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CHEMICAL ENGINEERING

Breakthrough analysis of continuous fixed-bed dehydration of gas streams using 4A zeolite molecular sieve

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ABSTRACT. The experimentally investigation for dehydration of gas streams in a fixed-bed adsorption column using a 4A molecular sieve was reported for the first time. Different operating conditions of the adsorption system were performed, such as flow rate (2.0, 5.0 and 8.0 L min.$^{-1}$) and bed depths (10, 20 and 30 cm). A significantly decreased ($p \leqslant 0.05$) in exhaustion time of the adsorption column was verified as a result of the increase in the flow rate. On the other hand, a significantly increase ($p \leqslant 0.05$) in the rupture time and the mass transfer zone (MTZ) were observed when the bed depths rise. Furthermore, the experimental data were satisfactory described by the Yoon-Nelson model due to high values of correlation coefficient (R^2). These results suggested the suitability of 4A zeolite molecular sieve for the dehydration of gas streams in continuous operating mode. Moreover, it could be interesting for the scientific communities and industry.

Keywords: adsorption; 4A zeolite molecular sieve; carbon dioxide; dehydration; natural gas; breakthrough curves.

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Introduction

Natural Gas (NG) has gained significant importance in the world energy matrix and has shown great environmental advantages such as the reduction in CO_2 emissions and insignificant emissions of oxides from SO_X and NO_X (Faramawy, Zaki, & Sakr, 2016). Therefore, a strong growth in demand from NG electricity generation is expected to increase almost 2.5 times until 2050. For this purpose, the processing of NG must be significantly increased to fulfill this global demand (Santos, Correia, Medeiros, & Araújo, 2017; Nam, Nam, & Lee, 2021).

During the processing of NG, the removal of water vapor is a fundamental and mandatory step (Kim et al., 2016). Natural gas drying is a very important process to the industry because, water vapor in gas streams can result in line plugging due to hydrate formation, reduction of line capacity due to collection of free water in the line, and a higher risk of damage to the pipeline due to the corrosive effects of water (Gholami, Talaie, & Roodpeyma, 2010; Santiago et al., 2018; Antunes et al., 2018; Rodrigues, Naccache, & Mendes, 2019; Salman, Zhang, & Chen, 2020).

Currently, four methods of for natural gas streams dehydration widely applied in the industry are: membrane separation; absorption by triethylene glycol, condensation and adsorption on solid desiccants (Netusil & Ditl, 2011; Dalane, Dai, Mogseth, Hillestad, & Deng, 2017). Among these methods adsorption processes are considered an economic alternative for the removal of water vapor from gas streams due to the possibility of adsorbent material reuse, simplicity of operation, simultaneous dehydration and removal of water vapor, high product recoveries and low energy costs (Golubovic, Hettiarachchi, & Worek, 2006; Oliveira et al., 2019).

Desiccant materials used in dehydration retain water molecules present in the gas stream allowing the passage of dry gas (Tatlier, Munz, & Henninger, 2018). In literature, different adsorbents were studied to remove water vapor from gas mixtures, such as silica gel (Gandhidasan, Al-Farayedhi, & Al-Mubarak, 2001), alumina (Takbiri, Jozani, Rashidi, & Bozorgzadeh, 2013), 13 X zeolite (Nastaj & Ambrożek, 2015) and 3A molecular sieve (Farag, Ezzat, Amer, & Nashed, 2011). Within this context, we highlight the use of molecular sieves, especially the 4A molecular sieve. The number 4 (four) associated with the 4A molecular sieve represents the size of pore opening in Angstroms and good uniformity in the. Thus, the 4A molecular sieve can be used in adsorption processes because they have a high selectivity. It is also worth noting that the 4A molecular sieve can be regenerated and used again in several cycles of moisture adsorption, in order to restore its adsorption capacity. Currently, the TSA (Thermal Swing Adsorption) regeneration method is widely used in the drying of gases by the oil industry. In this method, the adsorbent is regenerated by increasing the temperature. However the TSA method has some disadvantages, such as premature aging of the adsorbent and energy inefficiency, in addition to not being suitable for fast cycles, because in this case the adsorbent would not be used at its maximum capacity (Netusil & Ditl, 2011).

Given these considerations, this work aimed to study the adsorption of water vapor in equilibrium conditions and fixed bed using molecular sieves of 4A zeolite as adsorbent. An experimental system was developed for this research. Thus, water vapor rupture curves were obtained in a fixed bed column of 4A zeolite molecular sieve and the influences of different flow rates and bed depths.

Materials and methods

Materials

The molecular sieve type 4A (Size of MS pellets: 3.2 mm; Specialty Chemicals Actor – CECA – Company [France]) was gently donated by the Petrobras Research Center. Carbon dioxide (Linde AG) was used as gas stream in the study of dehydration. Continuous dehydration experiments in a fixed-bed column were conducted in a PVC column in the form of a cylinder (Length x Diameter; 650 x 12.7 mm). Figure 1 provides details concerning the equipment used to carry out the dehydration experiments [(1) Carbon dioxide bottle, (2) Globe valve, (3 and 7) Manometer Rovenco, (4) Flowmeter (Omega FMA5520), (5) Retention valve, (6) humidification vase, (8 and 11) Humidity sensor (SHT20 – sensirion), (9, 10 and 12) Temperature sensor (MAX31855-Adafruit), (13) Throttling valve, (14) Load cell HX711, (15) Arduino MEGA, (16) Computer for data acquisition].

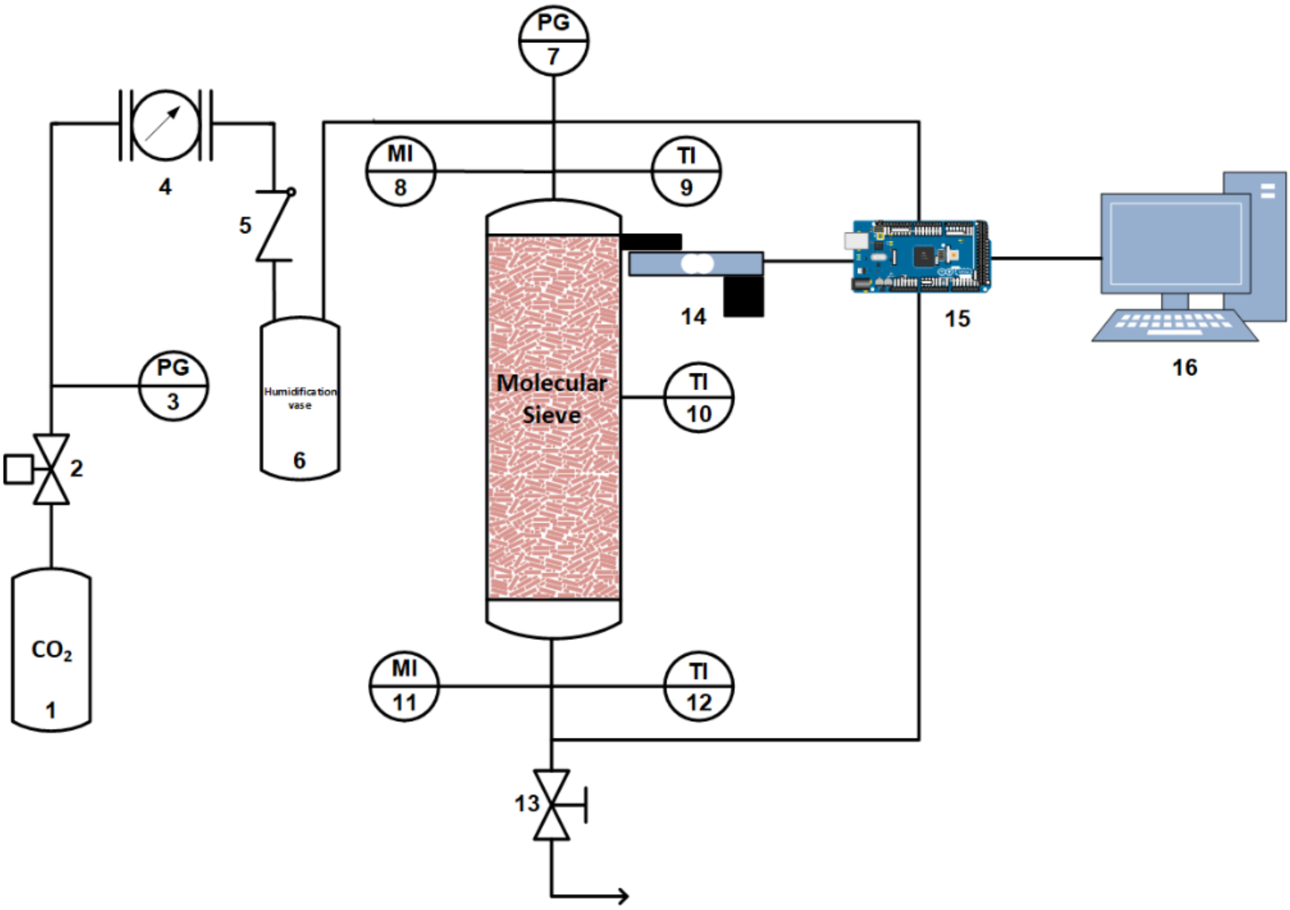

Figure 1. Schematics of the experimental apparatus.

Column studies

The effects of the following column parameters were studied during experimental work:

(i) Effect of flow rate: flow rate was varied between 2.0; 5.0 and 8.0 L min.$^{-1}$, while bed depths and inlet H_2O concentration were held constant at 20 cm and 20 mg L^{-1}, respectively.

(ii) Effect of bed depths: bed depths was varied between 10 (25 g), 20 (50 g) and 30 cm (75 g), keeping flow rate and initial H_2O concentration constant at 8.0 L min.$^{-1}$ and 20 mg L^{-1}, respectively.

Mathematical description

The mass transfer zone (MTZ) was calculated by Kumari, Mishra, Siddiqi, and Meikap (2021) and Alalwan, Abbas, Abudi, and Alminshid (2018) using experimental breakthrough data and mass balance.

$$q_b = \frac{C_0 Q}{m_{ads}} \int_0^{t_b} \left(1 - \frac{C_{(t)}}{C_0}\right) dt \qquad (1)$$

$$q_e = \frac{C_0 Q}{m_{ads}} \int_0^{t_e} \left(1 - \frac{C_{(t)}}{C_0}\right) dt \qquad (2)$$

$$MTZ = \left(1 - \frac{t_b}{t_e}\right) . h_{bed} \qquad (3)$$

where:
C_0 is the water vapor concentration at the entrance of the column (mg L^{-1}), Q is the fluid flow rate (L min.$^{-1}$), m_{ads} is the mass of dry adsorbent (g), $C_{(t)}$ is the water vapor concentration at the exit of the column in the saturation point (mg L^{-1}). Where h_{bed} is bed depths (cm); q_b is the amount adsorbed per unit of adsorbent at the breaking point, defined as the condition in which the output concentration is 5% of the input concentration; q_e is the amount adsorbed by mass of adsorbent at the saturation point, where the output concentration is 95% of the input concentration; It should be noted that the values corresponding to the integrals are given by the area under the curve 1- $C(t)/C_0$ versus time.

Breakthrough curves modelling

The model proposed by Yoon and Nelson (2010) was selected to describe the behavior of the rupture curve in the process of dehydration in a fixed bed column. Equation 4 represents the model, where k_{YN} (hour^{-1}) is the adsorption constant and τ (hour) is the time required for the saturation of 50% of the adsorbent. Equation 5 is used to calculate the maximum adsorption capacity q_{YN} (mg g^{-1}), C_0 is the concentration of water vapor at the entrance of the column (mg L^{-1}), Q is the fluid flow rate (L min.$^{-1}$) and W is the mass of the dry adsorbent (g).

$$\frac{C_{(t)}}{C_0} = \frac{1}{1 + exp\left(k_{YN}(\tau - t)\right)} \qquad (4)$$

$$q_{YN} = \frac{\tau C_0 Q}{W} \qquad (5)$$

Statistical analysis

Data were analyzed by the Tukey test of means comparison ($p \leqslant 0.05$), using Statistica 12.0 (StatSoft/USA) software.

Results and discussion

Effect of flow rate on breakthrough curve

The gas flow rate is an important parameter to analyze the performance of fixed-bed dehydration column. The effect of the feed flow rate on the adsorption of water vapor onto 4A molecular sieve was investigated by varying Q from 2.0 to 8.0 L min.$^{-1}$ while holding the other parameters constant. Figure 2 shows the breakthrough curves for different values of Q.

In general, increasing the flow rate decreases the breakthrough time. The rise in the gas flow rate (2.0 – 8.0 L min.$^{-1}$) has resulted in the reduction of t_b (5.0, 2.0, and 1.5 hour) and t_e (8.2, 3.5, and 2.8 hour). This result occurs because the time required for equilibrium between water vapor and 4A zeolite molecular sieve is higher than the retention time. Thus, the water vapor leaves the column before the attainment of equilibrium adsorption. It is also that as the inlet gas flow rate increases the adsorption capacity of water vapor decreases (Table 1). The adsorption capacity of water vapor is reduced from 168 to 126 mg g^{-1} ($p \leqslant 0.05$) with the increase of gas flow rate from 2.0 to 8.0 L min.$^{-1}$, respectively. This can be attributed to the decrease of the residence time of the gas as its flow rate increases (Farag et al., 2011). Furthermore, the increased gas flow rate reduces the thickness of external mass transfer film as well as resistance. The increment of gas flow rate causes the increment of overall mass transfer coefficient and mass transfer flux. Hence, the risen gas flow rate widens the mass transfer zone (MTZ – a Kumari et al., 2021). The mass transfer zone is the region within the adsorbent bed where the water vapor is actually being adsorbed on the adsorbent (Miretzky, Muñoz, & Carrillo-Chávez, 2006; Hernández-Abreu et al., 2020). A high flow rate implies a shorter contact time between water vapor and 4A molecular sieve which provides less time for equilibrium to be achieved, as a consequence the MTZ is expected to be larger for a flow rate of 8.0 L min.$^{-1}$ (Table 1). Similar observation is noted by Farag et al. (2011) when using the 3A molecular sieve in the dehydration of natural gas.

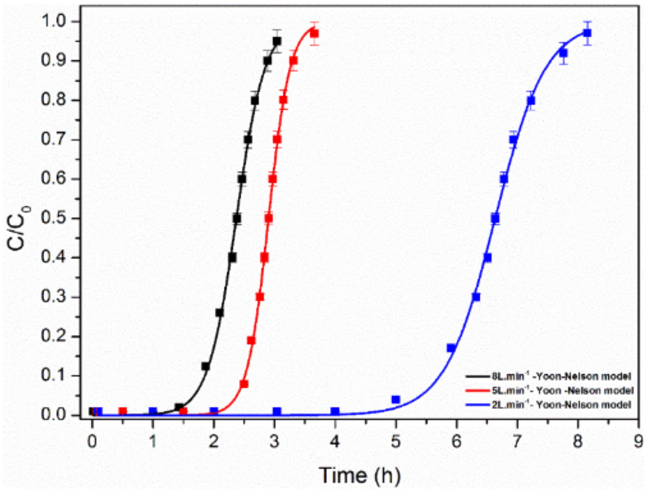

Figure 2. Breakthrough curves for adsorption of water vapor on 4A molecular sieve beds at different flow rates.

The breakthrough test result for the 4A molecular sieve in the fixed bed was fitted using the Yoon-Nelson theoretical model (Figure 2). The Yoon-Nelson model parameters K_{YN}, τ and q_{YN} for different flow rates are calculated based on the nonlinear regression Equation 4 and 5. The K_{YN} (hour^{-1}) and τ (hour) designate the velocity rate constant, and time taken for 50% water vapor breakthrough, respectively. The (τ) value decreased the increase in the gas flow rate. In addition, when the flow rate was increased, the (q_{YN}) magnitude vigorously declined, and (K_{YN}) increased (Table 2). In general, the estimated magnitudes for adsorption capacity (q_{YN}) were near to those values of the experiment under different flow rate. Furthermore, the coefficient of determination (R^2) obtained from the fits were greater than 0.9, indicating that the Yoon-Nelson model adequately represents the dehydration process of the 4A zeolite molecular sieve.

Table 1. Parameters obtained from the breakthrough curves analysis at different flow rates.

Q (in-1)	q_b (g)	q_s (mg g^{-1})	t_b (hour)	t_e (hour)	MTZ (cm)
2.0	8.4 ± 0.10^a	168 ± 2.0^a	5.0 ± 0.15^a	8.2 ± 0.10^a	7.8 ± 0.21^a
5.0	7.7 ± 0.15^b	154 ± 3.0^b	2.0 ± 0.07^b	3.5 ± 0.20^b	8.6 ± 0.25^b
8.0	6.3 ± 0.20^c	126 ± 4.0^c	1.5 ± 0.06^c	2.8 ± 0.10^c	9.3 ± 0.04^c

Equal lowercase letters in the same column do not present significant difference between the Tukey test ($p \leqslant 0.05$).

Table 2. Yoon-Nelson model parameters obtained for adsorption of water vapor on 4A molecular sieve beds at different flow rates.

Q (L min.$^{-1}$)	k_{YN} (hour^{-1})	τ (hour)	Q_{YN} (mg g^{-1})	R^2	q_{exp} (mg g^{-1})	Error (%)
2.0	2.40 ± 0.09^c	6.63 ± 0.15^a	168.6 ± 2.1^a	0.99	168 ± 2.0^a	0.3
5.0	3.63 ± 0.02^b	2.90 ± 0.10^b	156.6 ± 3.3^b	0.99	154 ± 3.0^b	1.6
8.0	4.24 ± 0.03^a	2.37 ± 0.08^c	127.4 ± 4.2^c	0.99	126 ± 4.0^c	1.1

Equal lowercase letters in the same column do not present significant difference between the Tukey test ($p \leqslant 0.05$).

Effect of bed depth on breakthrough curve

The effect of different bed depths (10, 20, and 30 cm) of H_2O on breakthrough curves is demonstrated in Figure 3. The fastest breakthrough curve of 10 cm bed depths and the slowest breakthrough curve of 30 cm bed depths were both experimental.

As the bed depths increased from 10 to 30 cm, the adsorption capacity increased as well as the quantity of water vapor removed (Table 3). The increment of bed depths (10, 20, and 30 cm) has contributed to the rise of breakthrough time (t_b) (0.5, 1.5, and 3.5 hour), in addition to exhaustion time (t_e – 1.5, 2.8, and 5.2 hour). The higher breakthrough time (tb) and higher exhaustion time (te) are favorable parameters for the improved performance of the column, which is reinforced by the obtained results. The parameters suggest that the adsorbent bed depths strongly influence the capture of water vapor in the column and thus the increased bed depth of the column provides enhanced contact time for the capture of water vapor. Additionally, the previous results also indicate that increased bed depth saturates more slowly compared to the bed of lower depth. Finally, the bending nature of breakthrough curve tends to increase with the increment of bed depth. Both the aforementioned changes lead to the widening of the MTZ. The higher bed depth is a result of resulted the higher amount of 4A molecular sieve which provides more active sites (Tan, Ahmad, & Hameed, 2008; Song, Zou, Bian, Su, & Han, 2011; Canteli, Carpiné, Scheer, Mafra, & Igarashi-Mafra, 2014). This could explain the change of adsorption quantity of water vapor (Table 3).

The evaluated values of various parameters of the Yoon-Nelson model are tabulated (Table 4). It indicates a good correlation coefficient ($R^2 > 0.9$) of Yoon-Nelson model to experimental data (Figure 3), suggesting that experimental data of the column study is following the Yoon-Nelson model.

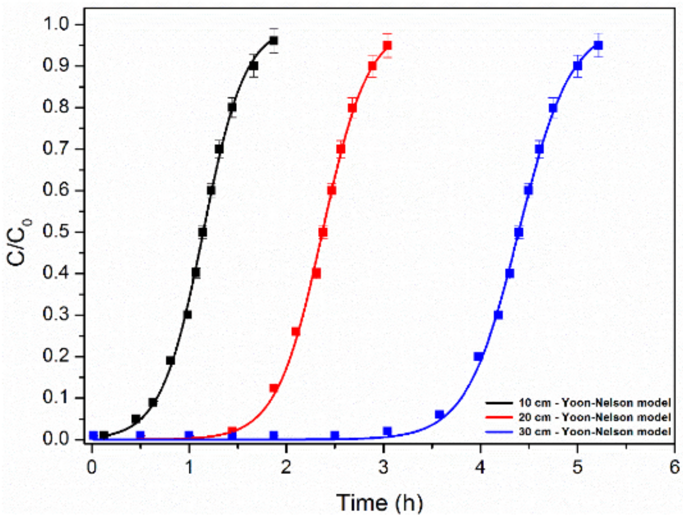

Figure 3. Breakthrough curves for adsorption of water vapor on 4A molecular sieve beds at different depths.

Table 3. Parameters obtained from the breakthrough curves analysis at different bed depths,

h_{bed} (cm)	q_b (g)	q_e (mg g^{-1})	t_b (hour)	t_e (hour)	MTZ (cm)
10	3.5 ± 0.10^c	140 ± 4.0^a	0.5 ± 0.02^c	1.5 ± 0.05^c	6.6 ± 0.04^b
20	6.3 ± 0.20^b	126 ± 4.0^b	1.5 ± 0.06^b	2.8 ± 0.10^b	9.3 ± 0.04^a
30	7.2 ± 0.36^a	96 ± 4.8^c	3.5 ± 0.15^a	5.2 ± 0.06^a	9.8 ± 0.61^a

Equal lowercase letters in the same column do not present significant difference between the Tukey test ($p \leq 0.05$).

Table 4. Yoon-Nelson model parameters obtained for adsorption of water vapor on 4A molecular sieve beds at different depths.

h_{bed} (cm)	K_{YN} (hour^{-1})	τ (hour)	q_{YN} (mg g^{-1})	R^2	q_{exp} (mg g^{-1})	Error (%)
10	4.60 ± 0.15^a	1.14 ± 0.06^c	142.2 ± 4.2^a	0.99	140 ± 4.0^a	1.5
20	4.24 ± 0.08^b	2.37 ± 0.07^b	127.4 ± 4.4^b	0.99	126 ± 4.0^b	1.1
30	3.71 ± 0.04^c	4.39 ± 0.10^a	98.3 ± 4.6^c	0.99	96 ± 4.8^c	2.3

Equal lowercase letters in the same column do not present significant difference between the Tukey test ($p \leq 0.05$).

The increase of bed depth results in the reduction of K_{YN} values and enhancement τ values. This occurs because of the increment amount of 4A molecular sieve. In conclusion, the values evaluated for the adsorption capacity by Yoon-Nelson model fitting (q_{YN}) are aligned with the data obtained in the experiment (q_e).

Conclusion

This study investigated experimentally the adsorption water vapor from gas streams using 4A zeolite molecular sieve as a low-cost adsorbent material in a continuous fixed-bed column. The experimental evidence confirms a major effect on the dehydration of gas streams based upon gas flow rate and a bed depth (based on the initial weight for the loaded 4 molecular sieve). The key findings of this study are:

– 4A zeolite molecular sieve has the potential of continuous dehydration while on operating column mode for the treatment of gas streams.

– As the flow rates increase, the breakthrough curve was steeper. With an increase in gas flow rate, the breakpoint time and the exhaustion time decreases.

– Breakthrough time and time of exhaustion increases as bed depth expands; While using a 30 cm bed depth the maximum adsorption quantity was achieved.

– The results obtained show that the Yoon-Nelson model can represent the behavior of fixed bed dehydration data.

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