

Experimental Study of Oxygen Separation in Oxygen-Pressure Swing Adsorption Unit

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The pressure swing adsorption (PSA) units are widely used as oxygen sources where oxygen is produced in a gaseous form. The start-up time of minutes is an undeniable advantage of PSA technology compared to cryogenic air separation having start-up time taking hours or days. The purity of oxygen produced by PSA using nitrogen selective zeolites (type A and type X zeolites) is limited to 95 % oxygen. The pilot-plant adsorption unit utilizes two-bed pressure swing adsorption technology. The nominal capacity of this unit is 1.4 kg h⁻¹ of gaseous oxygen with a purity of 95 % oxygen. The paper deals to analyze process characteristics of oxygen separation from the air in the pilot-plant adsorption unit for the adsorption pressure of 5.5 bar and the defined adsorption cycle. The effect of the number of cycles needed to obtain relevant results was also investigated.

1. Introduction

Skarstrom (1960) designed the first PSA technology for oxygen enrichment using nitrogen-selective zeolite 5A. The adsorbent was regenerated by blowdown and low-pressure purge using a part of the oxygen that was produced. Later, Guerin and Domine (1964) modified the Skarstrom cycle by regenerating the adsorbent under a vacuum. Adsorption at elevated pressure and desorption under vacuum are the principles of the hybrid pressure-vacuum PSA system (PSA/VPSA system). A comprehensive overview and insight into the science and technology of pressure swing adsorption are presented, e.g., by Ruthven et al. (1994).

The purity of oxygen produced by PSA using nitrogen selective zeolites (type A and type X zeolites) is limited to 95 % oxygen. The rest of the product stream is mainly argon. Argon-oxygen selective silver modified X-zeolites or carbon molecular sieves are used as adsorbents (Santos et al., 2007) to obtain higher oxygen purity, e.g. for medical purposes or welding. Šulc and Dítl (2021) analysed the potential of consumed energy-saving by integrating PSA technology as an oxygen source for the oxy-fuel combustion unit to reach an ecologically more sustainable process.

There are three basic process characteristics of PSA technology: i) product purity, ii) adsorbent productivity, and iii) recovery. Recovery is strongly affected by the purge flow. For a given type of PSA equipment, the optimum purge flow depends on the purity of the product and the adsorption pressure. Some manufacturers of oxygen PSA generators use the term 'air factor'. The air factor is the air ratio defined as the ratio of the air flowrate at the PSA inlet to the gaseous oxygen stream that is produced.

The paper deals to analyze process characteristics of oxygen separation from the air in the pilot-plant adsorption unit for the adsorption pressure of 5.5 bar and defined adsorption cycle. The effect of the number of cycles needed to obtain relevant results was also investigated.

2. Methods

2.1 Experimental set-up

The pilot plant adsorption unit O₂ (manufactured by OXYWISE Ltd., Slovakia) was used to carry out all the experiments. The adsorption unit utilizes two-bed pressure swing adsorption technology. The nominal capacity of this unit is 1.4 kg h⁻¹ of gaseous oxygen with a purity of 95 % oxygen. The PSA units require an inlet air quality of class 1.4.1 (solid particles, air humidity, oil). It is necessary to treat compressed air to achieve entering air

quality. A refrigeration air drying produces an air of the required pressure dew point of +3 °C. The adsorption unit is plotted in Figure 1 and the unit is shown in Figure 2.

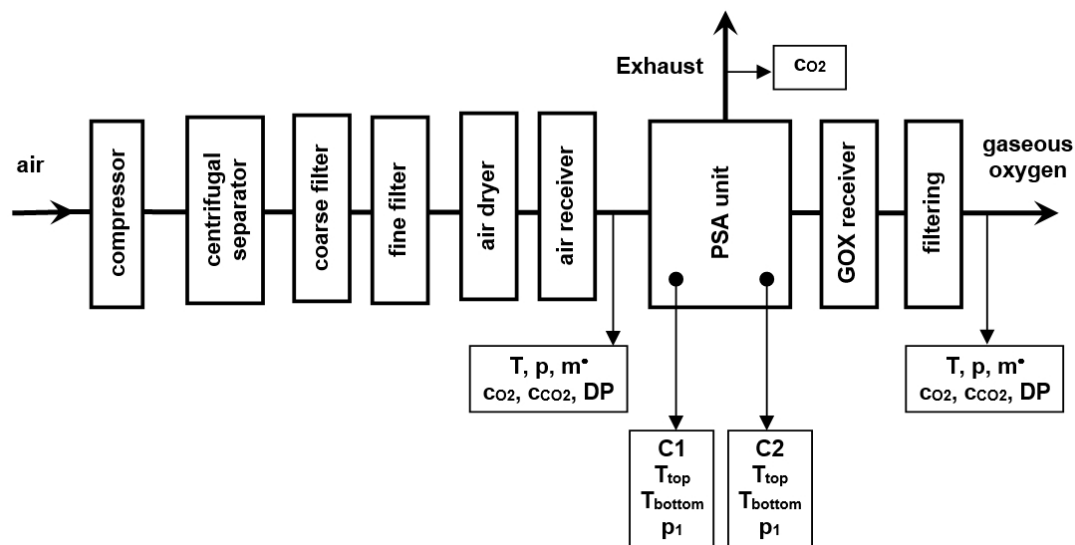


Figure 1: The technological schema of the pilot-plant adsorption unit



Figure 2: The pilot-plant adsorption unit: 1) compressor, 2) air dryer, 3) air receiver, 4) PSA columns, 5) GOX receiver, 6) exhaust pipe, 7) control unit, 8) mass flowmeter of inlet air, 9) mass flowmeter of product gas

The experiments were carried out for porous zeolite molecular sieve UOP MOLSIV™ PSAO2 XP (UOP LLC, Honeywell) with enhanced N₂/O₂ selectivity. Adsorbent particles are homogeneous spheres of 2 mm diameter (8 × 12 mesh) with a bulk density of 657 kg/m³. The adsorbent properties are presented in Table 1. The mass gas flow rate, the volumetric concentrations of oxygen and CO₂, and the dew point were measured in the air and product gas streams. The standard cycle consists of these sequential steps: 1) equalization, 2) pressurization, 3) production, 4) equalization, and 5) purging. At present, the industrially used cycles combine pressurization and production steps, as demonstrated in Figure 3. The following time intervals were used for experiment: i) 48 s for the pressurization, ii) 19 s for the equalization, and iii) 24 s for the production step.

Table 1: Properties of UOP MOLSITM PSAO2 XP

Particle diameter [m]	BET surface area [m ² g ⁻¹]	Outer surface area [m ² g ⁻¹]	Micropore area [m ² g ⁻¹]	Pore volume [m ³ g ⁻¹]	Micropore volume [m ³ g ⁻¹]	Particle porosity [-]	Bulk density [kg m ⁻³]	Particle density [kg m ⁻³]
0.002	523	40.2	247	3.47 × 10 ⁻⁴	2.47 × 10 ⁻⁴	0.38	657	1,095

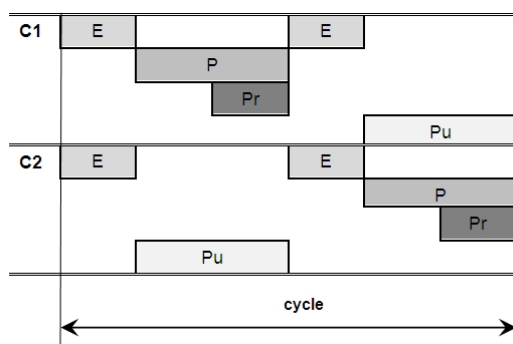


Figure 3: The scheme of adsorption cycle used for columns C1 and C2: E-equalization, P-pressurization, Pr-production, Pu-purging

2.2 Data analysis

The mean mass flowrate per cycle was calculated for each cycle: i) by the trapezoidal integration of instantaneous mass flowrate shown by the flowmeter used, and ii) using internal integration built in the flowmeter. The mean values of the oxygen content and the dew point in the air entering the PSA unit and the product gas were calculated by averaging instantaneous values recorded by sensors. The mean oxygen content in the exhaust stream per cycle was calculated for each cycle by the trapezoidal integration of instantaneous oxygen content measured by the oxygen sensor.

3. Results and Discussion

The analysis was executed for 10 cycles. The time courses of instantaneous values of the mass flow and the dew point are presented for the inlet air and the product gas in Figures 4 and 5. The time course of the oxygen content in the exhaust gas is shown in Figure 6.

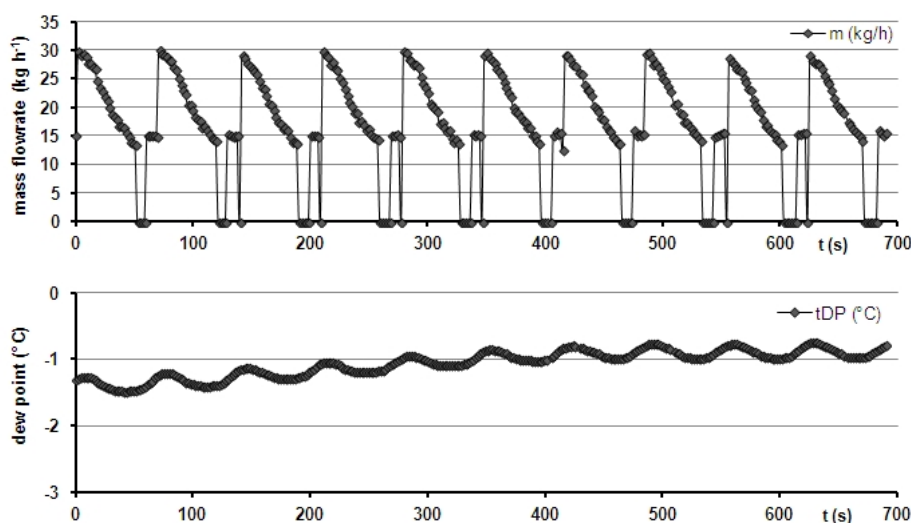


Figure 4: The inlet air: a) mass flowrate (upper), b) dew point (bottom)

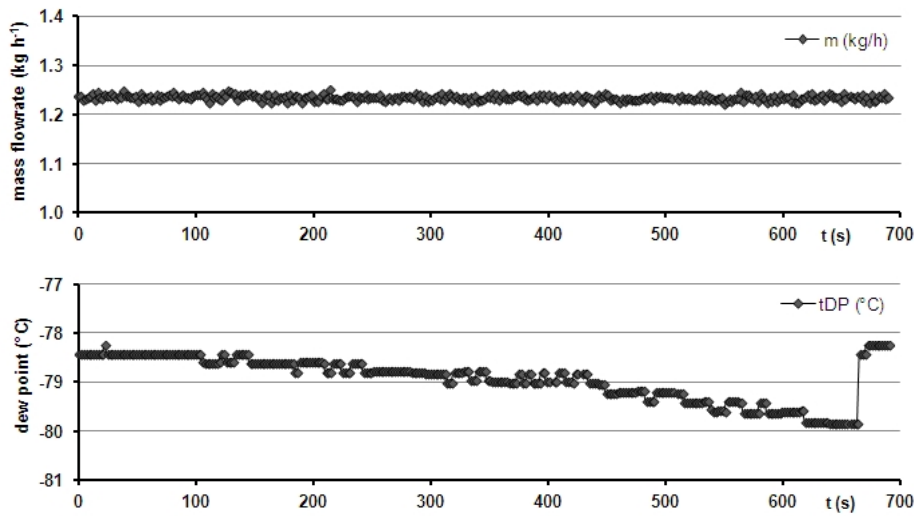


Figure 5: The product gas: a) mass flowrate (upper), b) dew point (bottom)

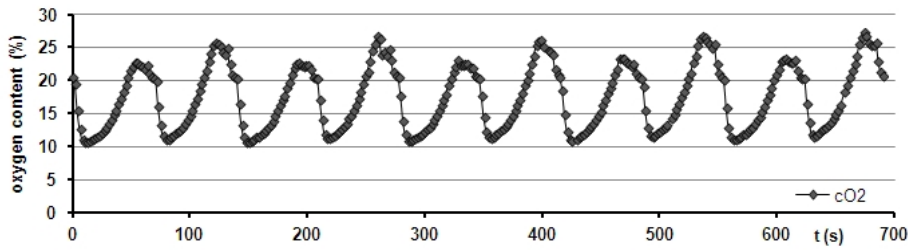


Figure 6: The exhaust gas: oxygen content

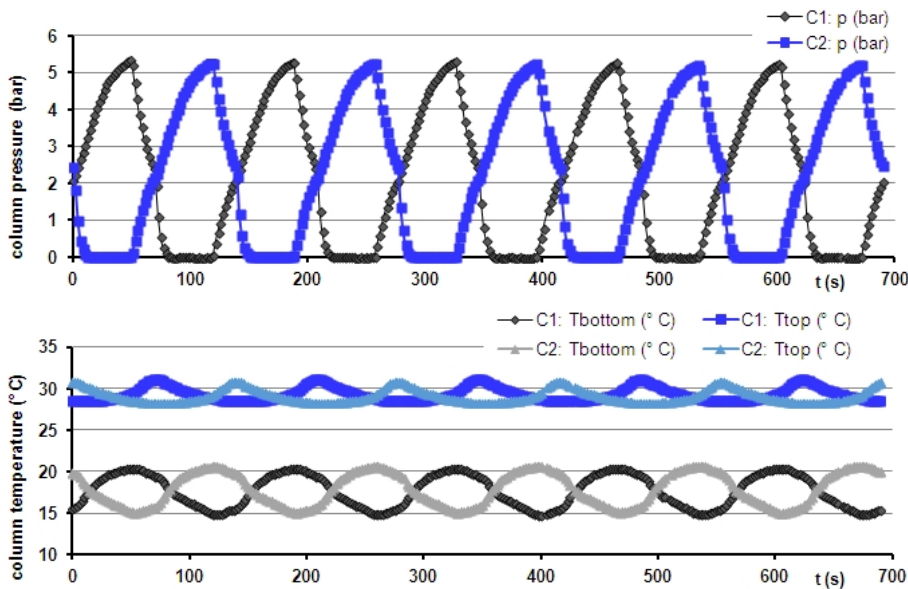


Figure 7: The adsorption cycle: a) column pressure (upper), b) column temperature (bottom)

The time courses of column pressure and the temperature at the top and bottom of the columns are presented for both PSA columns in Figure 7. The oxygen content, the dew point and flowrate evaluated for each cycle are presented in Table 2. Using averaged data for 10 cycles, the mass balance was calculated assuming no nitrogen

in the product gas and argon content of 0.94 mol % in inlet air. Water and CO₂ were not taken into account. The recovery and air factor were calculated. The calculated data are summarized in Table 3. The mass flowrates determined by the external trapezoidal integration are 1.3 % and 2.6 % lower compared to the internal integration for inlet air and product gas. This leads to a lower recovery value of 1.4 % and a higher value of the air factor by 1.4 % compared to the results based on internal integration. Therefore, data based on the internal integration function built in the flowmeter is recommended to be used.

Table 2: Stream composition and flowrate – evaluated data

Cycle No.	Inlet air				Product gas			Exhaust gas	
	mass flowrate (kg/h)		oxygen content (% vol.)	dew point (°C)	mass flowrate (kg/h)		oxygen content (% vol.)	oxygen content (% vol.)	
	-external	-internal			-external	-internal	dew point (°C)		
1	18.085	17.477	21.10	-1.37	1.236	1.240	94.40	-78.42	16.58
2	17.611	17.188	21.07	-1.30	1.202	1.240	94.41	-78.49	17.45
3	16.453	16.973	21.09	-1.20	1.198	1.241	94.41	-78.60	16.02
4	16.762	17.159	21.11	-1.11	1.198	1.209	94.43	-78.74	17.60
5	16.716	17.052	21.08	-1.02	1.198	1.243	94.44	-78.83	16.19
6	16.889	16.843	21.09	-0.93	1.199	1.246	94.44	-78.94	17.65
7	16.916	17.228	21.09	-0.89	1.197	1.238	94.44	-79.08	16.34
8	17.118	16.902	21.06	-0.87	1.198	1.235	94.46	-79.38	18.12
9	15.803	16.833	21.09	-0.87	1.198	1.241	94.47	-79.37	16.49
10	16.220	17.082	21.11	-0.85	1.199	1.210	94.47	-79.23	18.23
Average	16.857	17.074	21.090	-1.04	1.202	1.234	94.44	-78.93	17.07
b _{calc}	-0.041	-0.011			-0.01	-0.004			
t	3.4	2.8			3.3	0.7			
Hypothesis* ¹	No	No			No	Yes			

Note: *¹ Hypothesis is acceptable (Yes)/not acceptable (No). The critical t-distribution $t(8,0.05) = 2.306$.

Table 3: Mass balance

Cycle No.	Inlet air		Product gas		Exhaust gas	
	External integration	Internal integration	External integration	Internal integration	External integration	Internal integration
Composition (mol %)						
– oxygen	21.09	21.09	94.44	94.44	16.10	16.03
– nitrogen	77.97	77.97	0 ^{*2}	0 ^{*2}	83.27	83.35
– argon	0.94 ^{*1}	0.94 ^{*1}	5.56	5.56	0.63	0.62
Mass flowrate (kg h ⁻¹)	16.857	17.074	1.202	1.234	15.655	15.839
Volume flowrate (Nm ³ h ⁻¹)	13.044	13.211	0.831	0.853	12.213	12.358
Oxygen content (mass %)						
– experiment					17.07	17.07
– calculated					17.93	17.85
– relative error (%)					5.06	4.61
Recovery (-)			0.285	0.289		
Air factor (Nm ³ Nm ⁻³)			15.70	15.49		

Note: *¹ estimated according to dry air. *² no nitrogen assumed.

The effect of the number of cycles on the mass flowrate of inlet air and product gas was tested by hypothesis testing (Bowerman and O'Connell, 1997) for each flowrate separately. In this case, the independence of flowrate on the number of cycles was tested as the hypothesis, i.e. $m^* = B \cdot (N_{\text{cycles}})^0 = \text{const.}$, that is $\beta_{\text{pred}} = 0$. The critical value of the t-distribution for eight degrees of freedom and significance level $\alpha = 0.05$ is 2.306. The results of the hypothesis test are presented in Table 2. For illustration, the values of the calculated |t| value and the evaluated power-law exponent β_{calc} are presented here also. The independency of the mass flowrate on the number of cycles was found only for the product gas flowrate obtained by the internal integration function. Therefore, the effect of the number of cycles needed to obtain relevant results was investigated. Using the data obtained for each cycle, the cycle-averaged mean value of the flowrate was calculated for the averaging interval of various numbers of cycles. The number of cycles of the averaging interval that is necessary to stabilize the flowrate line (i.e. to reach the stable flowrate value) is the minimum number of cycles needed to obtain relevant

results. Using this approach, the minimum number of cycles was found at the time when the volatility of the cycle dependence of cycle-averaged flowrate is less than the defined percentage of stable value. The maximum measured error and repeatability of the air mass flow meter are 0.35 % and ± 0.25 % of reading. Therefore, the volatility range of ± 0.5 % was applied. The course of the mean value of flowrate on the number of cycles used to calculate the averaging is presented in Figure 8 including the volatility limit for air and internal integration. Using this approach and the applied volatility limit, a minimum of six cycles is needed to obtain relevant results.

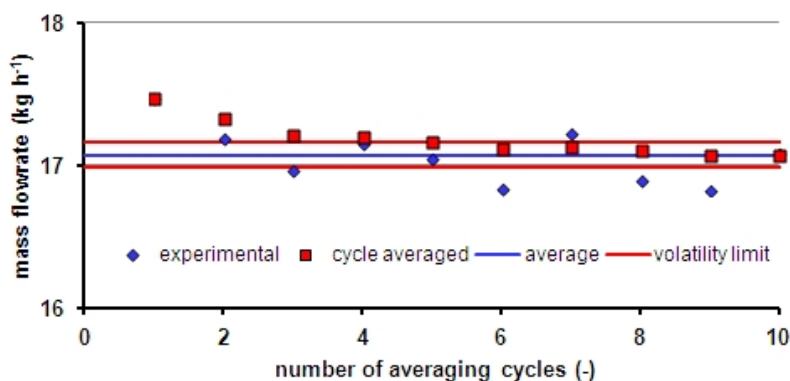


Figure 8: The minimum number of cycles – flowrate of inlet air by internal integration function

4. Conclusions

The process characteristics of oxygen separation from the air in the pilot-plant adsorption unit utilizing two-bed pressure swing adsorption technology were investigated for the adsorption pressure of 5.5 bar and the industrially used adsorption cycle, which combines pressurization and production steps. The experiments were carried out for porous zeolite molecular sieve UOP MOLSITM PSAO2 XP (UOP LLC, Honeywell) with enhanced N₂/O₂ selectivity. Data based on the internal integration function built in the flowmeter are recommended to be used for data treatment. Using averaged data for 10 cycles, the recovery of 0.289 and the air factor of 15.49 were evaluated for given conditions. Furthermore, the effect of the number of cycles needed to obtain relevant results was investigated. Using the proposed approach, the number of six cycles was found to be a minimum needed to obtain relevant results for the applied volatility limit of 0.5 % for the flowrate obtained by the internal integration function.

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