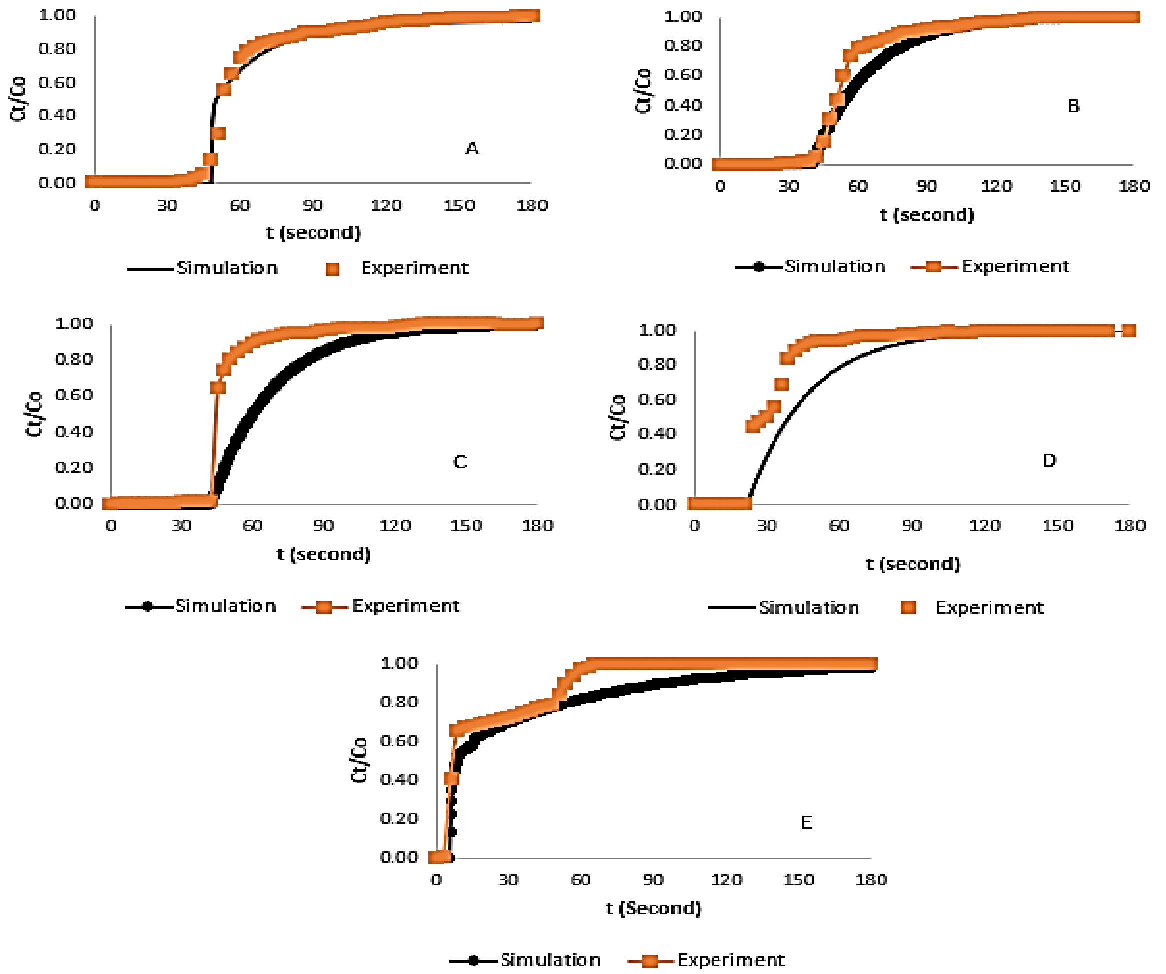


Fig. 7: Comparison of simulation and experimental breakthrough curves. (A) 2.2 bar; 30°C, (B) 2.6 bar; 30°C, (C) 3 bar; 30°C, (D) 2.6 bar; 50 °C, and (E) 2.6 bar; 100 °C

Physical adsorption occurs if the enthalpy value is less than -40 kJ/mol. Positive values of ΔS reflect the affinity of the adsorbent with CO_2. A negative value of the Gibbs free energy ΔG indicates that adsorption is a spontaneous process or a process that occurs from the gas phase to the adsorbent phase. The same results were proven by Azizian *et al.* (2018b), that adsorption produces heat, and the desorption process is very easy to do with adding heat.

Tabel 4. Thermodynamic adsorption properties

Tempe-rature (°C)	Gibbs ΔG kJ/mol	Entropy (ΔS) J/mol.K	Enthalpy (ΔH) kJ/mol
30	-7,795		
50	-7,427	-18,377	-13,360
100	-6,509		

Effect of Column Length and Volume Flow Rate

This subsection will discuss column length and volume flow rate related to breakthrough time using the adsorption mass balance equation. Figure 8 shows the breakthrough curve of the variation in column height at a pressure 3 bar and a temperature of 30°C with zeolite 13X as adsorbent. The higher the column, the longer the breakthrough time, because more and more 13X zeolite adsorbs CO_2 gas. The percentage changes in breakthrough time from 0.33 m to 1 meter and 1.5 meters are 100% and 150%, respectively.

Figure 9 shows the effect of the volume flow rate on the breakthrough curve. The figure shows that faster the biogas flows into the fixed bed column, the smaller the breakthrough time will be. This is biogas volume flow rate, which will increase the mass axial dispersion coefficient value. Mass transfer zone at the volume flow rate of 1 L/min is smoother than at 7L/min because the adsorption zone at high speed will be completed more quickly.

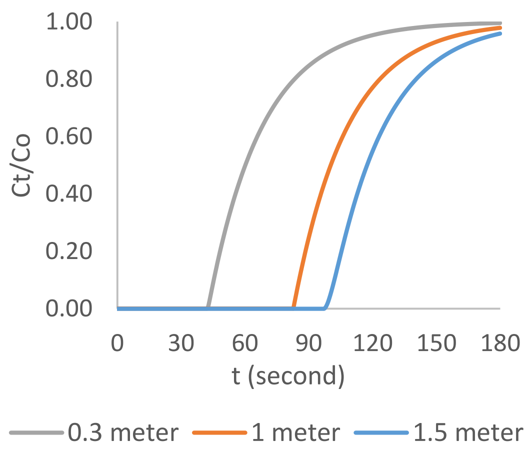


Fig. 8: Effect of bed length on breakthrough curve

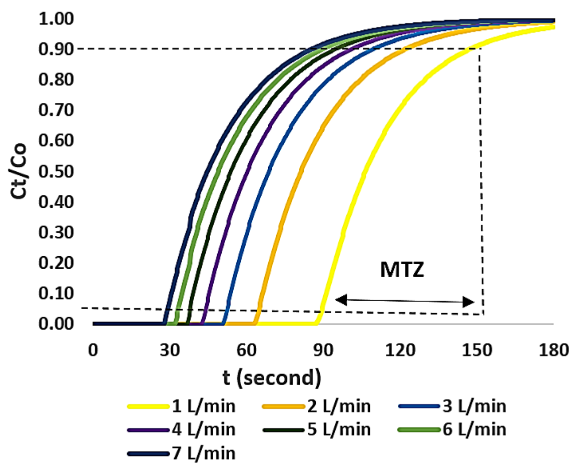


Fig. 9: Effect of volume flow rate on breakthrough curve

CONCLUSIONS

Adsorption in a pressurized column is a mass transfer phenomenon consisting of axial dispersion, convection flow or advection, and adsorption. Simulations can be carried out using the finite difference method. The comparison of the simulated breakthrough time with experimental data is not much different with R^2 0.9882; the striking difference is in the adsorption zone with ARE values ranging from 9.57% to 20.49%. From this research, the adsorption process should be carried out at low temperatures and high pressure.

NOMENCLATURE

c_t	:	concentration of component at any time [mol/cm^3]
C_i	:	concentration of component at initial time [mol/cm^3]
D_{ax}	:	mass axial dispersion coefficient [cm^2/s]
d_p	:	particle diameter [cm]
k	:	the adsorption rate constant [1/s]
Re	:	Reynolds number
Sc	:	Schmidt number

u	:	interstitial linear gas velocity, [cm/s]
Q	:	Volume rate of gas [cm^3/s]
A	:	Cross section area of column [cm^2]
m_s	:	Dosage of zeolite in the adsorption column

Greeks

ε	:	bed porosity
ρ	:	gas density [g/cm^3]
ρ_S	:	adsorbent density [g/cm^3]
μ	:	gas viscosity [Pa s]

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