

# Experimental Verification of Dynamic Modelling of Nitrogen Adsorption on Zeolite 13X with VSA Process

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**Abstract** The four-step vacuum swing adsorption process applying zeolite 13X is studied in order to separate oxygen from air. Effects of the duration of vacuum stage, pressure drop and NMP solvent as a coat of the adsorbent are evaluated. An adsorption model is utilized for isothermal adsorption system by different mechanisms containing local equilibrium and solid diffusion model. Linear isotherm can describe the adsorption process, appropriately. Moreover, linear driving force, LDF, is used as the mass transfer approximation, in solid diffusion model. By this approximation and finite difference solution, ODE equations are solved by Runge-Kutta method using MATLAB. Solid diffusion model utilizing one fitting parameter predicts the breakthrough curve properly more compatible than another theory. The appropriate solid diffusion coefficient utilized as the fitting parameter is  $10^{-10} \text{ m}^2/\text{s}$ . The model accuracy is evaluated, and the error values of equilibrium and solid diffusion model from experimental data are reported 11.7, 1.17, respectively.

**Keywords** Vacuum swing adsorption, Zeolite 13X, Oxygen, Nitrogen, Mathematical model

## 1. Introduction

Oxygen is used in the various aspects such as metallurgical industry, glass-making and ceramic industry, and wastewater treatment [1]. Vacuum/ pressure swing adsorption process (VSA/PSA) is the developed operation to separate and purify gasses, which is based on adsorption ability and selectivity of an adsorbent [5, 6]. In 1970, this method was initially utilized to generate oxygen from air. This technology has advantages compared with various procedures used to separate of air and manufacture oxygen such as cryogenic distillation, membrane and chemical absorption method [2]. In other words, VSA/PSA process is a safe and automatic method, which requires small investment with low energy requirement that would be put in/exit from the service easily [1]. The development of highly selective adsorbents such as LicaX and LiAgX facilitates the generating oxygen up to 200 tons/day by VSA process, which is competitive with cryogenic distillation [2]. Also, VSA process is more economic to produce of over 15 ton/day oxygen in comparison with PSA process [1, 2].

In VSA process, adsorption and desorption pressure, and the adsorbent capacity are key factors to separate of air, efficiently. The optimal vacuum pressure and time play the critical role to reduce the cost of equipment and energy. For

air separation, most industries operate VSA process in the ambient temperature, adsorption pressure of 1.1 to 1.8 atm and desorption pressure of 0.05 to 0.3 atm. Moreover, choosing the regeneration method depends on the adsorbent, adsorption ability and operating condition of the unit [2].

Apart from the experimental investigations, mathematical modeling of VSA process is widely utilized to determine the optimum conditions. Moreover, modeling methods can be useful for process development and scale-up, and provide conceptual procedures to estimate some parameters which are necessary for process design purposes [1, 5, 9].

Linear driving force (LDF) has been applied as a suitable approximation to describe the rate coefficient for extra particle and intra particle mass transfer controlling. Also, intra particle mass transfer can be predicted by four main region: local equilibrium, pore diffusion, solid diffusion, and combined pore and solid diffusion. By these approximation procedures and finite difference method, solid concentration is obtained by solving Ordinary Differential Equation (ODE) equations instead of Partial Differential Equation(PDE) [10, 11].

LDF approximation with linear equilibrium has been utilized to model the PSA process [12, 13]. Pore diffusion model with assumption of linear adsorption isotherm and axial dispersion has been used to simulate non isothermal process [14, 15]. Hwang and Lee have also applied LDF model and binary Langmuir isotherm to predict the experimental breakthrough behaviors of CO and CO<sub>2</sub> on activated carbon in isothermal condition with axial dispersion, plug flow and no radial gradient of concentration

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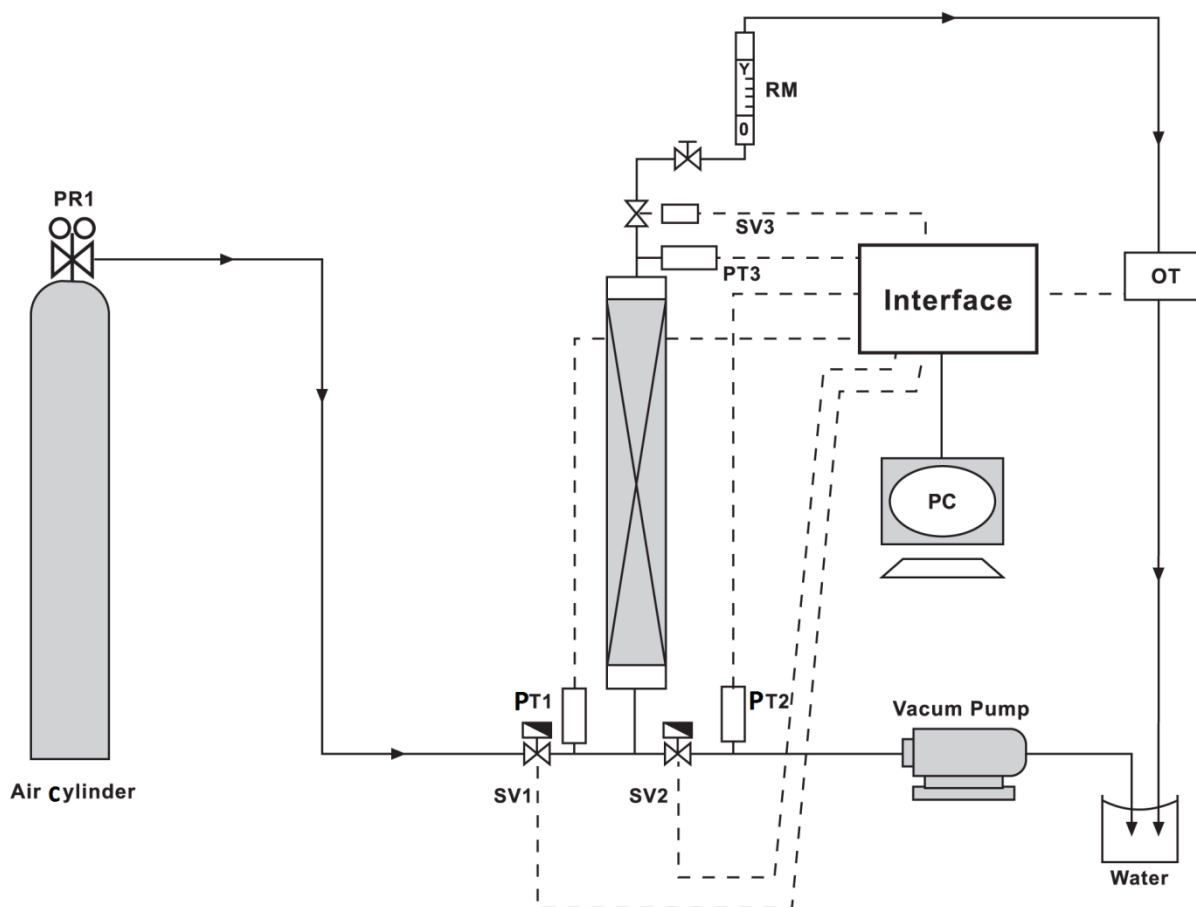
[16]. Moreover, this model has been employed for isothermal PSA process using zeolite in a column with plug flow and no radial gradient of concentration [17, 18]. LDF model with Langmuir isotherm has been studied to predict breakthrough in the fixed bed with axial dispersion and plug flow [19]. A non-isothermal dynamic model using LDF model and the Langmuir-Freundlich isotherm has been applied for VSA process of removing  $N_2$  from air using zeolite 13X and 10X. In this study zeolite 13X has produced product with higher purity [2]. Budner et al. has generated oxygen by VSA process using CaX zeolite, and provided the non-equilibrium model [1].

The main goal of the present work is the investigation of the operating condition of VSA process which is used to adsorb nitrogen from air by zeolite 13X. For this purpose, the experiments operated in consecutive processes are designed to determine the adsorption time and vacuum required, and the appropriate result is presented in this work. Also, two models of VSA process including equilibrium and solid diffusion model are studied. In order to evaluate the model validity and determine the appropriate model, the results from the mathematical model are compared with experimental data.

## 2. Experiment

### a) Vacuum-swing adsorption system:

The schematic of the vacuum-swing adsorption apparatus is shown in figure 1. VSA process consists of four stages: pressurize, adsorption, depressurize, and vacuum operation. Adsorption system designed as bench scale includes an adsorption column with pipes, valves, rotameter, oxygen sensor (SENKO-SS1118), vacuum pump (VALUE- VE115), hardware for Data acquisition and software package of SENKO Co. to convert and monitor results. The length and inner diameter of adsorption column made of stainless steel pipe are 0.2 and 0.1 m, respectively. Two perforated plates with mesh consisting of thin hole are installed on both ends of column to fix adsorbent. Oxygen sensor and its transmitter which are located in a handmade sheath can measure the concentration with response time of 15 to 20 seconds. Oxygen concentration versus sampling time is stored as HEX format, and it is converted to decimal numbers by coding in MATLAB. Sampling time is adjustable, and it is considered 1 second which is appropriate regarding to adsorption rate. Moreover, since oxygen sensor should not be under gas pressure, a needle valve is utilized after the adsorbent column and in front of the sensor.



**Figure 1.** Schematic of VSA apparatus, PR = pressure regulator, PT = pressure transducer, RM=Rotameter, OT= oxygen transducer, V=valve, SV=switch valve

**Table 1.** Properties of the adsorbent

Adsorbent	Size range (mm)	Tapped bulk density(g/l)	N <sub>2</sub> adsorption (ml/g) -1013 mbar, 25°C	porosity	Pore diameter (nm)
Zeolite 13X (Zeochem Co.)	0.6	580-680	>21.5	0.4	0.8

The air mixture which contains N<sub>2</sub> and O<sub>2</sub> (78/21 Vol.%) enters the fixed bed as feed gas. 532 g of zeolite 13X, which does not absorb oxygen and contains spherical particles, is located as an adsorbent in the column. The characteristics of the adsorbent are listed in Table 1. The process is operated with two consecutive cycles by pressurizing of 2 bar, adsorbing with flow rate of 1.5 lit/min, depressurizing to 0 bar and evacuating the bed. By using pressure in the adsorption step, nitrogen molecules pierce in particles pores, so the bed should be regenerated. By reducing pressure, the fixed bed cannot adsorb N<sub>2</sub> in the second cycle, which states that the bed cannot be revived. Hence, in order to regenerate the adsorbent, the vacuum operation is applied. The vacuum pump is utilized in the range of 570 mmHg (25% of absolute atmosphere pressure) for 15 minutes. In this stage, input and output (I/O) routes are closed, and nitrogen adsorbed by bed is evacuated from bypass path. In order to assure of completely removing of nitrogen molecules, the outlet pipe of vacuum pump is put in a vessel of water. Therefore, whenever bubbles do not exit, it means that no gas exists in the bed. Moreover, by this way, air cannot enter to sensor chamber through outlet route in low flow rates. The VSA experiments will continue until outer concentration of O<sub>2</sub> becomes near to inner concentration, which states that the adsorbent is saturated.

#### b) Determining the bed porosity:

A graduated cylinder whose volume is  $V_b$  is filled with the adsorbent. By weighing this cylinder containing adsorbent ( $m_s$ ) and one particle of zeolite, the number of particles is determined in this volume, so the volume of all particles is calculated. By subtracting of this volume from  $V_b$ , the volume of empty space is calculated as follow.

$$\varepsilon_b = \left( \frac{6V_b m_p - \pi d_p^3 m_s}{6m_p V_b} \right) \quad (1)$$

#### c) Calculating the amount of adsorption:

The amount of adsorbed nitrogen per mass of adsorbent is obtained by equation (2).

$$m = \frac{Q_p}{m_s} \int_0^t \left( 1 - \frac{c}{c_0} \right) dt \quad (2)$$

The mole fraction and density of adsorbed nitrogen considered as ideal gas law is calculated as follow:

$$y_{N_2} = 1 - y_{O_2} \quad (3)$$

$$\rho = \frac{PM}{RT} \quad (4)$$

### 3. Mathematical Modeling

Mathematical model is required to perceive the behavior

of the adsorption bed during the VSA process. This model is based on the following assumptions:

- (1) The process is isothermal.
- (2) The particles are spherical and they are packed uniformly into the fixed bed with homogeneous porosity.
- (3) Equilibrium equations of nitrogen and oxygen components are represented by binary Henry isotherm.
- (4) The gas phase behaves as an ideal gas.
- (5) The flow pattern is described by the axially dispersion flow.
- (6) Radial concentration gradient is negligible.

The component mass balance for the gas phase in the adsorption bed is derived by the following equation [8]:

$$-D_z \frac{\partial^2 C_{(i)}}{\partial z^2} + \frac{\partial(uC_{(i)})}{\partial z} + \frac{\partial C_{(i)}}{\partial t} + \frac{1-\varepsilon_b}{\varepsilon_b} [\rho_p \frac{\partial \bar{q}_{(i)}}{\partial t}] = 0 \quad (5)$$

The axial dispersion coefficient,  $D_z$ , is obtained as follow [23]:

$$D_z = \frac{20D_m}{\varepsilon_b} + \frac{0.5ReScD_m}{\varepsilon_b} \quad (6)$$

$D_m$  is calculated by following equations (7-9) [10]:

$$D_{i,j} = \frac{T^{15}}{p\sigma_{i,j}^2\Omega} \left[ \frac{1}{M_i} + \frac{1}{M_j} \right]^{0.5} \left[ 0.0027 - 0.0005 \left[ \frac{1}{M_i} + \frac{1}{M_j} \right]^{0.5} \right] \quad (7)$$

$$\sigma_{i,j} = \frac{\sigma_i + \sigma_j}{2}, \quad \varepsilon_{i,j} = (\varepsilon_i \varepsilon_j)^{0.5} \quad (8)$$

$$\Omega = [44.54(K_B T / \varepsilon_{i,j})^{-4.909} + 1.911(K_B T / \varepsilon_{i,j})^{-1.575}]^{0.1} \quad (9)$$

$$\frac{\varepsilon}{K} = 0.75T_c$$

$$\sigma = (2.3551 - 0.087w) \left( \frac{T_c}{P_c} \right)^{\frac{1}{3}} \quad (10)$$

Therefore, Diffusion coefficient in the mixture is calculated as follow [10].

$$D_{m,i} = \frac{1-y_i}{\sum_{x=j}^n \frac{y_j}{D_{i,x}}} \quad (11)$$

The boundary and initial conditions are presented below.

$$C_{(i)}(z, t) = C_{(i)}(0, t) + \frac{D_z}{u} \frac{\partial C_{(i)}(0, t)}{\partial z}; z=0 \quad (12)$$

$$\frac{\partial C_{(i)}(z, t)}{\partial z} = 0; z=L \quad (13)$$

$$C_i(z, 0) = 0 \quad (14)$$

The binary adsorption equilibrium can be estimated by Henry isotherm as follow [10, 20]:

$$q_i = k_h RT c_i \quad (15)$$

In order to calculate the pressure drop, the Ergun equation is utilized [21].

$$\Delta p = \frac{150 \mu L (1-\varepsilon_b)^2}{d_p^2 \varepsilon_b^3} v_s + \frac{1.75 L \rho (1-\varepsilon_b)}{d_p \varepsilon_b^3} v_s^2 \quad (16)$$

For the each following cases, these equations are resolved numerically by Runge-Kutta method, coded by MATLAB. In order to confirm the model accuracy, the error between the experimental data and model result is obtained as follow:

$$\% \text{ error} = \frac{1}{N} \sum_{i=1}^N \text{abs} \left( \frac{y_{\text{model}} - y_{\text{exp}}}{y_{\text{model}}} \right) \times 100 \quad (17)$$

#### a) Local equilibrium theory

In this theory, the rate of mass transfer is assumed rapid and immediate. Therefore, the mass transfer resistance can be neglected, and the equilibrium between fluid and adsorbed phase is established rapidly in each part of the adsorbent bed. Hence,  $\frac{\partial \bar{q}}{\partial t}$  in equation (5) is replaced by  $\frac{\partial q^*}{\partial t}$ .  $q^*$  is the adsorbed equilibrium concentration in solid phase, which is determined by Henry isotherm as follow [10, 11]:

$$-D_{z(i)} \frac{\partial^2 C}{\partial z^2} + \frac{\partial(uC)}{\partial z} + \frac{\partial C}{\partial t} + \frac{1-\varepsilon_b}{\varepsilon_b} [\rho_p \frac{\partial q^*}{\partial C} \frac{\partial C}{\partial t}] = 0 \quad (18)$$

$$\frac{\partial q^*}{\partial C} = k_h RT \quad (19)$$

#### b) Solid diffusion theory:

The zeolite crystal contains micropore states in which diffusion of adsorbate molecules happen within surface wall of the pore. In this case, the diffusion rate can be written in terms of the solute concentration. Hence, rate of mass transfer in adsorbent particles can be obtained as follow [10, 11].

$$\frac{\partial q_i}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left( D_{si} r^2 \frac{\partial q_i}{\partial r} \right) \quad (20)$$

Boundary and initial conditions without external resistance are:

$$\left( \frac{\partial q_i}{\partial r} \right)_{r_p=0} = 0 \quad (21)$$

$$(C_{pi})_{r=r_p} = C_i \quad (22)$$

Initial condition is as follow [10].

$$(C_{pi})_r = 0 \quad (23)$$

With considering LDF approximation, the last term in the equation (5) is defined as:

$$\frac{\partial q_i}{\partial t} = k_n (q_i^* - \bar{q}_i) \quad (24)$$

For solid diffusion,  $k_n$  is calculated by equations (23-24) considering  $R=1$  for linear isotherm [8].

$$k_n = \frac{15 \psi_s D_{si}}{r_p^2} \quad (25)$$

$$\psi = \frac{0.894}{1-0.106sf^{0.5}} \quad (26)$$

$q_i^*$  is estimated by Henry isotherm, and the average amount of adsorption ( $\bar{q}_i$ ) is calculated by solving the set of differential mass balance equations of particle. Finally the equation of gas phase in the bed (Eq. 5) and particle phase (Eq. 20) are solved simultaneously, according to the boundary conditions of the particle.

## 4. Result and Discussion

Figure 2 represents experimental oxygen composition data versus time. The characteristics of air as feed is stated in table 2. According to figure 2, the maximum concentration of oxygen is produced during 0 to 100 seconds because the sites of adsorbent contain no adsorbate at the beginning of the process, so it has the comprehensive potential to adsorb nitrogen. By passing air, the adsorbent becomes saturated, and the adsorption rate decreases between 100 to 280 seconds.

**Table 2.** Specification of inlet air as feed

Parameter	Unit	Value
Density	kg/m <sup>3</sup>	2.4*10 <sup>-3</sup>
viscosity	kg/m.s	1.846*10 <sup>-5</sup>
Molecular weight	g/mole	28.8
flow rate	lit/min	1.5
inner pressure	Bar	2
outer pressure	Bar	1.6

To calculate the theoretical pressure drop, velocity is calculated by volumetric flow rate and cross section of bed, and bed porosity is determined by equation 1. The experimental and theoretical pressure drop (eq. 16) is 0.4 and 0.3558, respectively.

#### a) Evaluating the time of vacuum stage:

In order to estimate the appropriate duration of vacuum stage, temporal oxygen compositions are studied in different time periods of applied vacuum (560 torr). Therefore, the effect of vacuum condition is determined by calculating the amount of adsorbed nitrogen (Eq. 2) in various time of applied vacuum pressure, which is shown in Table 3. which states that the time of 3 minutes is appropriate for the vacuum stage.

**Table 3.** The amount of adsorbed nitrogen in different time of vacuum pressure (560 torr)

Time of vacuum stage (min)	Amount of adsorbed nitrogen (g/g adsorbent)	%change $\left( \frac{m_2 - m_1}{m_2} \times 100 \right)$
1	0.0143	—
2	0.0144	0.7
3	0.016	11.11
5	0.0174	8.75

#### b) Coating the adsorbent by NMP solvent:

In order to coat the pores of the adsorbent by NMP(N-Methyl-2-pyrrolidone), liquid acetone is used as a carrier. The mixture of NMP and acetone blended well is added to the adsorbent and put in the bath at 60°C. Since acetone and NMP boil at 60°C and 202°C, respectively, acetone evaporates in the bath. Hence, NMP can be entered to inner pores of adsorbent by acetone gas. The outer tube of column is placed in a glass of water. Until acetone exists in the column, outer bubbles can be seen in the water.

The breakthrough curves of zeolite 13X which is covered by 5% and 10% of NMP are shown in figure 4. By much more amount of NMP, the adsorption of nitrogen decreases in the bed. Also, considering to the experiment, coating zeolite by NMP is an exothermic reaction.

### c) Comparing the results of experiment and model:

In the mathematical model, Henry constant is put  $1.1839 \times 10^{-5}$  in the Henry isotherm [20]. In order to compare the results of model with experiments, the dimensionless of

nitrogen mole fraction versus time is plotted as the breakthrough curve.

### Local equilibrium theory:

Figure 5 shows the effluent nitrogen concentration which is modeled by local equilibrium theory with error of 11.7%. Therefore, this model cannot predict the experimental data appropriately. As a result, non-equilibrium model based on mass transfer rate is applied in the following section.

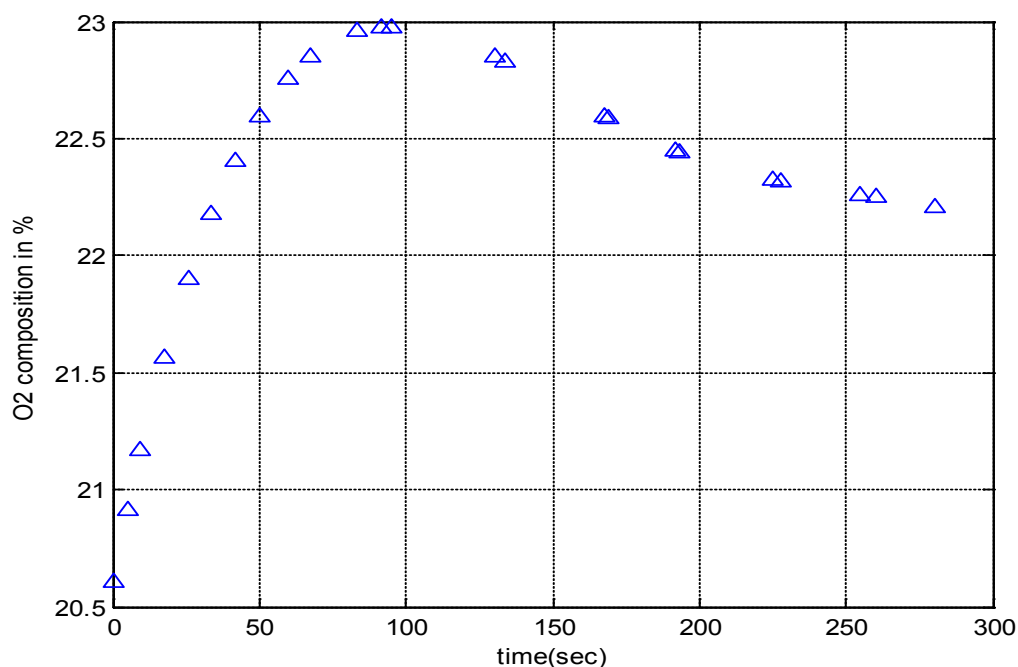


Figure 2. Temporal profiles of effluent oxygen composition

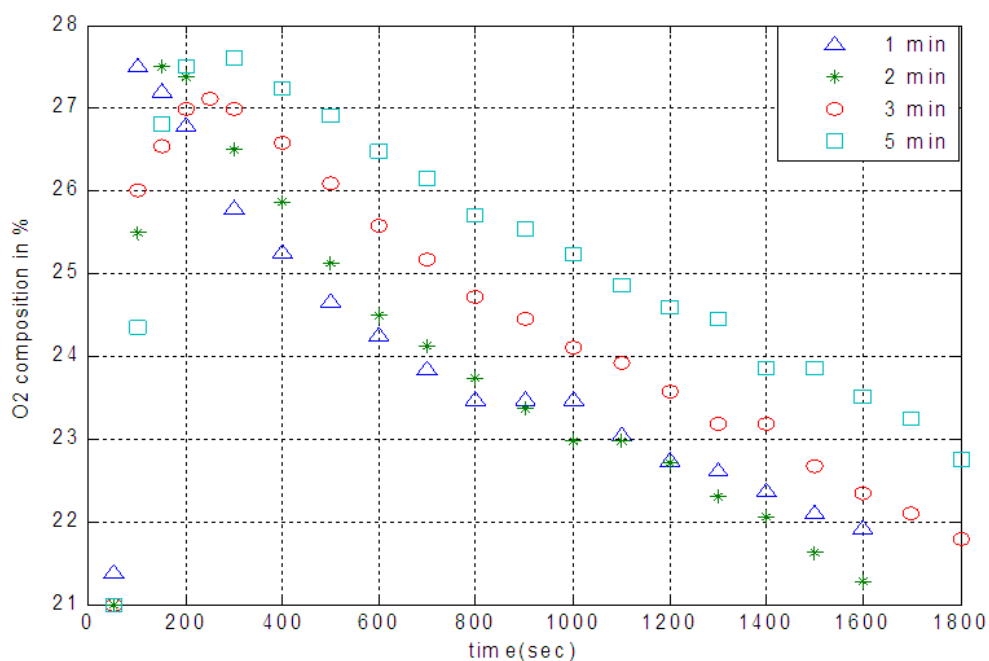


Figure 3. Temporal profiles of effluent oxygen composition in different time of vacuum pressure (560 torr)

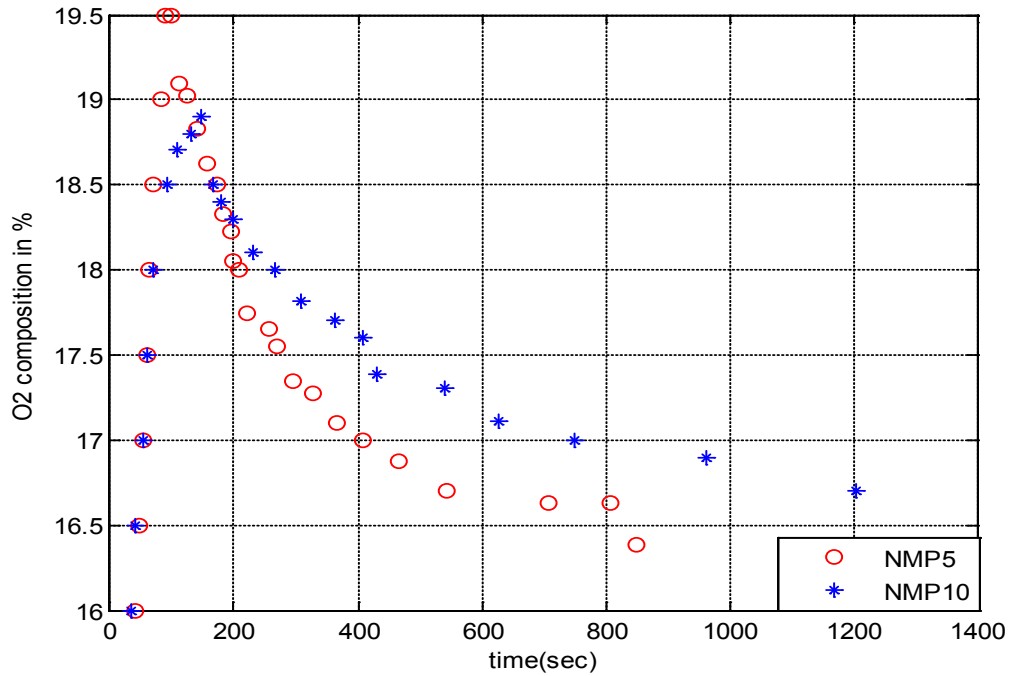


Figure 4. Temporal profiles of effluent oxygen composition of zeolite 13X covered by the various amount of NMP

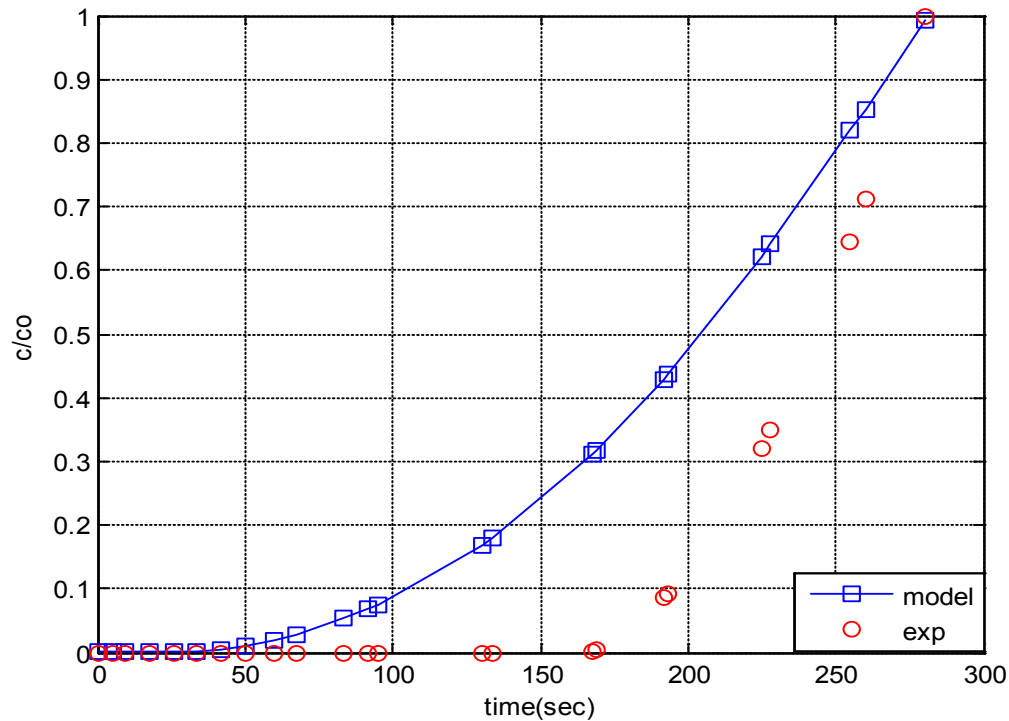


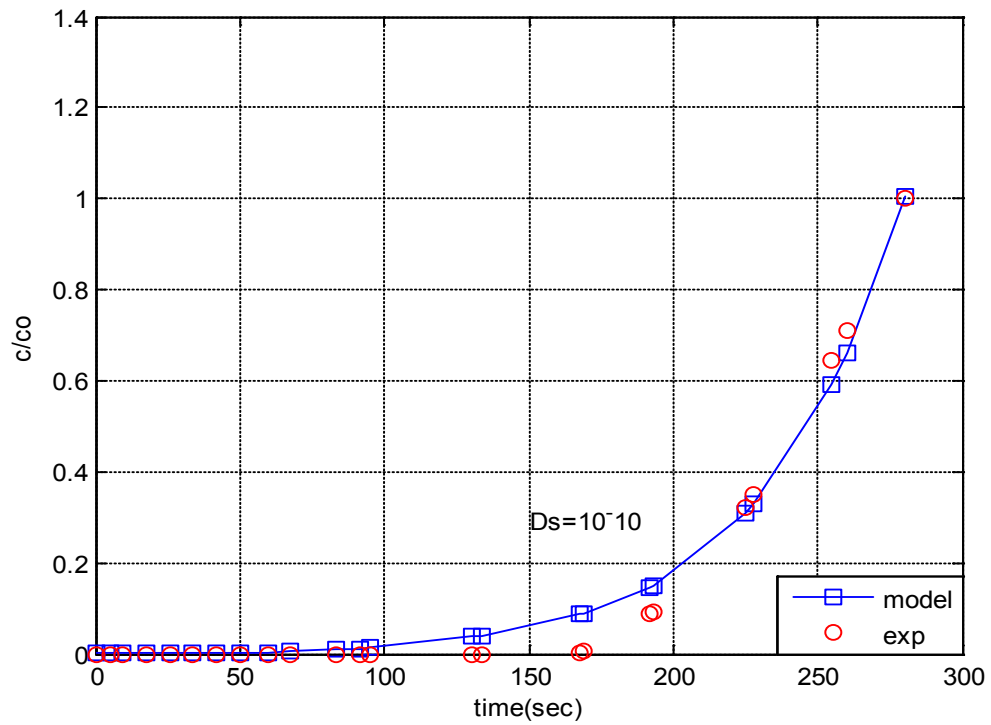
Figure 5. The experimental breakthrough curve of nitrogen modeled by equilibrium theory

### Solid diffusion model

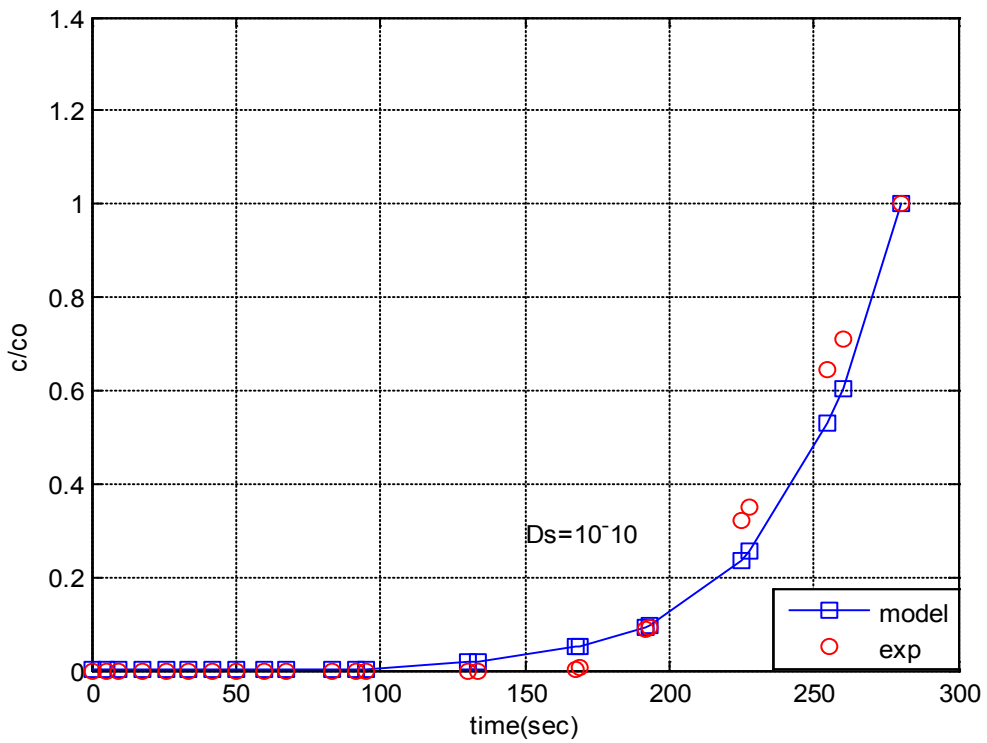
Modeling of adsorbed nitrogen by solid diffusion theory is displayed by figure 6. In this model, zeolite crystallite diffusivity ( $D_s$ ), which is in the range of  $10^{-7}$  to  $10^{-14}$  [8], is used as the fitting parameter. According to this figure, the single fitting parameter model results in more accurate correlation. Moreover, solid diffusion model is capable to predict the breakthrough curve better than the equilibrium model, with error of 1.17%. Since the pore diameter of

zeolite 13X is less than 1 nm, it is considered as micropore. Hence, its dominant diffusion mechanism occurs on the surface of micropore. Also, the effect of zeolite diffusivity as the fitting parameter is shown by figure 8. Considering the %error values, this parameter affects the model and the amount of  $10^{-10}$  is suitable to predict the data.

If instate of axil diffusion ( $3.45 \times 10^{-4}$ ) used molecular diffusion coefficient ( $1.38 \times 10^{-5}$ ) reduced error percent to 1% that shown in figure 7.



**Figure 6.** The experimental breakthrough curve of nitrogen modeled by solid diffusion theory



**Figure 7.** The experimental breakthrough curve of nitrogen modeled by solid diffusion theory without dispersion

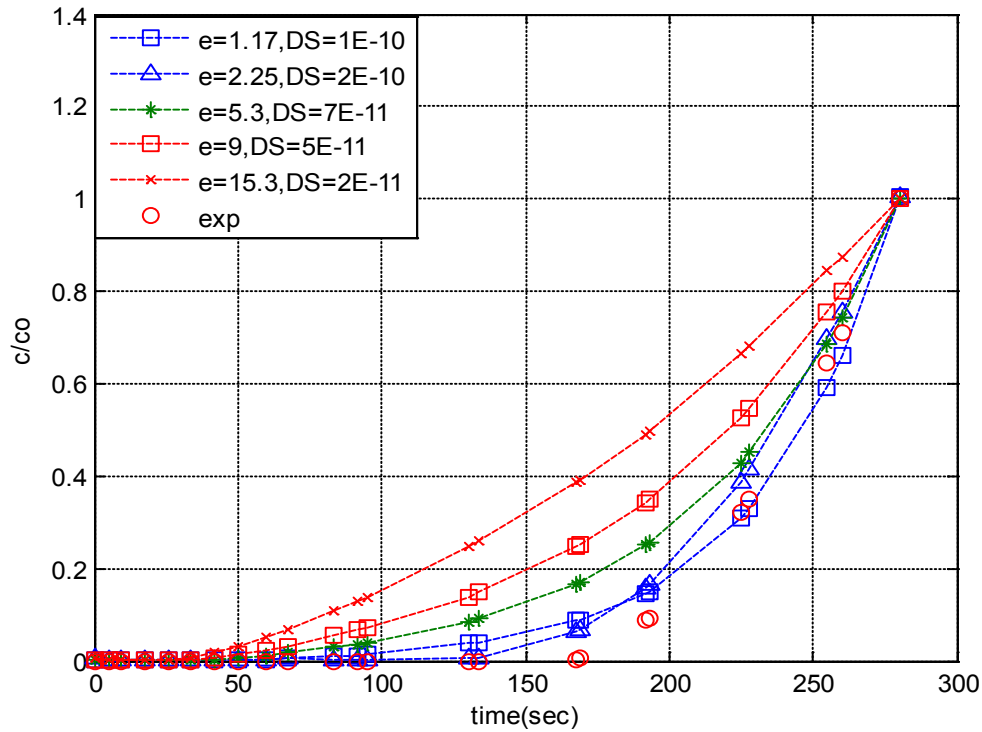


Figure 8. The effect of zeolite diffusivity on solid diffusion model

## 5. Conclusions

Vacuum swing adsorption using zeolite 13X is carried out in order to produce oxygen by separating air. It is obtained that VSA process is much more appropriate and applicable than PSA. According to study the effect of vacuum stage on adsorbing nitrogen, the time of 3 minutes is suitable to regenerate the adsorbent. Moreover, it is observed that coating the zeolite by NMP solvent has the detrimental effects on the structure of the adsorbent. Hence, the low amount of NMP decreases the adsorption, and its high amount deactivates the zeolite. Also, the pressure drop during the experiments is compared with Ergun equation, which has 11% error. The mass balance of the adsorption bed with isothermal process, axially dispersion flow and Henry adsorption isotherm is applied to study the adsorption of nitrogen. This model is investigated for two cases containing local equilibrium, and solid diffusion model. Solid diffusion model with LDF approximation is used successfully to correlate the adsorbed nitrogen versus time. The modeling result is improved by using adsorbent diffusion coefficient as a fitting parameter. % error values are 11.7% for equilibrium model and 1.17% for solid diffusion model with one fitting parameter.

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## Abbreviation

$C$	fluid-phase concentration, $\text{mol}/\text{m}^3$
$C_p$	pore fluid-phase concentration, $\text{mol}/\text{m}^3$
$C_0$	initial fluid-phase concentration, $\text{m}^2/\text{s}$
$d_p$	particle diameter, m
$D_z$	axial dispersion coefficient, $\text{m}^2/\text{s}$
$D_s$	adsorbed-phase (solid) diffusion coefficient, $\text{m}^2/\text{s}$
$D_m$	axial diffusion coefficient, $\text{m}^2/\text{s}$
$k_n$	rate coefficient based on adsorbed-phase concentration driving force, $\text{s}^{-1}$
$k_h$	Henry constant
$k_B$	Boltzmann constant, J/K
$L$	bed length, m
$m_p$	mass of the adsorbent particle, kg
$m_s$	mass of adsorbent
$m$	adsorbed nitrogen, kg/kg adsorbent
$M_i$	molecular weight i, kg/kmol
$P$	pressure, bar
$p_c$	critical pressure, bar
$Q$	flow rate, $\text{m}^3/\text{s}$
$\bar{q}$	average amount of adsorption, $\text{mol}/\text{kg}$ adsorbent
$q$	amount of adsorption, $\text{mol}/\text{kg}$ adsorbent
$q^*$	adsorbed equilibrium concentration, $\text{mol}/\text{m}^3$
$R$	gas constant, $\text{Pa m}^3/(\text{mol K})$
$sf$	separation factor
$Re$	Reynolds number
$r$	particle radial coordinate, m
$r_p$	particle radius, m



$Sc$	Schmidt number
$T$	time, s
$T$	Temperature, K
$T_c$	critical Temperature, K
$u$	velocity, m/s
$V_b$	volume of graduated cylinder, $m^3$
$V_c$	critical volume, $m^3$
$v_s$	superficial velocity, m/s
$w$	acentric factor
$y$	fluid-phase mole fraction
$Z$	bed axial coordinate, m
$\varepsilon_p$	particle porosity
$\varepsilon_b$	bed porosity
$\varepsilon$	void fraction of packing (extraparticle)
$\Lambda$	partition ratio
$\mu$	fluid viscosity, kg/(m s)
$\Omega$	diffusion collision
$\psi$	LDF correction factor
$\rho$	fluid density, kg/ $m^3$
$\rho_p$	particle density, kg/ $m^3$
$\sigma$	collision diameter, ( $^{\circ}A$ )
$\tau$	adsorption time
$i, j$	component index

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