Experimental Study on a Four-Bed PSA Air Separation Process

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A 200-s PSA cycle involving both pressure equalization and product backfill steps has been experimentally studied on a four-bed system, where LINDE 5A zeolites were used as the adsorbent to separate oxygen from air. This cycle is operated under a pressure ratio of 4.3. During the experiment, the pressure history and flow rates, as well as the concentration of the product stream have been continuously monitored. This is the first time detailed experimental data on a four-bed system are presented. Under favorable conditions, this system produces better than 90% oxygen at a recovery of 17%. For the low-pressure ratio, such a recovery could not have been achieved without the pressure equalization step and the reduced purge operation. Recovery and throughput, however, are not as high as one would expect from a linear local equilibrium model. The self-broadening effect of the purge wave has been identified as the major cause of underperformance.

Introduction

Pressure swing adsorption (PSA) is a low-cost, highly efficient, and highly selective gas separation process. It has been successfully applied in many industrial problems including the separation and drying of air, the purification of hydrogen, and the separation of either carbon monoxide, carbon dioxide, hydrogen or methane from the syngas or coke oven gases. Zeolites, carbon molecular sieves, and active carbon are the most commonly employed adsorbents. Selectivity based either on adsorption equilibria or on adsorption rate have been capitalized. Cycle time as long as several minutes or as short as tens of seconds can be found in existing units.

The original PSA cycle proposed by Skarstrom (1972) involves four operation steps; that is, pressurization, production, blowdown, and purge. Developments in the past 30 years have led to several additional operations that greatly improve the process efficiency. The theoretical foundations of these process improvements, however, have only been established in recent years. For example, pressurization with product (a backfill step) is proven to increase the recovery of the light product by reducing the feed penetration (Knaebel and Hill, 1985; Liow and Kenney, 1990). A pressure equalization step may reduce the total feed gas utilized. A forward depressurization step is shown to concentrate the heavy component (Cen and Yang, 1985). Finally, a savings on purge gas could be made when a large fraction of the feed is the heavy component (Matz and Knaebel, 1988).

To implement these beneficial steps in a PSA cycle, more than two adsorption columns must sometimes be involved. Multibed PSA cycles involving both pressure equalization and product backfill steps are common in industrial processes, but detailed process data have seldom been disclosed. In the meantime, all the published experimental studies have been limited to systems with one or two adsorption columns. For example, the production of nitrogen from air with a single 4A zeolite column was examined by Shin and Knaebel (1988), as a typical kinetic controlling PSA process. A single column PSA system was presented by Matz and Knaebel (1988) to demonstrate the effect of incomplete purge. Results involving the separation

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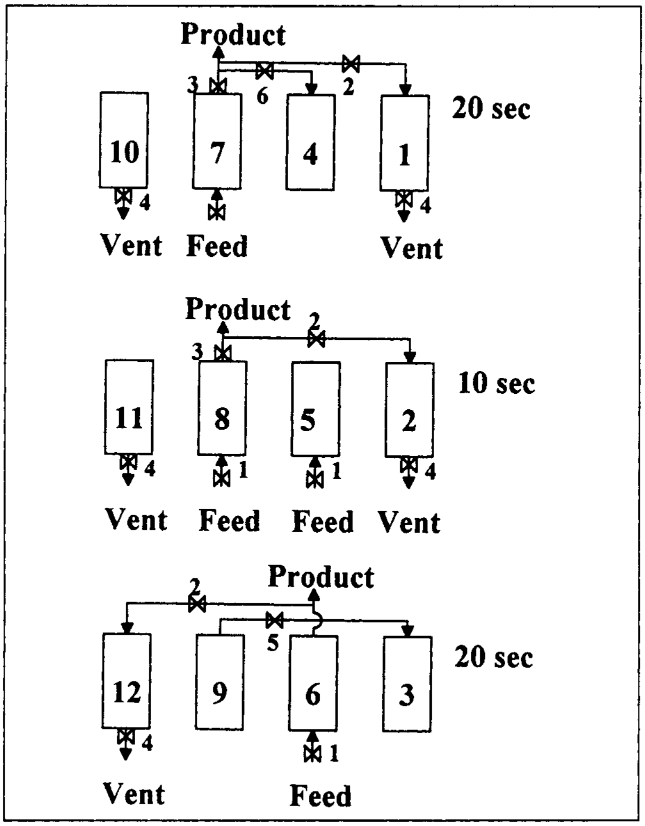

Figure 1. Operation schedule of four column PSA process studied.

of methane, carbon dioxide, and hydrogen in a single column of carbon molecular sieve, as well as the production of nitrogen by a 4A zeolite column, were reported by Yang and his colleagues (Cen and Yang, 1985; Doong and Yang, 1986; Kapoor and Yang, 1989; Ackley and Yang, 1990). A 5A zeolite packed bed was employed by Chiang et al. (1988) to examine the oxygen enrichment of air.

Studies which employed dual adsorption columns could be traced back to the original work of Skarstrom (1972). Two 5A zeolite packed beds were recently utilized by Kayser and Knaebel (1986) for the investigation of air separation. Hassan et al. (1986) also studied the air separation process with two carbon molecular sieve beds. An investigation involving dual 5A beds for oxygen enrichment was reported recently by Chou et al. (1992).

The fundamental principle of a multicolumn PSA process is exactly the same as that of a single column one. However, due to the interconnection of different columns, the system dynamics may be more complicated. Simple trial and error is unlikely to optimize the operation conditions of a multicolumn system. Guidance from theoretical analysis or from model simulation are therefore necessary. The optimum operation conditions may, in some cases, be predicted from simplified models. For example, Chiang (1994) developed an analytical solution to a system with linear isotherm under local equilibrium conditions. It is not clear in which direction the predicted operation parameters should be modified when the isotherm is not linear.

Experiments are therefore done on a multicolumn PSA process to study the difference between a nonlinear system and the prediction from linear local equilibrium theory. In particular, the pressure ratio has been kept low, where the linear theory predicts the largest gain on recovery. Furthermore, these data will enable the verification of a simulation program developed by us, where the nonlinear isotherms as well as the thermal effects have all been taken into account (Chou et al., 1992; Chou and Huang, 1993).

Experimental Designs

The layout of the pilot PSA system is provided in Figure 1. The process lines used were all 1/2-in. (23-mm) steel pipes. The adsorption columns were constructed from 4-in stainless steel pipes (Schedule 40) with an inside diameter of 102.3 mm. They were one meter in height, of which only 80 cm was filled with the adsorbent. The rest of the column volume was occupied by flow distributors and stainless steel screens on both ends. The valves indicated in the figure actually consisted of several units including pressure regulators, check valves, and solenoid valves. The combined flow coefficients represented by valves 4, 5, and 6 could be back calculated from the transient flow rates and the pressure history of the columns to give values of 0.65, 0.2, and 0.085, respectively. These coefficients remained constant throughout our study. The flow coefficients represented by the other valves varied with the operation conditions.

The feed gas was taken from the utility air supply in China Steel Co., which had a dew point of roughly −10°C and a maximum pressure of 709 kPa. The Linde 5 A zeolite was obtained in the form of 1/16-in. pellets. The amounts of zeolite packed in each column were 5.25, 5.27, 5.30, and 5.26 kg, respectively. The bulk density of the adsorbent was measured as 1.21 g/cm^3 by a mercury pycnometer. The zeolites were put in an open basket and activated at 350°C for 24 h in an open oven. The room temperature adsorption isotherm of this zeolite was confirmed to follow the Langmuir-Sip relationship reported by Miller et al. (1987) as indicated in Figure 2. These equilibrium data were obtained from breakthrough curves of nitrogen, oxygen, and air under various pressures.

The cycle time and the operation sequences of the systems were controlled by a Hitachi programmable logic controller through individual solenoid valves. An OM-11 oxygen analyzer from Sensor Medic Co. (U.S.) was used to follow the product oxygen concentration. This analyzer, however, could not detect the presence of argon. We had to account for the amount of argon in the system by assuming that argon has a similar adsorption characteristic as oxygen. Mass-flow meters, HFM-201 from Teledyne (U.S.) were placed in the feed, the product, and the purge streams. In addition to the pressure gauges on every column, transducers were placed on one particular adsorption column to follow the pressure history. Initially, our process design included a multipoint thermal couple in the column. However, such a design did not function well in another dual bed setup. As a consequence, we discontinued the temperature wave measurement.

The outputs of these instruments were connected through appropriate interfaces to a personal computer. The PC monitor provided an on-line display of the dynamic change of all meas-

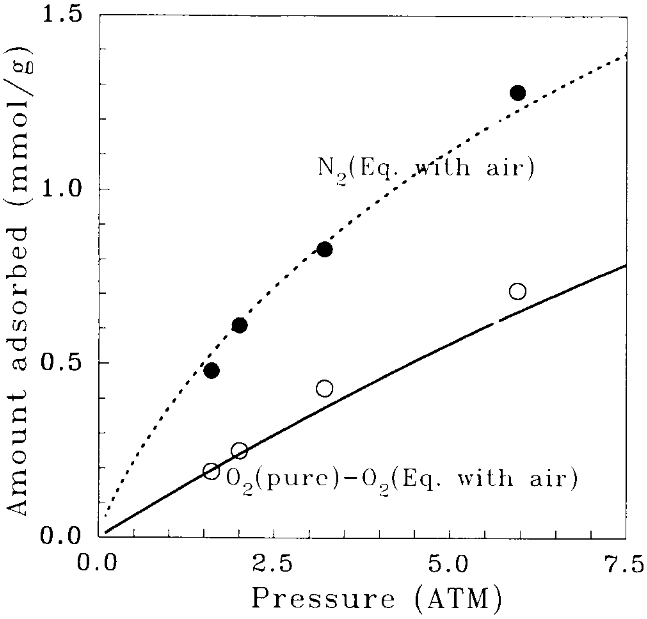

Figure 2. Comparison of experimental equilibrium data with Langmuir-Sip isotherm given by Muller et al. (1987).

ured variables. The cyclic steady-state condition could be easily identified from the display. When the cyclic steady state was reached, the data collected were immediately integrated to obtain the oxygen recovery, purge quantity, productivity, and so on.

There were a total of eight steps to each process cycle. The pressure of the feed gas was regulated to 456 kPa. The pressure of the product stream was set at 380 kPa. Following each adjustment of pressure or flow rate, it took approximately 6 to 8 h to reach a cyclic steady state. This process scheme was evolved from a series of preliminary designs. The product concentration was low and unstable when the product backfill step was omitted. With the scheme presented, the product oxygen concentration stayed reasonably stable once the cyclic steady state was reached.

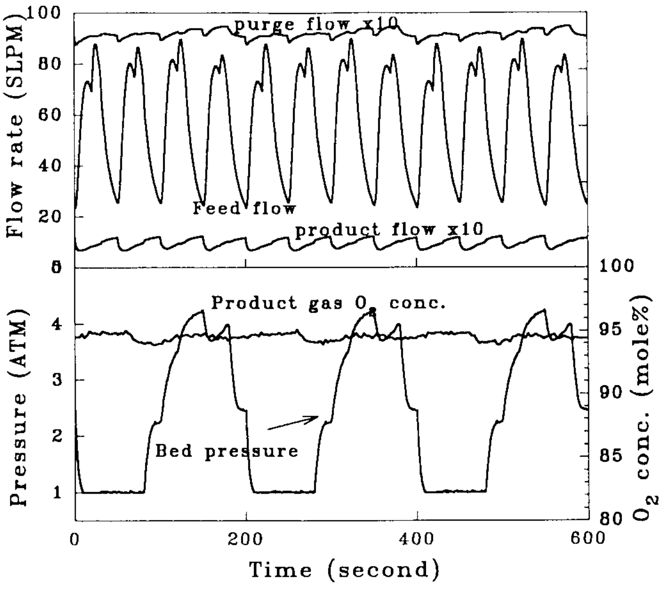

Figure 3. Dynamic data collected in typical experimental run.

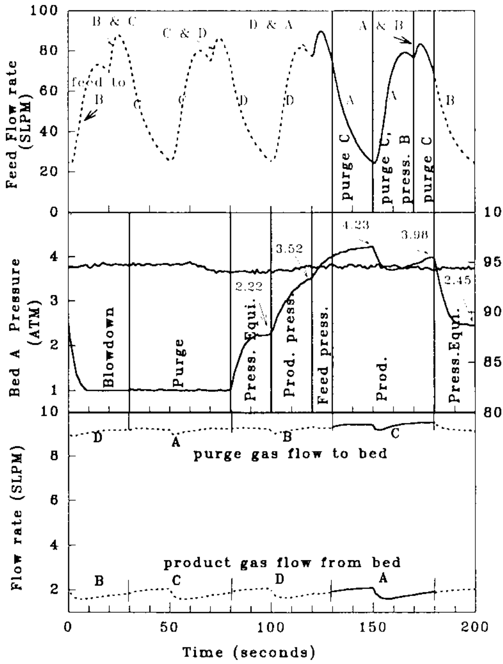

Figure 4. Dynamics of typical experimental cycle.

Compress air is fed to different columns at different steps as indicated on top graph. Pressure of column A is given in middle graph. Rate of production and purge are indicated in bottom graph.

Results and Discussion

A typical example of the data collected during the experiments is shown in Figure 3. This figure represents a period of three operation cycles. The transient flow rates, product composition, as well as the pressure history have all been recorded. This figure shows that the four adsorption columns were not operated under exactly the same condition. The feed rate exhibits cyclic variations when flowing into different columns. This variation suggests that a different flow resistance has been encountered during the backfill of a particular column. The same reason can also explain the slight drop of product concentration when this column was producing. We are not certain about the origin of this difference in flow resistance.

The same data are magnified in Figure 4, where the various operations occurred during a cycle are also indicated. We will now follow Figure 4 and go through our process cycle step-by-step.

(1) The given cycle started from the blowdown of column A and the feed repressurization of column B. After finished with blowdown and purge steps, column A was first partially pressurized by equalizing its pressure with column C. Then, it was further pressurized by the product gas from column D. It, however, was not possible to pressurize column A to the feed pressure in this step, since the product is always at a lower

pressure than the feed. An additional feed repressurization step was thus inserted.

(2) The production of oxygen started while the pressure of column A was still on the rise. But the feed rate dropped quickly as the column pressure increased. For the particular run indicated in this figure, 3.7 L (STP) of product gas were collected from column A during the first 20 s of the production step, of which 3 liters were used to purge column C. During the same time, 7 liters of feed gas entered the column, most of which was adsorbed.

(3) For column A, the feed and production step continues for another 30 s. All the time a purge to column C was split from the product stream. In addition, column B, whose pressure was about 223 kPa at this moment, had been connected to the product stream for 20 s. A sudden drop of column A pressure was immediately noticed upon the connection. Accompanying the pressure drop was an increase of the feed gas flow rate, and a slight decrease of the purge and product rate. Such a pressure decrease during the feed and production step was actually not desirable. We have, by mistake, forgotten to put a pressure regulator between the product and the backfill lines. This mistake was noticed only after the conclusion of the experimental work, and was too late for any correction.

(4) After the feed and production step, column A was pressure equalized with the newly purged column C. This brought the pressure of column A down to 253 kPa, and a new cycle initiated again.

Before we present the overall performance of our system, let us take a look at the expected performance of a linear system. The relevant equilibrium constants can be estimated from the average slopes of the pure component isotherms in the pressure range employed. According to the previously identified isotherm, values of 3.99 and 6.79 has been obtained for oxygen and nitrogen, respectively. For a cycle with product backfill and pressure equalization steps, the optimum purge and feed conditions as well as the corresponding oxygen recovery have been calculated (Chiang, 1994). The results are given in Table 1.

If the isotherm were linear, 5.96 STP liters of pure oxygen (95.7% if adjusted for the argon in feed) can be produced per cycle per bed at a recovery of 44%. It will be operated under a purge rate of 11.8 STP liter per cycle per bed (LPCPB) and a feed rate of 64.1 LPCPB. The suggested optimum purge accounts for only 44% of what a complete purge condition will require.

Compared to the actual performances of our experimental system summarized in Table 1, the predictions from linear local equilibrium theory is simply too optimistic. The best product purity we have obtained was only 98.9% (O_2+Ar). This was at a recovery of merely 15%. Recovery higher than 30% was never reached without a great sacrifice on product purity.

There are at least three fundamental deficiencies to the linear local equilibrium (LLE) theory we have applied to arrive at a recovery of 44%. First of all, a local equilibrium theory always predicts the formation of a concentration shock under favorable conditions. In the real process, both column dispersion and finite adsorption rate prevent the formation of a true shock. At best, a constant pattern profile may be formed instead. Secondly, the previous theory is confined to conditions where the product is pure light component. While in reality pure product gas is seldom achieved unless the production cut is extremely small. The third, and the most important shortcoming, is the linear isotherm assumption.

The first two imperfections can only be analyzed in conjunction with the simulation results published elsewhere (Chou and Huang, 1993). But the difference between linear and nonlinear isotherms can be understood directly with the well-known results on column dynamics. During the purge step, a desorption process in essence, a self-broadening concentration wave will be formed for both linear and nonlinear isotherms. The spreading of wave front, however, will be much accelerated due to the isotherm nonlinearity. Compared in Figure 5 are the gas-phase concentration profiles after the purge step. The oxygen concentration of the purge effluent raises very early if the isotherm was nonlinear. Less purge should thus be used

Table 1. Overall Performance: Four Bed System vs. Prediction from Linear Local Equilibrium Theory

	Feed	Purge	Produce	Product Purity		Recovery	Purge to Feed Volume Ratio
				O_2	O_2+Ar		
	LPCPB	LPCPB	LPCPB	%	%	%	
	From Linear Local Equilibrium Theory						
	64.05	11.8	5.96	95.7	100	44	0.78
Run No.	Experimental Results						
1	47.5	7.70	1.54	94.5	98.9	15	0.65
2	47.05	7.38	1.88	91.4	95.7	17	0.63
3	45.71	5.37	1.72	91.3	95.5	16	0.47
4	47.94	7.19	2.55	89.1	93.2	23	0.6
5	43.2	4.20	2.07	88.6	92.7	20	0.39
6	51.44	9.45	3.27	87.1	91.2	26	0.73
7	46.18	6.46	3.23	84.8	88.8	28	0.56
8	50.2	8.44	3.98	83.8	87.7	32	0.67
9	42.97	3.34	3.05	82.4	86.3	28	0.31
10	47.78	6.30	6.32	63.3	66.2	40	0.53
11	46.16	4.30	8.05	51.5	53.9	43	0.37
12	43.56	2.00	7.53	48.7	50.9	40	0.18

LPCPB: STP Liters Per Cycle Per Bed

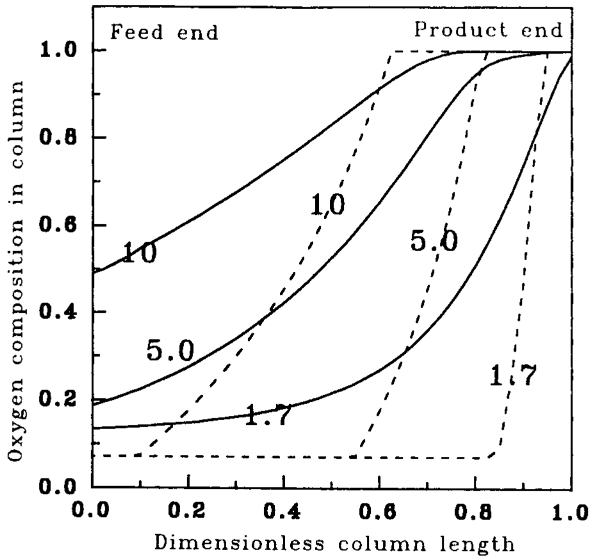

Figure 5. Composition profiles in column after different amounts of purge.

Numbers indicated are in STP liter per cycle per bed. Calculated based on measured isotherm; ----- are based on linear isotherm.

in such a case to cut down the oxygen lost for higher recovery. This is what has been demonstrated in Table 1. All the experimental runs have been operated with a purge less than the prediction of LLE theory. In fact, the experimental data indicate that there was simply not enough product gas to accomplish the optimum purge specified by the LLE theory.

If less purge has been given, more nitrogen will then be left in the column. As a consequence, less nitrogen can be adsorbed in the following feed and production step: the feed may penetrate deeper into the column. It is known that concentration shock is easier to form in a nonlinear system due to the self-sharpening effect. However, the nitrogen tail left in the column under insufficient purge precludes the completion of a simple shock until very close to the product end. As a result, a deep shock front will be formed in a nonlinear system under insufficient purge and limited feed. This conjuncture can be substantiated by the experimental data. According to the linear theory, none of the experimental conditions should be able to complete a simple shock. Yet it is clear that simple shock (or a constant pattern front) was indeed formed for run 1.

If the concentration shock has been pushed very close to the product end, there may not be enough oxygen in the column for the following pressure equalization step. Nitrogen will then break into the lower pressure column which has been pressurized. In such a case, the product purity cannot be maintained in the next cycle. This is likely to be what happened for runs 3 to 8. For runs 9 to 11, it is obvious that breakthrough of nitrogen occurred even during the feed and production step.

The experimental findings can now be summarized as follows:

(1) A highly dispersed desorption wave must have been generated during the purge step. Recovery was greatly cut down due to the breakthrough of light component at the feed end. The extent of purge could not be operated at the condition suggested by the linear local equilibrium theory if a positive recovery was to be maintained.

(2) Due to insufficient purge, feed penetrated deep into the column even for a moderate amount of feed. Only a small cut of purity light component could be extracted during the feed step. The purity of this limited product was further limited by the column dispersion and the adsorption rate.

(3) If too much product gas was extracted, the shock front would be driven too close to the product end. There would not be enough light component to feed the low-pressure column in the equalization step. Nitrogen would leak into the low-pressure column. This in turn deteriorated the already insufficiently purged condition. The overall effect would be a reduction of the product purity.

(4) If the feed was extended past the point of breakthrough, the average product purity would drop very quickly. The gas transferred during the following pressure equalization step might contain even less light component than the feed gas. In such case, the pressure equalization step became counterproductive. For a process intended for high purity product, the pressure equalization should be removed.

Although the LLE theory provided a vastly overoptimistic prediction, the simplicity of its calculation is still very attractive for a first estimation of the operation conditions. Furthermore, the purge-to-feed volume ratio, which is an indicator commonly used in the PSA process, might have been predicted correctly. As indicated in Table 1, a purge-to-feed volume ratio of 0.78 was suggested by the linear theory. Comparison of experiment data (run 2 with 3, 4 with 5 and 8 with 9) suggested that conditions closer to this value seemed to give a better recovery under higher product purity. In addition, simulation works based on a nonisothermal, nonlinear, dispersed local equilibrium model (Chou and Huang, 1993) suggested that one should be able to obtain about 5 LPCPB product gas with 95% oxygen plus argon at a recovery of 37%, if 12.5 LPCPB of purge were used. This corresponded to a purge-to-feed volume ratio of 0.8. Therefore, without more accurate information, the purge-to-feed ratio predicted from linear theory may be a good starting point for choosing the operation conditions even for a nonlinear system.

The overall performance of our system can be compared to the results of Kayser and Knaebel (1986), who have obtained better than 99.7% oxygen plus argon at a recovery of 10%. It was produced with a dual column four step cycle including product backfill. The pressure ratio was 6.4 and the adsorbent used was similar to ours. They suggested that no product will be obtained had the pressure ratio been lower than 4.5. We have demonstrated that production of 98.2% oxygen plus argon at a recovery of 15% is possible even under a pressure ratio of just 4.3. This was accomplished with the addition of pressure equalization step and a reduced purge. Recovery gain due to the savings on purge have been examined experimentally by Matz and Knaebel (1988) with a single column process. However, they have operated their system at a rather high-pressure ratio, where the earnings on purge are not as critical. On the other hand, the effect of pressure equalization step has been demonstrated only with the pattern literatures.

The productivity obtained by Kayser and Knaebel was roughly 0.1 SLPM per kilogram of zeolite, at a purity of 99.7% (O_2 + Ar). Our four column process produced 0.09 SLPM/kg zeolite at 98.2% purity (O_2 + Ar), and 0.2 SLPM/kg if at 91% purity. For the production of 90% purity oxygen, Sircar and Zondio (1977) reported a value of 0.3 SLPM/kg zeolite at a

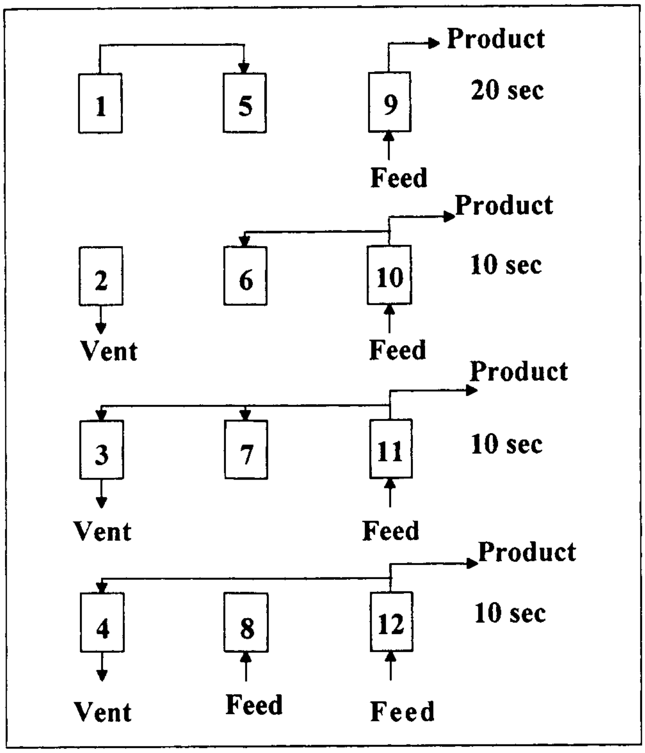

Figure 6. Process schedule of proposed three column PSA system.

Each column goes through same sequences as four column process studied, except shorter blowdown and purge steps.

recovery as high as 40%. Still higher was the rapid pressure swing adsorption (RPSA) systems on which a value of 1 SLPM/kg at a recovery of 38% have been reported (Jones and Keller, 1981). Our experimental system is therefore far from optimized compared to the industrial practices.

Room for productivity improvement does exist in our process. For example, a 70 s of nonactive period per cycle has been observed on each bed during the blowdown and purge steps. At least a 30% increase in the productivity can be expected if this period were eliminated by a different process schedule, such as that illustrated in Figure 6. These type of three column designs are indeed employed in many commercial processes. Secondly, the sudden pressure drop during the feed and production step in our system came from a design mistake. Had it been corrected, the oxygen recovery could also be improved. Furthermore, we have noticed that the fluctuation of the feed rate was rather significant. This would be unfavorable since both the dispersion and pressure drop increased with velocity. A steady feed should be another point to work on.

It is however not our purpose to optimize the experimental system for the best productivity. In fact, it takes a tremendous effort to optimize a PSA process experimentally if one does not have some guidelines to follow. Even if such guidelines exist, optimizing a PSA process on an accurate simulator would first be more economic than doing it directly on a PSA system. Simulators capable of doing such work have been developed in many places and most are proprietary (Doong and LaCava, 1991; Ng et al., 1991; Chen et al., 1992; Chiang 1992). The experimental work reported here, in addition to demonstrating the suitability and deficiency of a linear local equilibrium theory, will also be useful for the verification of such simulators.

Conclusion

Dynamic data as well as the overall performance of a four column PSA system were presented. This process was operated under a pressure ratio of only 4.3 to amplify the need for pressure equalization step and reduced purge condition. The results were compared to the optimized operation conditions predicted from the linear local equilibrium theory. The spreading of the purge wave was identified to be the main cause of the lower recovery obtained in a nonlinear system. The feed and purge conditions predicted from the LLE theory was too high for a nonlinear system, if very high purity product was intended. However, the best purge to feed volume ratio suggested seemed to be valid.

The system and operation conditions presented were far from optimized. In fact, mistakes have been made which become apparent after studying the data. Suggestions on improvements have been made accordingly.

Acknowledgment

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Erratum

In the article titled "Modeling the Role of Bacteria in Leaching of Low-Grade Ores" by K. I. Batarseh and A. H. Stiller (October, 1994, p. 1741), the sentence immediately before the section on **Results and Discussion** should read: "Thus, the assumption imposed appears to be '*apposite*' for the present case."