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Isotherms and Kinetics Study for Adsorption of Nitrogen from Air using Zeolite Li-LSX to Produce Medical Oxygen

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Abstract

This research investigates the adsorption isotherm and adsorption kinetics of nitrogen from air using packed bed of Li-LSX zeolite to get medical oxygen. Experiments were carried out to estimate the produced oxygen purity under different operating conditions: input pressure of 0.5 – 2.5 bar, feed flow rate of air of 2 – 10 L.min^{-1} and packing height of 9-16 cm. The adsorption isotherm was studied at the best conditions of input pressure of 2.5 bar, the height of packing 16 cm, and flow rate 6 Lmin^{-1} at ambient temperature, at these conditions the highest purity of oxygen by this system 73.15 vol % of outlet gas was produced. Langmuir isotherm was the best models representing the experimental data., and the model parameters were the maximum monolayer coverage (qm) 200 mg. g^{-1} and Kl 0.00234 L.mg $^{-1}$. Also, from the Freundlich isotherm model, the sorption intensity (n) indicated favorable sorption of 1.435. The average free energy estimated from the DRK isotherm model was 0.02 KJ.mol^{-1}, which proved the adsorption process to follow physical nature. The results got from experiments showed a coincidence to the pseudo-first-order kinetic model.

Keywords: Zeolite Li-LSX, medical oxygen, adsorption isotherm, adsorption kinetic, nitrogen adsorption.

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1- Introduction

1.1. Pressure – Vacuum Swing Adsorption Process

COVID-19 outbreak stimulates researchers to investigate another source of medical oxygen as an alternative to the conventional method of the cryogenic process, which consumes a large amount of energy. That is because of the huge increase in the need for portable medical oxygen concentrators (MOCs) because of pulmonary failure caused by COVID-19 as well as chronic bronchitis, pneumonia and chronic obstructive pulmonary disease (COPD) to avoid problems caused by hypoxemia [1-3]. Adsorption, membranes, and cryogenic separation are the three main ways to separate the different parts of air [1]. Adsorption is the most promising method for its simplicity, appropriateness in terms of moderate conditions, and low energy consumption, moreover, the separation method by adsorption is used to make very pure oxygen [1]. A lot of equipment that produce medical oxygen are made to get pure oxygen [2]. Different adsorption methods were experimented with to adsorb nitrogen gas selectively from air to produce pure oxygen gas. Pressure swing as well as vacuum swing, or pressure/vacuum swing adsorption processes, which is symbolized by PVSA, were tried using N_2-selective adsorbents [3]. Also, Temperature Swing Adsorption (TSA) process was experienced [6].

It was demonstrated that PSA was more feasible than TSA process [2]. That is because PSA process cycle has a time of between one to several seconds, unlike the TSA process cycle which has a time extends to hours [6].

PVSA process is the common way to separate gases since the adsorption step is carried out at above atmospheric pressure and the step of regeneration is conducted under a vacuum pressure [7]. There are three points that make PVSA excel than PSA. The first is that the O_2/N_2 recovery rate and purity of the PVSA process is better than those of the PSA process [3]. The gas exits from heavy-reflux and light-reflux streams is used and that is why PVSA yield is more than PSA. This leads to an increase in both PVSA process productivity and capacity of adsorbents relative to those of PSA. The second point is in terms of total energy use. PSA process uses more energy than PVSA process. This is because PVSA includes vacuum pump which uses less energy in comparison with a conventional pump. The third point is that the mass transfer zones for the light and heavy components often get in a common way in a formal dual-reflux PSA process with has an intermediate feed. This problem can be solved with these newly integrated PVSA processes. Zeolites are likely to be used to separate air into its components because N_2 gas molecules' dipole and quadrupole moments interact with extra cations of the zeolites' frame. Lithium (Li) forms an attractive cation for adsorbing N_2 molecule better than O_2 molecule on LSX

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zeolite when it is mixed with air [8]. Talking about adsorbents' capacity and productivity leads to study the adsorption isotherms of the system used in this research since it gives information about the full benefit of the adsorbent. Also, the study of adsorption kinetics makes a clear image of the rate of adsorption and the mechanism it followed.

1.2. Adsorption Isotherm Models

Adsorption isotherms can be defined as suitable mathematical models to describe the distribution of the adsorbed ions between the adsorbent material and the solution at equilibrium. Langmuir, Freundlich, Dubinin–Radushkevich, and others [8, 9] came up with models of adsorption isotherms. Langmuir model, which was made to represent gas/solid phase interaction, was used in comparing, and measuring the ability of different adsorbents to take up molecules [8]. Langmuir's isotherm takes surface coverage into account by balancing the relative rates of adsorption and desorption (dynamic equilibrium). While adsorption depends on how much of the adsorbent surface is open, desorption depends on how much of the surface is covered [6]. One of several forms of the Langmuir equation is Eq. 1 [6]:

$$\frac{1}{\mathrm{qe}} = \left[\frac{1}{\mathrm{q_m.Kl}}\right]\frac{1}{\mathrm{Ce}} + \frac{1}{\mathrm{q_m}} \qquad (1)$$

Where: qe = the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg. g^{-1}), qm = maximum monolayer coverage capacity (mg. g^{-1}), Kl = Langmuir isotherm constant (L.mg^{-1}), C_e = the equilibrium concentration of adsorbate (mg. L^{-1}).

Freundlich model assumed non-ideal adsorption; the adsorbent has a heterogeneous surface and the adsorption is not restricted to the formation of a monolayer. The model can be linearized as Eq. 2 [9, 10]:

$$\ln qe = \ln Kf + (1/n) \ln Ce \qquad (2)$$

Where: qe = the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg. g^{-1}), K_f = Freundlich isotherm constant (mg. g^{-1}), n = adsorption intensity, C_e = the equilibrium concentration of adsorbate (mg. L^{-1}).

Generally, the Dubinin–Radushkevich isotherm is used to describe how a Gaussian energy distribution on a heterogeneous surface lead to adsorption [11]. The model has often done a good job of fitting data for high solute activities and the middle range of concentrations. The equation describes this model is Eq. 3 [11]:

$$\ln q_e = \ln q_m - K_{DR}\, \varepsilon^2 \qquad (3)$$

Where: qe = the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg. g^{-1}), q_m = theoretical isotherm saturation capacity (mg. g^{-1}), K_{DR} = Dubinin–Radushkevich isotherm constant (mol^2. KJ ^{2}), ε = is the polyanion potential (KJ. mol^{-1}).

ε can be estimated by the following Eq. 4 [11]:

$$\varepsilon = RT \ln(1 + 1/Ce) \qquad (4)$$

Where: R is the gas constant (8.31 J mol^{-1} K^{-1}), T is the absolute temperature and Ce is the equilibrium concentration of adsorbate (mg. L^{-1}).

The model is applied to indicate physical and chemical adsorption of metal ions as well as the free energy, E, which is defined per molecule of adsorbate (that is removing a molecule from its position in the sorption layer to the infinity) may be calculated by Eq. 5 [11]:

$$E = (1/\sqrt{2K}) \qquad (5)$$

E is the mean adsorption energy less than 8 KJ.mol^{-1} in physical adsorption but for the chemical adsorption the energy is between 8 to 16 KJ.mol^{-1} [12].

This study focused on the adsorption isotherm models of N_2 gas from ambient air on Li-LSX zeolite to get medical oxygen as a necessary step in scaling – up the system.

1.3. Adsorption kinetics

Kinetics studies the adsorption rates to explain the mechanism that dominates in a certain system. Studying adsorption kinetics means investigating the experimental conditions that affect the rate of adsorption and, in turn, finding the factors that affect reaching equilibrium. These kinds of studies tell us about the possible way adsorption works and the different steps that lead to the final adsorbate–adsorbent complex. They also help come up with the right mathematical models to describe how things work together. Once the rates and factors that affect them are clear, they can be used to make adsorbent materials for use in industry and to figure out how the dynamics of the adsorption process is complex [7]. Adsorption kinetics are very important for figuring out the equilibrium adsorption capacity and the rate constants. The most common types of kinetic models are pseudo-first order and pseudo-second order. [13]. In pseudo-first order model, the adsorption capacity is related to the rate of adsorption as follows in Eq. 8 [7]:

$$\ln(qe - qt) = \ln qe - k1\, t \qquad (6)$$

Where: qe= adsorption capacity at equilibrium, mg. g^{-1}, qt= adsorption capacity at any time, mg. g^{-1}, k_1 = rate constant for pseudo 1st order adsorption process, min^{-1}.

This model is found to be fit for the initial 20 to 30 min of interaction between the adsorbate and the adsorbent and not fit the overall extent of contacting [14].

While pseudo-second order, which Ho and McKay established within 1998 [15] can be represented in the following Eq. 7:

$$(dq/dt) = k_2\, (q_e - q_t)^2 \qquad (7)$$

Where: k_2 = the Ho-McKay equation's rate constant, g min.mg^{-1}.

The pseudo-second-order kinetic model can be solved to take the linearized form as in Eq. 8 [8]:

$$\frac{t}{qt} = \frac{1}{k_2\, qe2} + \frac{t}{qe} \tag{8}$$

The initial phases of the adsorption process are assumed to be described by the pseudo-first-order model but for the whole range of adsorption it is likely that the adsorption process follows a non-linear model that represents the complex mechanism of interaction between the absorbate and the adsorbent.

For porous adsorbents, the diffusion of the adsorbate molecules into the pores needs to be considered when looking for a good kinetic model for the process. In many cases, the rate at which an adsorbate is taken in may be controlled by intra-particle diffusion. This is presented by the following well-known expression [16].

$$q_t = k_i \cdot t^{0.5} \tag{9}$$

The crucial aspect of this formula is that the linear plot of q t vs $t^{0.5}$ must pass through the origin (zero intercepts). Consequently, the intra-particle diffusion model is easily testable, demonstrating that the diffusion process dominates the kinetics. The slope of the graph can be utilized to calculate the rate coefficient k_i (mg/ (g. $min^{0.5}$) [17].

2- Experimental Work

2.1. Materials

Zeolite Li-LSX was used in the experiments. The technical specification of it is listed in Table 1.

Table 1. The Technical Specification of Li-LSX Zeolite (Commercial Name JLOX-101)

Property	Unit	JLOX-101	Country
Diameter	mm	0.4 – 0.8	China
N_2 adsorption capacity	$ml.g^{-1}$	≥ 22	
N_2/O_2 Selectivity	~	≥ 6.20	
Crush strength	N	~	
Bulk density	$g.ml^{-1}$	0.63±0.03	
Moisture content	wt.%	≤ 0.5	
Particle ratio	%	≥ 95	

2.2. Equipment

The experimental setup is shown in Fig. 1. Fig. 2 shows photos of the experimental setup.

All the equipment used is listed in Table 2.

Table 2. Equipment used in the Research

#	Device	Specification	Range	Country
1	Air compressor	ingco industrial ,220-240V, ~50Hz, AC25508	0-8 bar	China
2	Pressure gauge with filter unit	Filter unit D =2.5 cm, L = 5 cm	Pressure gauge 0-10 bar	China
3	Inlet flow meter	PMB CV.P. A10.LM. G2	1-10 $L.min^{-1}$	China
4	Adsorption column	glass type QVF, L= 17 cm, id= 4cm		
5	Pressure gauge		0-3 bar	China
6	O_2 gas sensor	GDX-O_2, L = 15.5 cm, D = 2.8 cm	0-100% O_2	U. S.
7	Purge flow meter	Matheson U310	0.5 - 6 L min^{-1}	China
8	Valves			China
9	Drum	L= 60 cm, D= 37 cm	V=64480 cm^3	

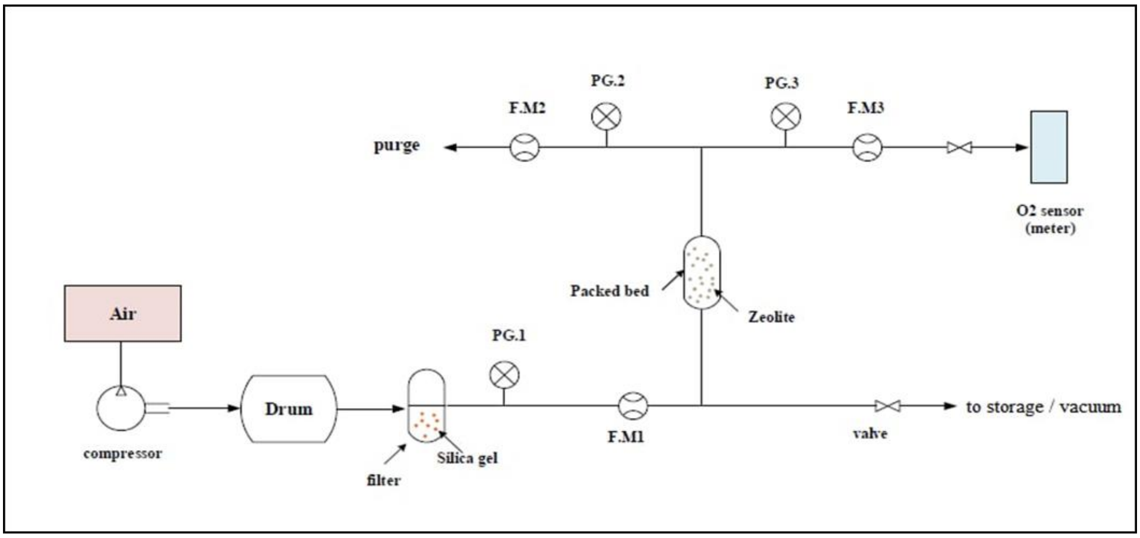

Fig. 1. Schematic Diagram of Experimental Setup

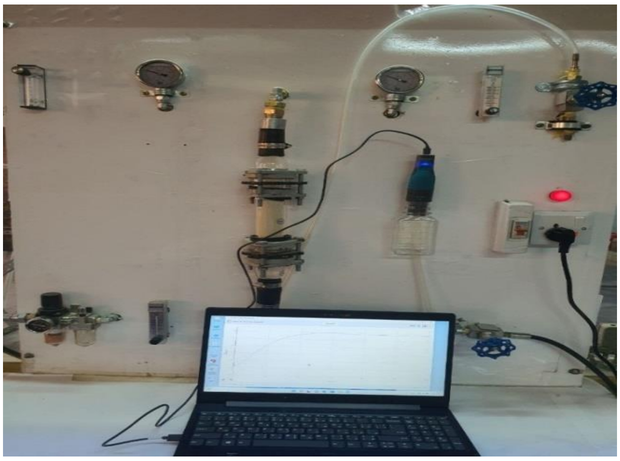

Fig. 2. View of the Experimental Setup

2.3. Procedure

At the beginning, the adsorbent (zeolite Li-LSX) was heated for 45 minutes in an oven at 110 ℃ to eliminate moisture and other impurities. Then zeolite was packed randomly in the column. Helium gas was let to flow passing the packing for refreshing it and prepare it to adsorb N_2. Air was compressed to a specific pressure through the drum to maintain the flow of air to be stable in the column during the experiments. Air was also passed through a filter filled with silica gel to get rid of moisture and impurities. The flow was set at a certain value of flow rate by a flow meter. Nitrogen gas was adsorbed on the zeolite and oxygen-rich gas was produced at the outlet. The oxygen produced was split into two streams: one to the sensor for measuring oxygen purity one to the storage cylinder and the remainder as a volume percentage of the generated gas. Nitrogen volume percent in the inlet and outlet was determined by subtracting the oxygen percent from total 100% complete air components. There was also a purge stream to manage any unusual increase in the outlet pressure. After each adsorption experiment, there was a desorption operation to regenerate the zeolite. The desorption operation was carried out under vacuum pressure of -0.9 bar for 2 minutes. The amount of adsorbed nitrogen was estimated by the difference between the inlet and outlet concentrations after converting the partial pressure to concentration considering nitrogen as an ideal gas at the experimental conditions.

2.4. Response Surface Methodology

The response surface approach was investigated. to investigate the interactions between the variables that affected the purity of O2. Also, it was used for optimizing and scaling up the current laboratory setup. In this respect, experiments were designed using the Box-Behnken Design (BBD). Fifteen experiments were carried out with various combinations of the studied variables which were: inlet pressure, packing height, and flow rate to determine which factors and their interactions had the major influence on the purity of the generated oxygen [18].

3- Results and Discussions

3.1. Isotherm Model

The Langmuir, Dubinin – Radushkevich, and Freundlich isotherm models were used to examine the adsorption data. The adsorption isotherms were applied at the best operating conditions obtained in this study: 2.5 bar pressure, and 16 cm height of packing which gives the highest purity of oxygen (73.15 vol % of outlet gas) which was the basis for optimizing the results to reach the utmost purity required for medical purposes [18]. These models relate the quantity of nitrogen adsorbed on a solid surface Fig. 3 to Fig. 5 below explain the adsorption isotherm model (Freundlich, Dubinin-Radushkevich, and Langmuir) with the results in Table 3.

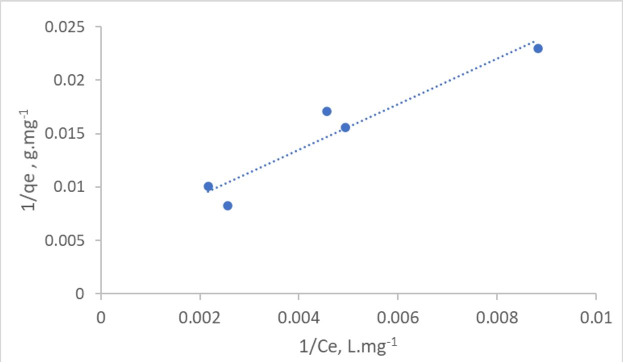

Fig. 3. Langmuir Isotherm Model for Adsorption of Nitrogen at 2.5 bar Input Pressure and 16 cm Packing Height

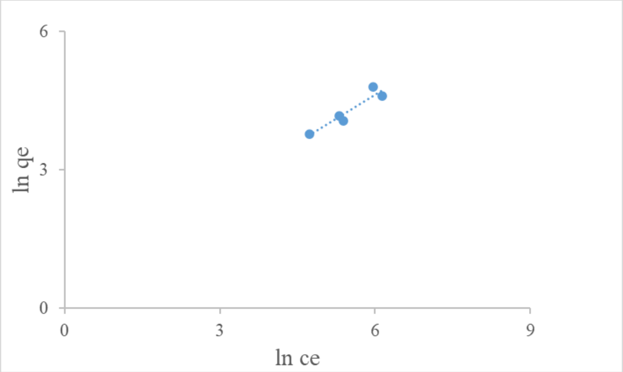

Fig. 4. Freundlich Isotherm Model for Adsorption of Nitrogen at 2.5 bar Input Pressure and 16 cm Packing Height

The results in Table 3 show that all the models fit well the experimental data, but Langmuir model has the maximum value of the correlation coefficient (R^2=0.917). This led to the conclusion that the adsorption process followed Langmuir hypotheses of monolayer of adsorbed molecules and that no forces of interaction existed between them [15]. Langmuir parameters were the maximum capacity of adsorption 200 mg. g^{-1} which indicated a large capacity for adsorption and Kl 0.00234 L.mg^{-1} which indicated a favorable adsorption [16]. Also, the apparent energy (E) was found to be +0.02 KJ.mol^{-1} from DRK isotherm model. The low positive value of energy agrees well with the heat of physical adsorption in the gas phase with n equal to 1.435 from the Freundlich model [10, 12].

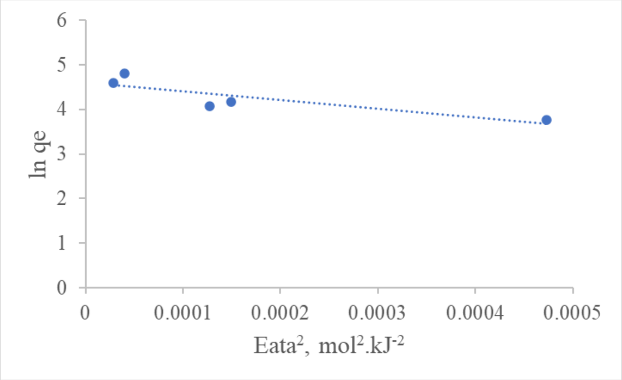

Fig. 5. Dubinin-Radushkevich Isotherm Model for Adsorption of Nitrogen at 2.5 bar Input Pressure and 16 cm Packing Height

Table 3. Langmuir, Freundlich, and Dubinin–Radushkevich Isotherm Constants for the Adsorption of Nitrogen at 2.5 bar Input Pressure and 16 cm Packing Height

Isotherm model	Model parameter	Value	R^2
Langmuir	q_m, mg/g	200	0.917
	Kl, L/mg	0.00234	
Freundlich	K_F, mg/ g	1.552	0.89
	n, -	1.435	
Dubinin	K_D, mol^2/kJ^2	1946.3	0.7219

3.2. Adsorption Kinetics

The kinetic data for the adsorption of nitrogen on zeolite Li-LSX adsorbent was Three fundamental kinetic models were investigated: the pseudo-first-order model, the pseudo-second-order model, and the intra-particle kinetic model. Fig. 6 through Fig. 8 show the three models. Table 4 lists the three kinetic models' correlation coefficients and other characteristics. The three models showed acceptable values of correlation coefficient (R^2) with the pseudo-first order model (0.9834) somewhat higher than the others. The equilibrium capacity of pseudo-first order 63.79 mg /g was close enough to the experimental value 58.5 mg/g to make the decision that the pseudo – first order model represented well the kinetics of this system.

Therefore, the mechanism of which the adsorption followed as well as the speed of adsorption depicted nonlinear interaction between nitrogen gas molecules and Li-LSX zeolite.

Table 4. Kinetics Models Constants for Adsorption of N_2 on Zeolite Li-LSX at 2.5 bar Pressure and 16 cm Packing Height

Kinetics Model	Model constants	Model constant value
Pseudo - first- order	K_1 (L/min)	2.746
	qe (mg/g)	63.79
	R^2	0.9834
Pseudo- second-order	K_2 (g /mg. min)	0.048
	qe (mg/g)	217.391
	R^2	0.9772
Intra particle	K_i (mg/g.min$^{1/2}$)	58
	R^2	0.9193

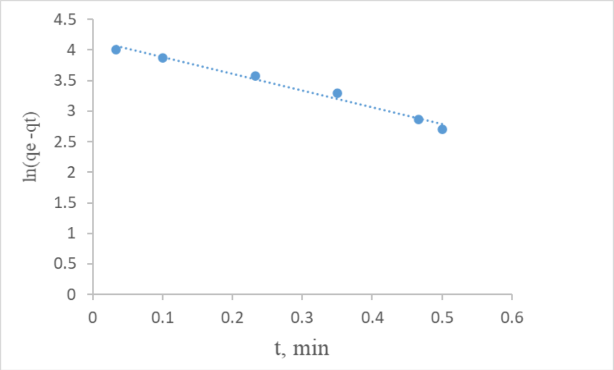

Fig. 6. The Pseudo-First-Order Kinetic Model of N_2 Adsorption on Zeolite Li-LSX at 2.5 bar Pressure and 16 cm Packing Height

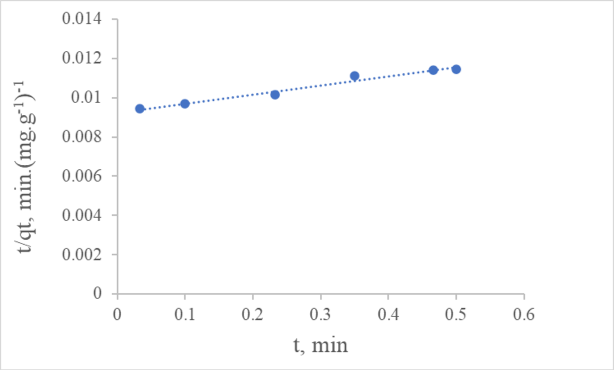

Fig. 7. The Pseudo-Second-Order Kinetic Model of N_2 Adsorption on Zeolite Li-LSX at 2.5 bar Pressure and 16 cm Packing Height

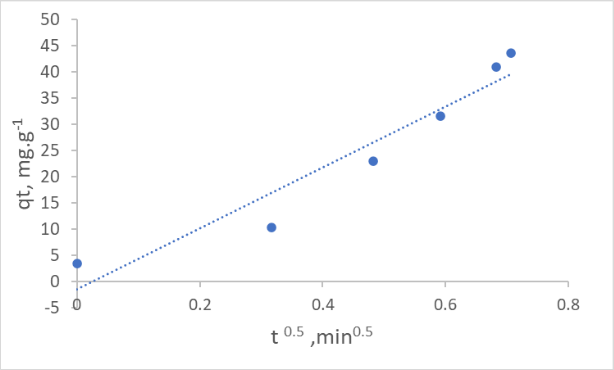

Fig. 8. The Intra-Particle Diffusion Model of N_2 Adsorption on Zeolite Li-LSX at 2.5 bar Pressure and 16 cm Packing Height

4- Conclusions

The equilibrium sorption was investigated in a packed bed of zeolite Li-LSX at pressure 2.5 bar, flow rate of 2-10 L.min^{-1}, and height of packing of 16 cm for the adsorption of N_2 from air to produce medical oxygen. The adsorption isotherm models were studied to get the necessary information of the adsorbent material that it confirmed its feasibility in industrial application. The sorption data were applied into Langmuir, Freundlich, and Dubunin - Radushkevich isotherms. Langmuir adsorption model was the best correlation represented the experimental data. All the models proved that the adsorption was physical in nature and the concept of one layer was applicable. The experimental data correlated well with the pseudo -first-order kinetic model which indicated the nonlinear interaction between nitrogen molecules and Li-LSX zeolite.

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دراسة متساوية الحرارة والحركية لامتزاز النيتروجين من الهواء باستخدام الزيولايت Li-LSXلإنتاج الأكسجين الطبي

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٢ مركز بحوث النفط والبتروكيمياويات، وزارة العلوم والتكنولوجيا، العراق

الخلاصة

يدرس هذا البحث متساوي حرارة الامتزاز وحركية امتزاز النيتروجين من الهواء باستخدام طبقة معبأة من الزيوليت Li–LSX لإنتاج الأوكسجين الطبي. أجريت تجارب لتقدير نقاء الأوكسجين الناتج تحت ظروف تشغيل مختلفة: ضغط الإدخال (٠,٥ – ٢,٥ بار)، معدل تدفق الهواء الداخل (٢–١٠ لتر. دقيقة $^{-١}$) وارتفاع التعبئة (٩–١٦ سم). تمت دراسة متساوي حرارة الامتزاز في أفضل الظروف التي أنتجت أعلى درجة نقاء للأوكسجين بواسطة هذا النظام (٧٣,١٥ % من حجم الغاز الخارج). كانت هذه الظروف هي: ضغط إدخال ٢,٥ بار وارتفاع التعبئة ١٦ سم ومعدل تدفق ١٦لتر. دقيقة $^{-١}$ عند درجة الحرارة المحيطة. ان نماذج متساوي حرارة الامتزاز التي بحثت كانت: متساوي حرارة لانكماير وفريوندليش ودوبينين رادوشكيفيتش (DRK). كانت قيم معامل الارتباط R^2 كالتالي: نموذج متساوي الحرارة لانكماير ٠,٩١٧، فريوندليش ٠,٨٩ و ودوبينين رادوشكيفيتش ٠,٧٢١٩. أظهرت جميع النماذج المدروسة تمثيلاً جيدًا للبيانات التجريبية، لكن متساوي الحرارة لانجموير كان الأفضل، وكانت معلمات النموذج هي أقصى تغطية أحادية الطبقة (q_m) ٢٠٠ مجم.غم $^{-١}$ و Kl ٠,٠٠٢٣٤ لتر.ملي غم $^{-١}$. أيضًا ، من نموذج متساوي الحرارة فريوندليش ، أشارت شدة الامتصاص (n) إلى امتصاص إيجابي قدره ١,٤٣٥. تم تقدير متوسط الطاقة الحرة من نموذج متساوي الحرارة ودوبينين رادوشكيفيتش ليكون ٠,٠٢ كيلوجول . مول $^{-١}$ والذي أثبت بوضوح أن تجربة الامتزاز اتبعت الطبيعة الفيزيائية. أظهرت النتائج التجريبية مطابقة جيدة للنموذج الحركي من الدرجة الأولى الزائف.

الكلمات الدالة: الزيوليت Li–LSX، الأوكسجين الطبي، متساوي الحرارة للامتصاص، حركية الامتزاز، امتزاز النتروجين.