Adsorption 10: 267–275, 2004

Pressure-Swing Adsorption Using Layered Adsorbent Beds with Different Adsorption Properties: II—Experimental Investigation

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Received September 18, 2003; Revised August 3, 2004; Accepted September 23, 2004

Abstract. An experimental study was conducted on a layered-bed pressure-vacuum-swing adsorption, PVSA, process with adsorbents that differ in their adsorption properties. An oxygen, O_2, PVSA process was employed as an example for investigating how the process performance is affected by bed-layering configuration under different operating conditions for specific purge, product purity, and cycle feature. For two adsorbents with similar nitrogen-to-oxygen, N_2/O_2, selectivity but different N_2 and O_2 capacities, placing the high-capacity adsorbent at the product end and the low-capacity adsorbent at the feed end of the adsorption bed results in a better performance than in the case of reversing the layer positions of those adsorbents. The benefit of placing the adsorbent with higher capacity at the product end becomes more significant at high O_2 product-purity levels. The experimental data obtained in this investigation agree well with simulation results reported earlier.

Keywords: PVSA processes, performance simulation, layered beds, positioning of adsorbents, adsorption capacity, adsorption selectivity

1. Introduction

Layered-bed VSA/PSA processes with adsorbents that have different adsorption properties can be used to improve significantly the process performance by utilizing the adsorbents' inherent potentials at their *maxima*, and to reduce overall product cost. Ideal bed-layer configurations of adsorbents depend on specific adsorption properties, feed-mixture composition, and product-purity requirements. It is common to place more than one adsorbent in a given adsorber when the PSA feed gas contains multiple components, e.g., the separation of H_2-CO-CO_2-H_2O mixtures by adsorption using activated carbons and/or zeolite adsorbents (Park et al., 1998, 2000). For processes such as O_2 PVSA, in which only one component, viz., N_2, is adsorbed selectively, it is known that layering NaX- and LiX-zeolite adsorbents could result in a decrease of overall product cost (Toussaint, 1993; Watson et al., 1996).

Baksh and Notaro (1998) described a N_2-PSA process that utilizes two layers of O_2 equilibrium-selective adsorbents, of which the high-capacity/high-selectivity adsorbent was placed near the product end of the

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bed. Fray (1982 and 1983) developed a simple model for layered beds in cycling-zone adsorption processes. Pigorini and LeVan (1997) studied the effects of adsorption-isotherm shape and adsorbent capacity on the performance of layered-bed PSA, by considering a feed that consisted of one single adsorbing component in inert gas.

In the first part of this work (Lü et al., 2003), a simulation study was conducted on layered-bed O_2-PVSA processes with adsorbents that differ in adsorption capacity and selectivity, to investigate the effect of adsorption properties on the process performance. The study showed that placing a high-capacity adsorbent at the product end and a low-capacity adsorbent at the feed end of the adsorption bed gives a better performance than the case of reversing the layering sequence for the two adsorbents. In the second part of this study, experimental work of O_2-PVSA processes was carried out using layered beds packed with adsorbent materials that have different adsorption capacities.

2. Experimental

2.1. Adsorbents

The key O_2-PVSA adsorbent used in this study is a faujasite-zeolite material in shape of beads with about 13 wt.-% attapulgite-type binder. Its XRD crystallinity as compared to an internal powder standard is *c*. 87%, and the Si/Al ratio for the beaded product amounts to 1.28. The zeolitic phase contains only alkaline-metal cations as monovalent lattice-charge compensating cations in its supercages. The average diameter of the beads is *c*. 2.0 mm, and the bulk density of the beads amounts to *c*. 0.68 g/cm^3. This material is referred to as Type 2 adsorbent throughout this paper.

Instead of preparing another adsorbent with different adsorption capacity but the same N_2/O_2 selectivity, the Type 2 adsorbent was mixed with alumina beads in a volume ratio of 1:1. The mixed material is referred to as Type 1 adsorbent. The average diameter of the alumina beads is *c*. 3 mm, and its packing density amounts to *c*. 0.82 g/cm^3. Prior to experiments, both adsorbents, Type 1 and Type 2, were activated at 580°C for 7 hours in an atmosphere comprised of 95% N_2 and 5% O_2.

Oxygen-PVSA experiments have been conducted in four different types of bed-layering configuration. The four layering configurations and related adsorbent-loading data are given in Table 1, in which 'Type 2 on Type 1' (2 on 1), for example, refers to layering Type 2 adsorbent on top of Type 1 adsorbent. The feed is introduced into the column bed from its bottom, and the product is withdrawn from its top.

2.2. Oxygen-PVSA Apparatus

The experimental setup is a two-bed O_2-PVSA bench-scale unit as shown schematically in Fig. 1. The adsorbent-bed columns are made of stainless steel with water-cooling jackets. The beds are of cylindrical shape, and their length and inner diameter amount to 86 cm and 7 cm, respectively. Adjusting the temperature of the water pumped through the bed jackets controls the bed temperature. The total volume of water that circulates through the thermostat and the two bed-jackets is about 15 l. The cooling-water temperature measured at the jacket outlet on top of Bed A is kept at 25°C. Dry air is used as the feed, which is introduced from the bottom of the bed. The temperature of the feed air is ambient, 23–25°C.

There are two product tanks in the O_2-PVSA bench-scale unit. The product tanks are connected in series, and the volume of each amounts to 7.5 liters. The air-feed flow meter is kept fully open, and the airflow rate depends on the opening of the air-pressure regulator, which is adjusted to maintain the set peak pressure. Adjusting the opening of the bleed valve of the vacuum line controls the vacuum level defined as bed pressure at the end of the evacuation step.

Table 1. Bed-layering configuration and adsorbent loading data.

Layering configuration	Type 2 on Type 1 (2 on 1)	Type 1 on Type 2 (1 on 2)	Volume percentage	Type 2 on alumina (2 on A)	Volume percentage	Alumina on Type 2 (A on 2)	Volume percentage
Top end	Alumina	Alumina	14.5	(empty)	14.5	(empty)	14.5
Top layer	Type 2	Type 1	35.5	Type 2	35.5	Alumina	14.5
Bottom layer	Type 1	Type 2	35.5	Type 2	35.5	Type 2	35.5
Bottom end	Alumina	Alumina	14.5	Alumina	14.5	Type 2	35.5

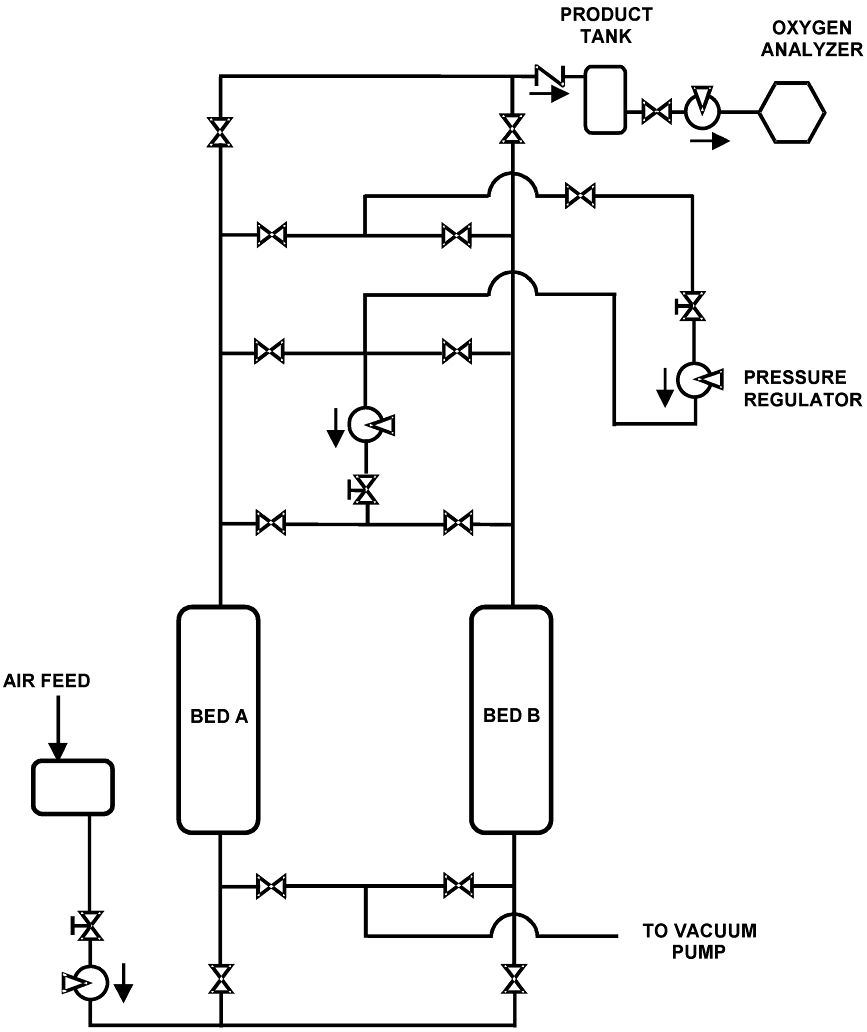

Figure 1. Scheme of the O_2-PVSA apparatus.

2.3. *Oxygen-PVSA Process Cycles*

The current study employs a two-bed O_2-PVSA cycle with equalization and purge. As shown in Table 2, the steps of the basic cycle in its first half are as follows:

Table 2. Cycle features of the O_2-PVSA process.

Actions in Bed A	Actions in Bed B	Basic cycle	Cycle without equalization
Half-cycle time, s		29	29
		Step length, s	Step length, s
Pressurization	Evacuation	10	10
Production	Purge-evacuation	12	19
Equalization (−)	Equalization (+)	5	0
Evacuation -equalization (−)	Equalization (+)	2	0

(1) Pressurization/Evacuation: Air is introduced into the bed from the bottom of Bed A, and O_2 production starts from the top when the pressure in Bed A exceeds that in the product tank, while Bed B is regenerated by connecting its bottom to a vacuum pump.

(2) Production/Purge-evacuation: Production in Bed A continues while Bed B is being purged with product from the top and evacuated from the bottom.

(3) Equalization (−)/Equalization (+): Feed supply to Bed A is terminated, and Bed A is depressurized by transferring the O_2-rich gas from the top to the top of Bed B.

(4) Evacuation-equalization (−)/Equalization (+): Equalization from Bed A to Bed B is continued except Bed A is connected to a vacuum pump from the bottom through the feed end.

Steps 5 through 8 of the second half cycle are the same as step 1 through step 4, except that the actions are exchanged for Bed A and Bed B.

While the half-cycle time in all experiments was kept at 29 s, two different cycle variations were run for purposes of comparison. One of them is the basic cycle with equalization and purge steps, cf., Table 2. In the other cycle variation, the production/purge-step time is extended from 10 s to 19 s, and the equalization steps are eliminated.

3. Results and Discussion

The O_2 yield is defined as the percentage of O_2 in the feed stream, which is captured in the product. The specific product (SP) is the volume of O_2 produced per unit of time and per unit of adsorbent; it is expressed by normal cubic meter of O_2 per cubic meter of adsorbent per hour. Specific purge is defined as the average purge-flow rate (normal cubic meter per hour) per unit volume of adsorbent (cubic meter) in one cycle.

The peak pressure refers to the bed pressure at the end of the production step (Step 2 of Bed A or Step 6 of Bed B). The vacuum pressure is the bed pressure at the end of the evacuation step (Step 1 of Bed B or Step 5 of Bed A).

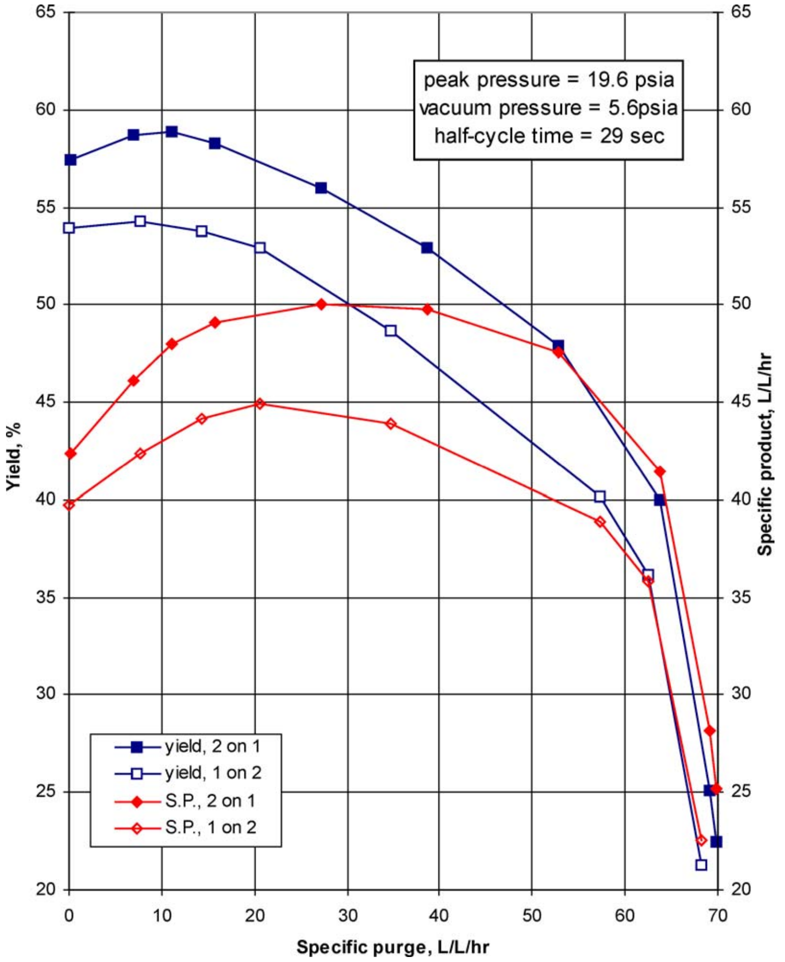

Figure 2. Performance of layered-bed O_2-PVSA by two adsorbents with different capacities, as functions of specific purge.

3.1. Layered-Bed O_2-PVSA by Adsorbents with Different Adsorption Capacities

To confirm our previous simulation-study results, the adsorption beds were packed with Type 1 and Type 2 adsorbents that have different adsorption capacities. As alumina has no significant preferential adsorption capacity for either N_2 or O_2, it was chosen as a dilution material, which was physically mixed with the faujasite-zeolite beads, Type 2 adsorbent, that adsorbs selectively N_2. The resulting adsorbent, Type 1 adsorbent, has about half of the adsorption capacity of the Type 2 adsorbent, and its selectivity is similar to that of the Type 2 adsorbent.

Figure 2 shows the O_2 yields and specific products in two layering configurations with the basic cycle as a function of specific purge. The peak pressure and low pressure were kept at 1.38 at and 0.39 at, respectively, and the oxygen-product purity was 90%. As shown in Fig. 2, if the high-capacity adsorbent, Type 2 adsorbent, is placed on the top (the product end) of the adsorption bed, the O_2 yield and specific product are by *c*. 10% higher than those parameters observed if the low-capacity adsorbent, Type 1 adsorbent, is packed on the top. The difference in performance between the two packing configurations becomes smaller if the specific purge is increased.

As discussed in the related simulation study, cf., Lü et al. (2003), from the viewpoint of N_2-loading difference in a cycle, the region near the product end at the top of the bed has a higher loading difference, and thus it is more important than the bottom region of the bed with respect to separation efficiency. Placing the adsorbent with higher capacity at the top makes best use of the adsorbents compared. The experimental result reported here confirms that it is beneficial to pack the

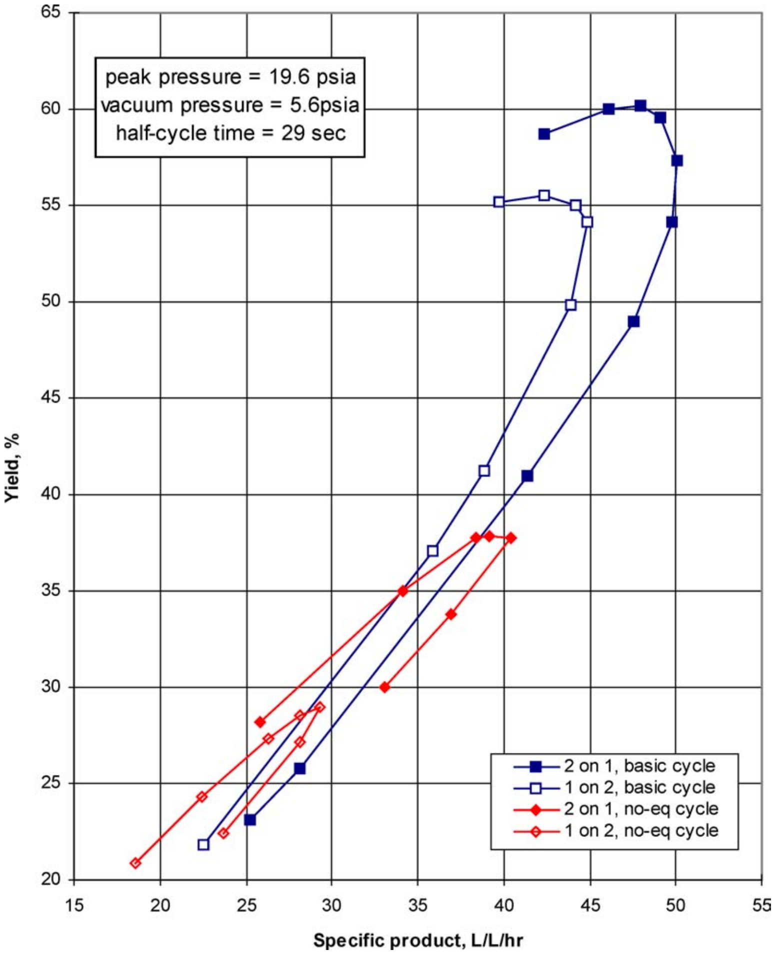

Figure 3. Performance of layered-bed O_2-PVSA with basic cycle and no-equalization cycle (faujasite and faujasite-alumina mixture).

high-capacity adsorbent in the region near the product end.

3.2. Effect of Cycle Feature on Layered-Bed O_2 PVSA by Adsorbents with Different Adsorption Capacities

Oxygen-PVSA performances of the two layering configurations for both the basic cycle and the cycle without equalization (no-equalization cycle) are shown in Fig. 3, where the O_2 yield is plotted vs. the specific product with specific purge as third parameter. Again, the O_2-product purity was kept at 90%. If the equalization steps were eliminated, both yield and specific product dropped significantly. Yet, it should be noted that, unlike what was observed in our simulation study (Lü et al., 2003), a greater difference in performance is observed between the two packing configurations for the no-equalization cycle. This is due to the fact that in the experimental study the N_2 capacities of Type I and Type II adsorbents are about half of those used in the simulation study. Generally speaking, PVSA performance will suffer more for the adsorbent with lower N_2 capacity at high O_2 product-purity levels.

As it can be seen from Fig. 3, the difference in performance between the basic and the no-equalization cycles is significant. In this case, the process performance becomes more sensitive towards specifics of layering configurations and bed voidage. If the capacities of the two adsorbents, Types I and II adsorbents, are relatively low, layering the low-capacity adsorbent, Type I adsorbent, on the top results in a higher loss of performance, as the low-capacity adsorbent on the top

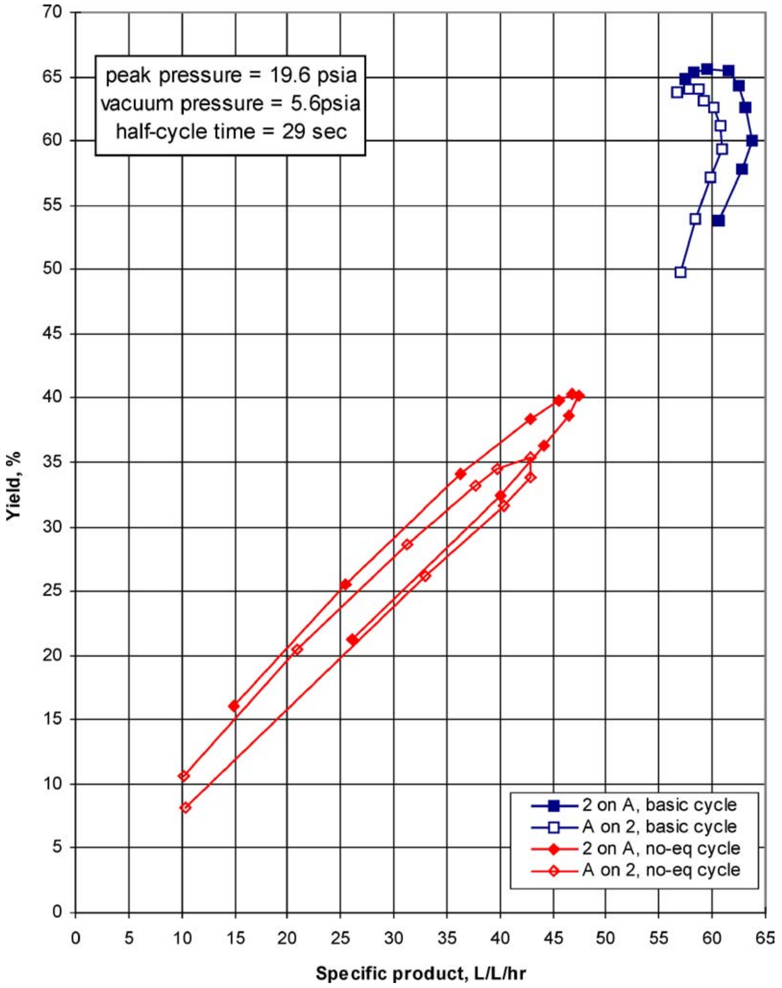

Figure 4. Performance of layered-bed O_2-PVSA with basic cycle and no-equalization cycle (alumina and faujasite).

creates more void space there. For this reason, N_2 capacity and product purity should be taken into consideration if performances of layered-bed PVSA processes are compared. The effect of O_2-product purity will be discussed later.

As the low-capacity adsorbent, Type 1 adsorbent, used in this study is a 1:1 mixture of Adsorbent 2 and alumina, its adsorption capacity is half of that of the Type 2 adsorbent. To further look into PVSA-performance differences between the two types of cycles and two layering configurations, the layered adsorbent beds were packed with Type 2 adsorbent and alumina, which is the extreme case for the Type I adsorbent since its capacity would be reduced to nearly zero. Essentially, the alumina layer becomes a layer of void space with its volume equaling to the total alumina volume minus the actual solid alumina volume. The volume percentages of the Type 1 adsorbent and of alumina are given in Table 1. As indicated in the plot oxygen yield vs. specific product, cf., Fig. 4, a similar trend was observed in the layered beds packed with Type 2 adsorbent and alumina. As the adsorption capacity of alumina is neglegible compared to that of the faujasite adsorbent, what is shown is practically the effect of void location, viz., due to placing the void at the top (A on 2, in Fig. 4) and at the bottom (2 on A, in Fig. 4).

3.3. *Effect of O_2-Product Purity on Layered-Bed O_2 PVSA by Adsorbents with Different Adsorption Capacities*

The effect of O_2-product purity on the performance of layered-bed O_2 PVSA is shown in Figs. 5 and 6. The

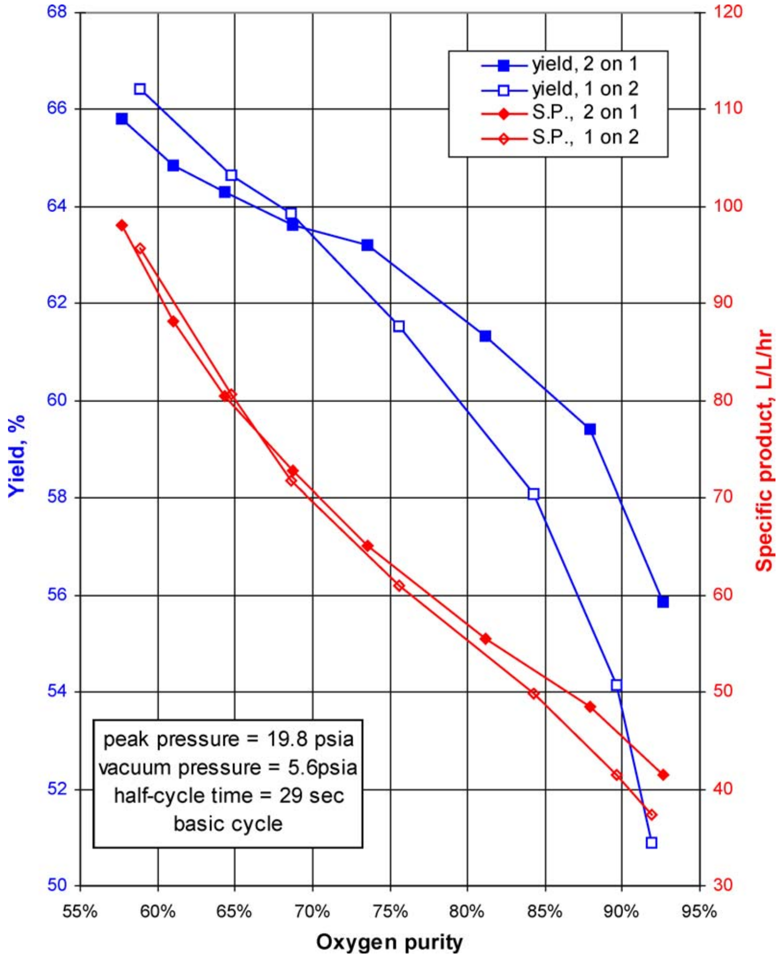

Figure 5. Effect of product purity on the performance of layered-bed O_2-PVSA with basic cycle.

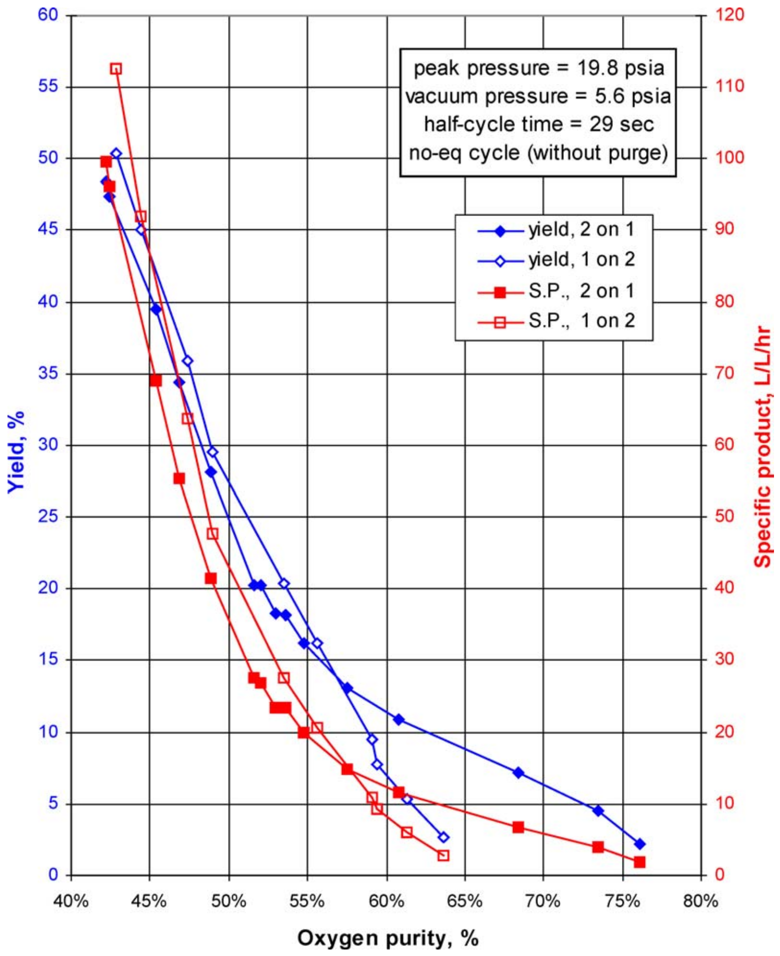

Figure 6. Effect of product purity on the performance of layered-bed O_2-PVSA with no-equalization cycle.

ratio of purge-flow rate to product-flow rate for the basic cycle was kept constant. In doing so, a similar pressure profile during the purge step was realized.

As the product purity increases, it becomes more advantageous to place the high-capacity adsorbent on the top of the layered bed. For low product purity, layering the high-capacity adsorbent at the product end does not result in any improvement of process performance. It seems that, if the product purity is below a threshold level, one should place the low-capacity adsorbent at the product end. These results suggest the importance of selecting the right layering configuration in accordance with product-purity levels chosen.

4. Conclusions

An experimental study was carried out on a layered-bed pressure-vacuum-swing adsorption, PVSA, process with adsorbents that have different adsorption capacities. Based on the results of an O_2 PVSA process with two adsorbents that have similar N_2-to-O_2 selectivity but different N_2 and O_2 capacities, placing the high-capacity adsorbent at the product end and the low-capacity adsorbent at the feed end of the adsorption bed results in a better performance than for the case of reversing layering of these adsorbents. The benefit of placing the high-capacity adsorbent at the product end becomes more significant at high O_2-product purity levels. The experimental observations confirm the results of a related process-simulation study, which were reported previously.

Acknowledgments

The authors thank BOC Gases for the permission to publish this paper.

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