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Abstract: Compact arrangement of the disk type PSA columns results in considerable reduction of the equipment size and the effect of adsorption heat on process performance. More effort has been made presently to improve the performance of the new design in producing oxygen. Considerable improvement is observed due to the replacement of adsorbent ZMS-5A with ZMS-VP800 and the adjustment of the operation plan. The feasibility of the compact arrangement of adsorption columns for a PSA process is further proven.

Keywords: PSA equipment, compact design, disk column, oxygen

INTRODUCTION

In order to reduce the general size of a PSA equipment, a compact arrangement of disk type columns has been recently proposed (1). In some cases, the constraint on equipment size is very tight. For example, the space

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allowed for the equipment supplying oxygen to hydrogen fuel cells is quite limited on a vehicle. The oxygen producer for personnel usage if smaller is also the better. The PSA technology for producing oxygen from air is already matured (2–5). However, traditional adsorption columns are of cylinder shape and are arranged separately, which cannot satisfactorily meet the requirement of size reduction. The clearance between the cylinder columns is the final limitation on the reduction of the general dimension of the equipment. In this work, each column is reduced to a disk form and all disks are tightly stacked one above the other as shown in Fig. 1. Because the general dimension of a multibed PSA equipment is largely controlled by the size and location of columns, the new design will certainly reduce the total size of the equipment. Preliminary tests (6) prove that the new design PSA equipment is feasible in operation and also advantageous over the traditional design in that the thermal effect of adsorption can partially be compensated for. However, the performance of the prototype equipment needs to further improve; therefore, more tests are carried out on the same setup as described (6) previously. Major changes include the replacement of adsorbent ZMS-5A with ZMS-VP800, and an adjustment in the operation plan and disk position.

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EXPERIMENTAL

The disks are made of stainless steel. The dimension of a disk is 88 mm (o.d.) × 10 mm (wall thickness) × 10 mm (height). The adsorbent fills only the inner square part; therefore, the volume for filling adsorbent is only $48 \times 48 \times 10\,mm^3$ in each disk. The gas inlet and outlet are put on the center of the opposite arcs. Small granular beads and fiber materials fill the arc area to function as the gas stream distributor and/or solid filter, while the two side arcs are blinded. A thermocouple is inserted into the center of

Figure 1. The testing 4-bed disk type adsorber.

each disk column to record the temperature of the adsorbent bed. Detailed design graphs of the disk equipment and the experimental flowsheet have been presented previously (6). The operation plan is adjusted based on previous studies (7–11), and is presented in Table 1. A residual gas analyzer model QMS-100, purchased from Stanford Instrument, is used for on-line analysis of the gas stream composition. The signals of pressure, temperature, flowrate, and stream composition are transferred to a computer via different LabCards. The computer controls the action of valves and controllers using an A/D converter. All experiments are performed according to a prescribed program written by us in Turbo Basis language.

ZMS-VP800 of particle size 0.5–1.0 mm, purchased from Shanghai Elegant Molecular Sieve Co. Ltd, is used as the adsorbent in the present study. It is treated at 450°C for 7 hrs before adsorption experiments. For comparison, zeolite molecular sieves 5A and 13X purchased from the same company are also tested. To collect breakthrough curves, a gas mixture composed of helium (72.37%, the carrier gas), oxygen (5.8%) and nitrogen (21.83%) is used in experiments. The ratio of oxygen to nitrogen closes to that of air. All gases are of purity above 99.99%. To have a quantitative comparison between adsorbents, separation coefficient is defined and determined based on the breakthrough curves (12).

$$\alpha_{ij} = \frac{(x/y)_i}{(x/y)_j} \tag{1}$$

where x and y is the molar fraction of components i and j in the adsorbed phase and gas phase at equilibrium.

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RESULTS AND DISCUSSION

Comparison Between Adsorbents

Adsorption isotherms of nitrogen and oxygen on different adsorbents are collected with a typical volumetric method as described previously (6). The

Table 1. Step sequence in an operation cycle

I	A			PE	BD	IL	PG	IL		PE	IL	RP	
II	PE	BD	IL	PG	IL		PE	IL	RP	A			
III	PG	IL		PE	IL	RP	A			PE	BD	IL	
IV	PE	IL	RP	A			PE	BD	IL	PG	IL		

Note: I to IV: columns; A: adsorption; PE: pressure equalization; BD: blowdown; IL: idle; PG: purging; RP: re-pressurization.

results are presented in Fig. 2. The preference sequence of adsorbents according to nitrogen adsorption capacity is: VP800 > 5A > 13X, and that according to oxygen adsorption is: 5A > VP800 > 13X. The sequence of VP800 and 5A transposes for oxygen adsorption. Therefore, VP800 is a better adsorbent for producing oxygen with a PSA process. Breakthrough curves collected with VP800 at the total pressure of 0.4 MPa are shown in Fig. 3, where the ordinate, c, is the molar concentration of components. Breakthrough curves are also collected at the total pressure of 0.2, 0.3, 0.5 and 0.6 MPa. Separation coefficients at different total pressures are evaluated and shown in Fig. 4 for VP800 and 5A, respectively. The priority of VP800 over 5A is clearly shown.

Effect of Adsorption Pressure

Adsorption pressure is an important condition for PSA operation. Breakthrough curves on VP800 at flowrate 230.23 Nml/min and different pressures are shown in Fig. 5, where c/c_0 is the ratio of a temporary composition to that initial. The breakthrough time increases remarkably with the total pressure until 0.4 MPa. It does not change much as pressure further increases. Breakthrough time relates closely to the productivity per unit volume of adsorbent; therefore, the total pressure of 0.4 MPa is appropriate for further tests. The highest overshoot was observed in oxygen curve at 0.4 MPa. Because overshoot is caused by the replacement of the adsorbed oxygen with nitrogen, the higher the overshoot peak the better the separation

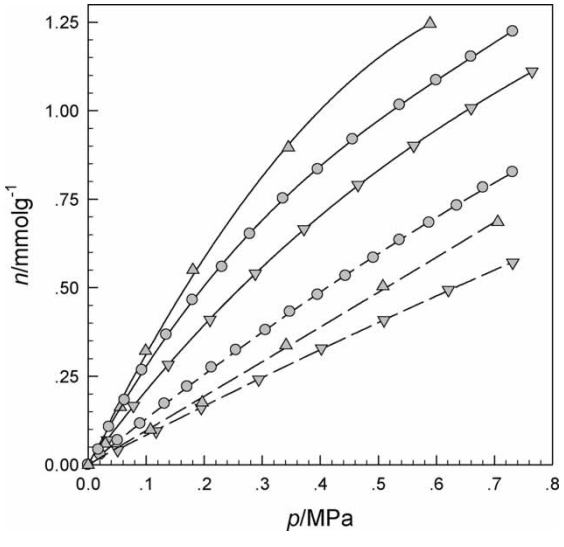

Figure 2. Adsorption isotherms at 298 K ——: N_2; – – –: O_2; △: VP800; ▽: 13X; o: 5A.

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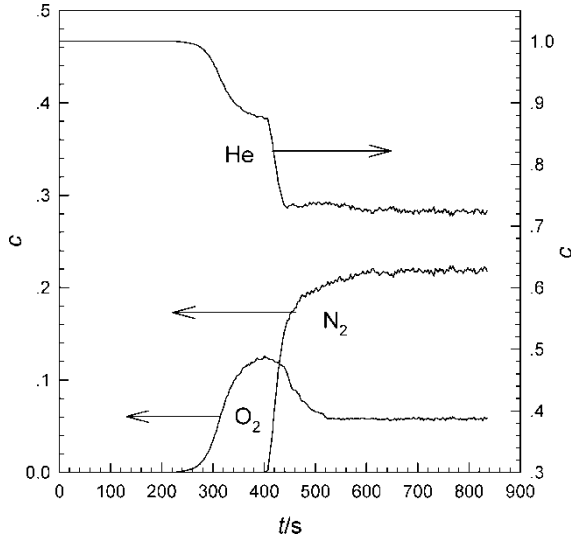

Figure 3. Breakthrough curves on VP800 at total pressure 0.4 MPa.

between the two components (2). A comparison of the operation performance is made for adsorption pressure of 0.4 MPa and 0.2 MPa, and the result is shown in Table 2. Lower adsorption pressure yields higher product recovery, productivity (in normal litter of product per kg adsorbent) and

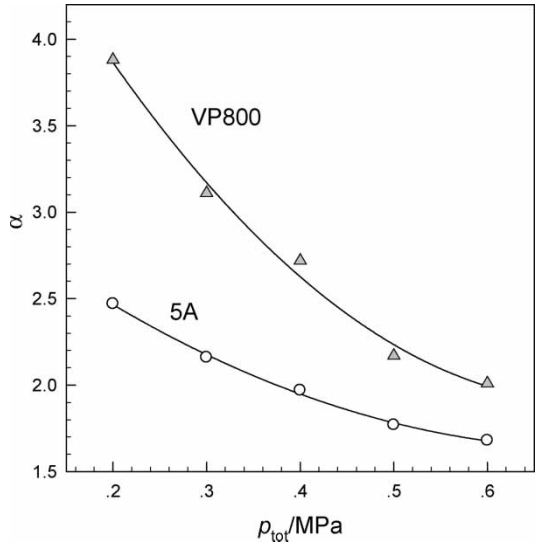

Figure 4. Separation coefficient as a function of the total pressure.

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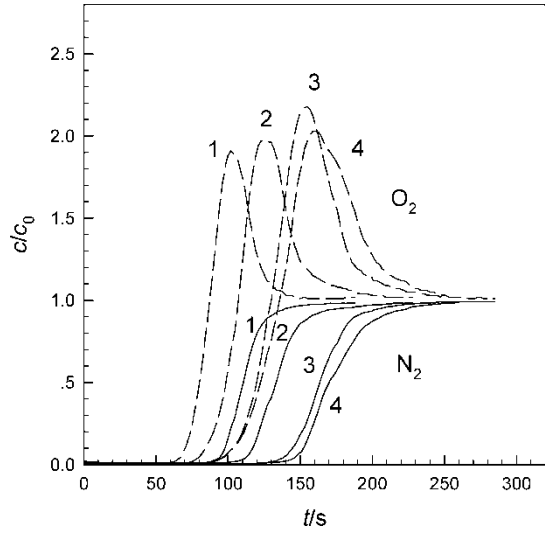

Figure 5. Breakthrough curves on VP-800 at different pressures (flowrate = 230.23 N ml/min) 1: 0.2 MPa; 2: 0.3 MPa; 3: 0.4 MPa; 4: 0.5 MPa.

higher energy efficiency. However, it requires higher adsorption pressure to obtain higher concentration of oxygen.

Effect of Disk Position

The disk column is horizontally located in previous study (6) as shown in Fig. 1. To test the effect of disk direction, performance is tested for the two disk positions at different purging ratio. As shown in Fig. 6, there is one or two percentage difference in the recovery or product concentration between the two positions. Although the difference is not much, it might become larger as the bed size increases considerably; therefore, the vertical position is preferred over the horizontal.

Table 2. Operation result at two adsorption pressures

Adsorption pressure/MPa	t_A/s	O_2%	Recovery, %	Productivity (NL/hkg)	Energy cost (kwh/Nm^3)
0.4	35	84.3	12.95	24.12	1.669
0.4	40	80.9	29.29	52.51	0.738
0.4	45	74.6	29.22	48.61	0.74
0.2	16	66.5	26.86	39.24	0.402
0.2	20	58.3	45.92	58.87	0.235
0.2	25	48.9	46.55	50.14	0.232

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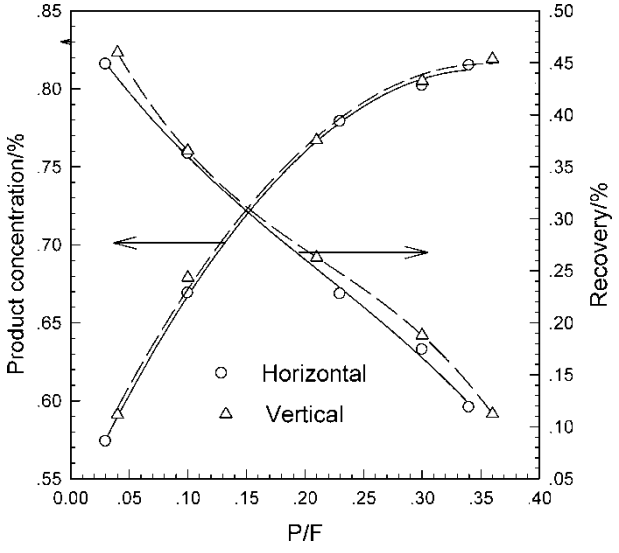

Figure 6. Effect of purging ratio for different position of disk columns (flowrate = 284.67 nml/min, room temperature = 18.8°C).

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Time Allocation for Each Step of an Operation Cycle

Nitrogen breaks the adsorption bed at 90 s when the bed has been pressured to 0.4 MPa with pure oxygen. Therefore, the adsorption time must be much less than 90 s. The effect of different adsorption times is shown in Fig. 7. Selection of the adsorption time is actually to make a trade-off between concentration and recovery of product. Practical selection depends on the requirement of a specific application. For example, if 40 s is selected as the adsorption time, t_A, the product will contain 80.9% oxygen with a recovery of 29% and energy consumption of 0.738 kwh/(Nm^3O_2). Since the adsorption step proceeds consecutively in four columns, an operation cycle will last for 160 s.

The time for pressure equalization between a pair of columns, t_{PE}, also affects the performance of a PSA process. Typical effect is shown in Figs. 8 and 9 for the condition of: adsorption pressure 0.4 MPa, $t_A = 40$ s, and P/F = 0.29. It seems that the process performance is quite sensitive to the time duration of the pressure equalization step. Four s is selected for t_{PE} in subsequent tests.

Figure 10 shows that the purging time exerts considerable effect on the product concentration and recovery for a definite value of purging ratio, and there is an optimal purging time between 6 and 10 s for the condition tested. However, six s was used for t_{PG} in experiments due to missing a timely data fitting.

The blowing down time is that needed for the pressure released to the atmospheric following the step of pressure equalization. It is 20 s for the

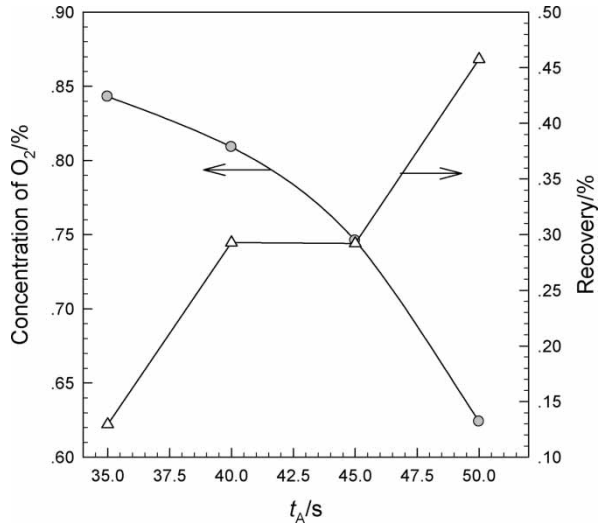

Figure 7. Effect of adsorption time on the performance at 0.4 MPa.

adsorption pressure of 0.4 MPa. The step of re-pressurization is necessary after pressure equalization in order to reach the adsorption pressure. There is some successful experience for pressure equalization aiming for higher product concentration and recovery (13). In this present study, re-pressure is completed by the product stream that counter-currently enters the column

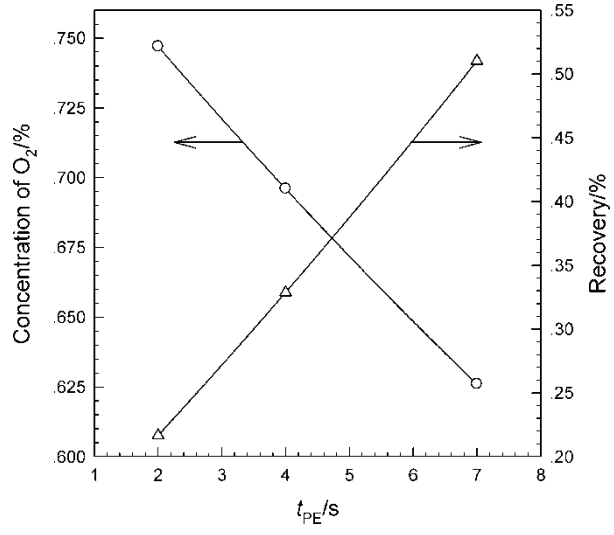

Figure 8. Effect of pressure equalization time on the product concentration and recovery.

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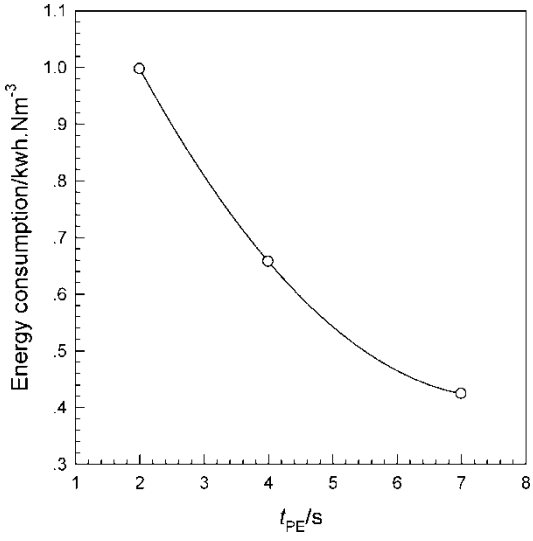

Figure 9. Effect of pressure equalization time on the energy cost of product.

from the exit, and is complemented by a stream of raw mixture that co-currently enters the column from the inlet at the last 1 or 2 s of the total re-pressurization time, t_{RP}. The effect of re-pressurization time on product concentration is less than 1%, and less than 0.05% on recovery for the

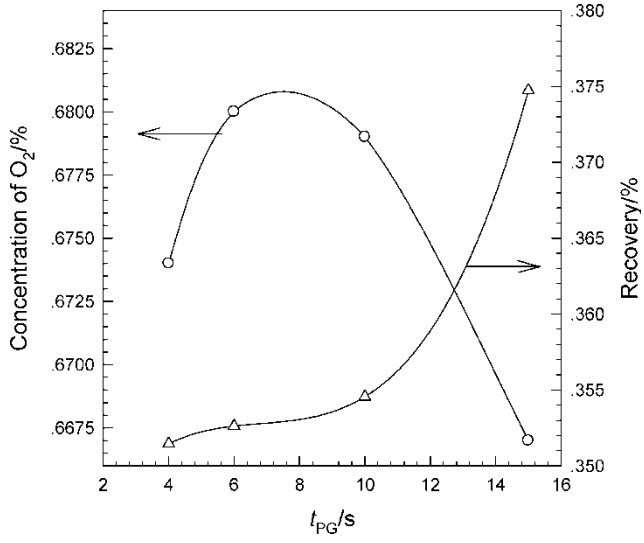

Figure 10. Effect of purging time on the product concentration and recovery (Adsorption pressure: 0.4 MPa; $t_A = 40$ s; P/F = 0.28).

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range of 20–30 s. However, t_{RP} must comply with the following constraint exerted by the operation plan shown in Table 1:

$$t_{RP} = \begin{cases} t_A - t_{PG} & t_{PG} \geq t_{PE} \\ t_A - t_{PE} & t_{PG} < t_{PE} \end{cases} \quad (2)$$

The pressure stability of the product tank is affected remarkably by the re-pressurization time. Longer re-pressurization time yields smaller amplitude of pressure fluctuation. Therefore, 30 s is allocated for this step. Some time clearance is allowed for idle to make up a continuous cycling of an operation period of 160 s.

Variation of Bed Temperature in an Operation Cycle

As shown in Figure 11, the bed temperature fluctuates between 23°C and 33°C. The temperature raises only about 2°C in the adsorption step; therefore, adsorption is quite close to isothermal. The bed temperature decreases in the following steps, but rises again to the adsorption temperature in the re-pressurization step. The small variation amplitude of the bed temperature may be attributed to the compact arrangement of the disks since the adsorption and desorption heat can partially be compensated for.

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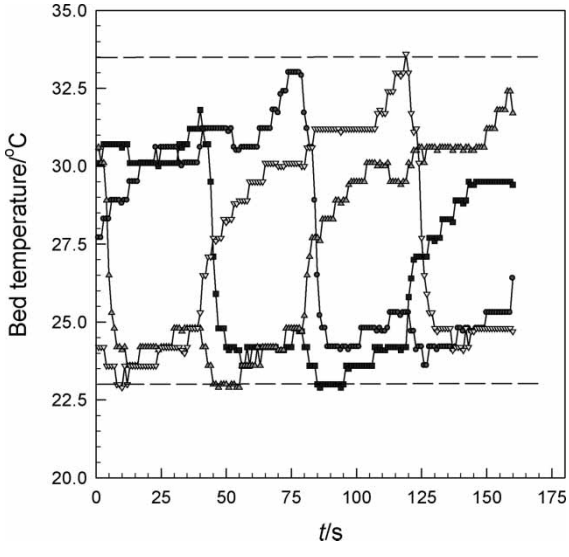

Figure 11. Variation of the bed temperature in a cycle.

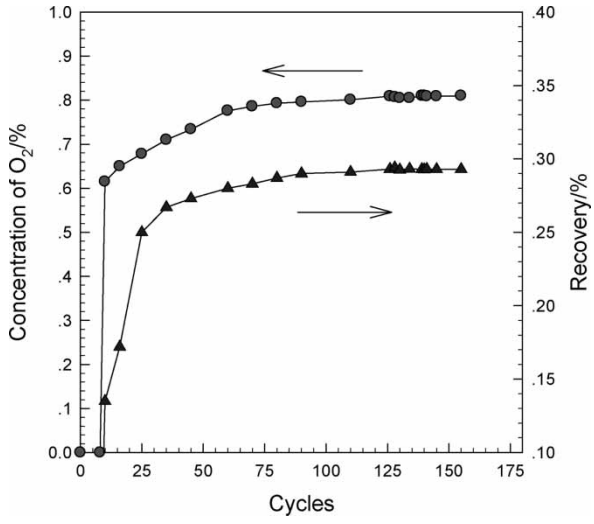

Figure 12. Performance in a multicycle operation.

Process Performance in Multi-Cycle Operations

Operation stability is of concern for the new arrangement of adsorption columns especially for the small disk size and a relatively complicated operation plan. Therefore, an operation test is conducted for 160 cycles under the condition that follows: Adsorption pressure 0.42 MPa, $t_A = 40$ s, $t_{PE} = 4$ s, $t_{BD} = 20$ s, F/P = 0.45, $t_{PG} = 6$ s and $t_{RP} = 30$ s. Variation of product concentration and recovery is shown in Fig. 12. The process begins to produce product after 10 cycles, but the product reaches stable concentration of 81% after about 80 cycles. The recovery reaches a stable value of 29% earlier at about 30 cycles.

Summary

Performance improvement of the disk type PSA unit made in present study is summarized in Table 3. The operation pressure reduces from 0.6 MPa to

Table 3. Comparison of operation performance using different adsorbents

Adsorbent	Flowrate ml/min	Adsorption pressure/ MPa	Regeneration pressure/ MPa	O_2 content in product	O_2 Recovery	Energy cost kwh/ Nm^3
5A	269.7	0.6	0.1	63.9%	24.0%	1.255
VP800	284.67	0.4	0.1	80.9%	29.3%	0.738

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0.4 MPa while a larger feed flow rate is maintained. Oxygen concentration in product rises from 63.9% to 80.9%, and the recovery rises from 24.0% to 29.3%, and the energy cost reduces from 1.255 to 0.738 kwh/Nm^3.

CONCLUSIONS

Present study further proves the feasibility of the compact disk columns for PSA operations. Considerable improvement is obtained due largely to the replacement of adsorbent ZMS-5A with ZMS-VP800. However, the flow channel in the testing disks is only 48 mm long, for which the end effect of the adsorption bed must be remarkable. Therefore, better operation performance can be expected in longer disks.

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REFERENCES

1. Zhou, L. and Zhou, Y.-P. (2001) Compact structure design of the small PSA equipment of producing oxygen. Chinese patent ZL 01141964.4.
2. Yang, R.T. (1987) *Gas Separation by Adsorption Processes*; Butterworths: London.
3. Ruthven, D.M., Farooq, S., and Knaebel, K.S. (1994) *Pressure Swing Adsorption*; VCH Publishers: New York.
4. Sircar, S. and Kratz, W.C. (1988) A pressure swing adsorption process for production of 23-50% oxygen-enriched air, *Sep. Sci. & Technol.*, 23: 437–450.
5. Yoshida, S., Ogawa, N., Kamioka, K., Hirano, S., and Mori, T. (1999) Study of zeolite molecular sieves for production of oxygen by using pressure swing adsorption, *Adsorption*, 5: 57–61.
6. Zhou, L., Li, J., Zhou, Y.-P., Su, W., and Sun, Y. (2005) Experimental studies of a new compact design 4-bed PSA equipment for producing oxygen. *AIChE Journal*, 51 (10): 2695–2701.
7. Berlin, N.H. (1966) Method for providing an oxygen-enriched environment. US Patent No. 3280536.
8. Mendes, A.M.M., Costa, C.A.V., and Rodrigues, A.E. (2001) Oxygen separation from air by PSA: modeling and experimental results Part I: isothermal operation. *Sep. & Pur. Technol.*, 24: 173–188.
9. Knaebel, K.S. and Kayser, J.C. (1986) Pressure swing adsorption: experimental study of an equilibrium theory. *Chem. Eng. Sci.*, 41: 2931–2938.
10. Shirley, A.I. and LaCava, A.I. (1993) Novel pressure methods in pressure swing adsorption systems for the generation of high-purity gas. *Ind. Eng. Chem. Res.*, 32: 906–910.

11. Farooq, S., Ruthven, M., et al. (1989) Numerical simulation of a pressure swing adsorption oxygen unit. *Chem. Eng. Sci.*, 44: 2809–2816.
12. Zhou, L., Guo, W.-C., and Zhou, Y.-P. (2002) A feasibility study of separating CH_4/N_2 by adsorption. *Chinese J. Chem. Eng.*, 10: 558–561.
13. Batta, L.B. (1972) Selective adsorption gas separation process. US Patent 3636679.

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