

PROCESS FOR PRODUCING OXYGEN-RICH GAS

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a process for producing an oxygen-rich gas. More specifically, the present invention relates to a process for producing an oxygen-rich gas comprising separating nitrogen from a gas mixture containing oxygen and nitrogen, such as air, by pressure swing adsorption using an adsorbent capable of the selective adsorption of nitrogen.

2. Description of the Related Art

It is known to produce an oxygen-rich gas from an oxygen-nitrogen gas mixture, such as air, by separating nitrogen therefrom through pressure swing adsorption utilizing the selective adsorptivity to nitrogen of an adsorbent such as synthetic or natural zeolites (for example, Japanese Examined Patent Publication (Kokoku) Nos. 51-40549 and 51-40550, and Japanese Unexamined Patent Publication (Kokai) Nos. 56-63804 and 58-84020). In accordance with this method, the oxygen-rich gas is produced by introducing an oxygen-nitrogen gas mixture into a plurality of adsorption columns each containing a bed of adsorbent, thus causing the nitrogen to be adsorbed on each adsorbent bed and thereby removing it from the gas mixture, and providing the product gas by discharging a gas enriched in the slightly adsorbable oxygen from the adsorption columns. In each adsorption column, the depressurizing, purging, and evacuating steps for regenerating the adsorbent in the adsorption column and the above-mentioned adsorption and separation step are successively alternated.

The conventional method for producing the oxygen-rich gas by pressure swing adsorption, however, involves disadvantages in that the efficiency of separation and recovery of oxygen is unsatisfactory and the energy consumption per volume of the product gas formed

is high. Therefore, there is still room for improvement in the conventional method.

SUMMARY OF THE INVENTION

A primary object of the present invention is to provide a process for producing an oxygen-rich gas by pressure swing adsorption wherein the efficiency of separation and recovery of oxygen is higher than that of the conventional method and the energy consumption per volume of the product gas formed is low.

In accordance with the present invention, there is provided a process for producing an oxygen-rich gas wherein three adsorption columns each filled with a bed of adsorbent capable of selective adsorption of nitrogen are used, and a gas mixture containing oxygen and nitrogen is made to flow through the adsorption columns to cause the nitrogen contained in the gas mixture to be adsorbed on the adsorbent.

The process according to the present invention comprises carrying out successively the following steps, in a first adsorption column:

(1) introducing a pressurized gas mixture to the first adsorption column at its feed end and simultaneously introducing an oxygen-rich gas derived from a second adsorption column to the first column at its product end, so as to equalize the pressures between the first and second columns;

(2) introducing the pressurized gas mixture to the first column at its feed end so as to cause the nitrogen contained in the gas mixture to be selectively adsorbed on the adsorbent contained therein and simultaneously discharging an oxygen-rich gas from the product end thereof, so as to increase the pressure in the first column;

(3) stopping the introduction of the pressurized gas mixture and introducing a part of the oxygen-rich gas discharged from the product end of the first column to a third adsorption column at its product

end, so as to equalize the pressures between the first and third columns;

(4) discharging an oxygen-rich gas from the product end of the first column to introduce the gas to the second column at its product end, thereby purging the interior of the second column;

(5) evacuating the first column at its feed end to depressurize it; and

(6) introducing an oxygen-rich gas derived from the third column to the first column at its product end while the first column is evacuated at its feed end, so as to purge the interior of the first column; wherein, during the operation of the first column, the above-mentioned process cycle is carried out in each of the second and third adsorption columns while the phase is being changed.

In accordance with another feature of the present invention, there is provided a variation of the above-mentioned process. This process comprises successively carrying out the following steps, in a first adsorption column:

(1) introducing a pressurized gas mixture to the first column at its feed end and simultaneously introducing an oxygen-rich gas derived from a second adsorption column to the first column at its product end, so as to equalize the pressures between the first and second columns;

(2) introducing the pressurized gas mixture to the first column at its feed end to cause the nitrogen contained in the gas mixture to be selectively adsorbed on the adsorbent contained in the first column, and simultaneously discharging an oxygen-rich gas from the product end of the first column, so as to increase the pressure in the first column;

(3) stopping the introduction of the pressurized gas mixture and introducing a part of the oxygen-rich gas being discharged from the product end

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of the first column to a third adsorption column at its product end, so as to equalize the pressures between the first and third columns;

(4) discharging the oxygen-rich gas from the product end of the first column to introduce the gas to the second column at its product end, thereby purging the interior of the second column;

(5) further discharging the oxygen-rich gas from the product end of the first column to introduce the gas to the second column at its feed or product end, thereby equalizing the pressures between the first and second columns;

(6) evacuating the first column at its feed end to depressurize it;

(7) introducing an oxygen-rich gas derived from the third column to the first column at its product end while the first column is evacuated at its feed end, so as to purge the interior of the first column; and

(8) introducing the oxygen-rich gas from the third column to the first column at its feed or product end, so as to equalize the pressures between the first and third columns,

wherein, during the operation of the first column, the above-mentioned process cycle is carried out in each of the second and third adsorption columns while the phase is being changed.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a system diagram illustrating the process of the present invention; Figs. 2 and 3 are diagrammatic views illustrating the sequence of operation of the steps according to the process of the present invention; Fig. 4 is a system diagram illustrating a variation of the process of the present invention; and Figs. 5 and 6 are diagrammatic views illustrating the sequence of operation of the steps according to the variation of the process of the present invention of Fig. 4.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention contemplates producing an oxygen-rich gas by separating nitrogen from a gas mixture containing mainly oxygen and nitrogen, such as air, through the use of pressure swing adsorption. That is, a pressurized gas mixture is fed into an adsorption column filled with an adsorbent such as synthetic or natural zeolites, where the nitrogen is allowed to be adsorbed on the adsorbent, and a gas enriched in oxygen is discharged as the product gas from the adsorption column. Then, suction is applied at the feed end of the adsorption column filled with the nitrogen-adsorbed adsorbent to bring the pressure in the column to a vacuum pressure, thereby desorbing the nitrogen adsorbed on the adsorbent.

According to the present invention, three adsorption columns filled with an adsorbent are used, and a pressure equalization step is carried out between any two of the three columns. That is, one column which is being supplied with a pressurized gas mixture at its feed end is communicated with the other column which has been already pressurized to the predetermined pressure by the gas mixture and is delivering the product gas, i.e., an oxygen-rich gas, at the product ends of these columns, so as to equalize the pressures between these two columns. Furthermore, while one adsorption column is being evacuated to a vacuum pressure at the feed end thereof to desorb the nitrogen adsorbed on the adsorbent, an oxygen-rich gas is introduced at the product end thereof from the other adsorption column to purge the interior of the former column. This operation is referred to as a purging step. Thus the product oxygen-rich gas is continuously discharged without interruption during the process cycle.

The pressure equalization is effective for keeping the adsorption front of nitrogen in one column short through the supply of an oxygen-rich gas from the

product end of the other column, thereby increasing the oxygen concentration of the resultant gas. The purging wherein the oxygen-rich gas from the product end of the other column is passed through one column during the vacuum desorption of nitrogen in the column so as to increase the desorption effect and simultaneously for shortening the time for vacuum desorption so as to reduce the power cost.

In accordance with the process of the present invention having the above-mentioned constitution, an oxygen-rich gas having a high oxygen concentration of 90% to 93% or more can be produced at a remarkably high efficiency. Furthermore, the operation of the vacuum pump is always effectively carried out during the cycle.

The process of the present invention will now be explained in detail with reference to Fig. 1. It is to be understood that the following operations are only an example to which the present invention is by no means limited.

In operation, an oxygen-rich gas is continuously discharged while the six steps described hereinafter are successively repeated. The operating time of each step can be optionally controlled by a timer.

Step 1

Valve 1A is opened and pressurized air 20, from which water mist has been removed in a mist separator 21, is introduced to column A at the bottom, i.e., the feed end, thereof. Simultaneously, valves 2A and 2C are opened and column A is connected through a manifold 18 to column C, in which the pressure is being increased to a predetermined pressure by pressurized air, at the preceding stage (step 6), and is already discharging the product oxygen-rich gas through the opened valve 12C. The oxygen-rich gas is introduced to column A at the product end thereof from the top, i.e., the product end, of column C, so as to equalize the pressures between columns A and C (pressure equalization). During this

period, the flow rate of the oxygen-rich gas derived from column C is controlled by valves 6A and 6C. During the above operation, valve 4B is opened and column B is depressurized while being evacuated by the vacuum pump 19.

Step 2

Valves 2A and 12C are closed and, simultaneously, valve 12A is opened. The product gas is discharged from column A. While delivering the product gas, column A is supplied with pressurized air through valve 1A, so that the pressure in column A is progressively increased. On the other hand, valve 3B is opened and an oxygen-rich gas is introduced at the product end of column B from the product end of column C. The introduced oxygen-rich gas flows countercurrently through column B to purge the interior thereof, while it is being discharged out of the system by the vacuum pump 19 (purging). Column C is further depressurized while delivering the oxygen-rich gas to column B. The feed rate of the oxygen-rich gas from column C is controlled by valve 17.

Step 3

Valves 1A, 2C, and 4B are closed and valve 1B is newly opened. Pressurized air is introduced to column B at its feed end. Simultaneously, valves 2A and 2B are opened and an oxygen-rich gas is introduced to column B at its product end from the product end of column A, to equalize the pressures between columns A and B (pressure equalization). During this period, the flow rate of the oxygen-rich gas derived from column A is controlled by valves 6A and 6B. On the other hand, valve 3B is closed and valve 4C is opened, and column C is depressurized while being evacuated by the vacuum pump 19.

Step 4

Valves 2B and 12A are closed and, simultaneously, valve 12B is opened, and the product gas is discharged from column B. While delivering the product gas, column B is supplied with pressurized air through

valve 1B, so that the pressure in column B is progressively increased. On the other hand, valve 3C is opened and an oxygen-rich gas is introduced at the product end of column C from the product end of column A. The introduced oxygen-rich gas flows countercurrently through column C to purge the interior thereof, while it is being discharged out of the system by the vacuum pump 19 (purging). Column A is further depressurized while delivering the oxygen-rich gas to column C. The feed rate of the oxygen-rich gas from column A is controlled by valve 17.

Step 5

Valves 1B, 2A and 4C are closed and valve 1C is newly opened, and pressurized air is introduced to column C at its feed end. Simultaneously, valves 2B and 2C are opened and an oxygen-rich gas is introduced to column C at its product end from the product end of column B, to equalize the pressures between columns B and C (pressure equalization). During this period, the flow rate of the oxygen-rich gas derived from column B is controlled by valves 6B and 6C. On the other hand, valve 3C is closed and valve 4A is opened, and column A is depressurized while being evacuated by the vacuum pump 19.

Step 6

Valves 2C and 12B are closed and simultaneously valve 12C is opened, and the product gas is discharged from column C. While delivering the product gas, column C is supplied with pressurized air through valve 1C, so that the pressure in column C is progressively increased. On the other hand, valve 3A is opened and an oxygen-rich gas is introduced at the product end of column A from the product end of column B. The introduced oxygen-rich gas flows countercurrently through column A to purge the interior thereof, while it is being discharged out of the system by the vacuum pump 19 (purging). Column B is further depressurized while

delivering the oxygen-rich gas to column A. The feed rate of the oxygen-rich gas from column B is controlled by valve 17.

In Fig. 1, reference numeral 22 represents the effluent of the product oxygen-rich gas, and reference numeral 23 represents the effluent of the separated nitrogen.

As will be understood from the above description, the above-mentioned six steps are repeatedly carried out in such a manner that steps 1 and 2 form one unit and the phase of each column is varied in column-to-column relationship. Figures 2 and 3 are diagrammatic views illustrating the basic two steps (steps 1 and 2).

Next, a variation of the process of the present invention mentioned above will be explained. In this variation, three adsorption columns filled with an adsorbent are used, and a primary pressure equalization step is carried out between any two of the three columns. That is, one column which is being supplied with a pressurized gas mixture at its feed end is connected to the other column which has been already pressurized to a predetermined pressure by the gas mixture and is delivering the product gas, i.e., an oxygen-rich gas, at the product ends of these columns, so as to equalize the pressures between these two columns. Furthermore, while one adsorption column is being evacuated to a vacuum pressure at the feed end thereof to desorb the nitrogen adsorbed on the adsorbent, an oxygen-rich gas is introduced at the product end thereof from the other adsorption column to purge the interior of the former column. This operation is referred to as a purging step. Also, after one adsorption column is evacuated to a vacuum pressure to desorb the nitrogen adsorbed on the adsorbent, thereby regenerating the adsorbent, the feed end or product end of this column is connected to the product end of the other adsorption column, so as to equalize the pressures between these two columns.

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This operation is referred to as a secondary pressure equalization step. The purging step and the secondary pressure equalization step are carried out in appropriate combination with the primary pressure equalization step.

- 5 Thus the product oxygen-rich gas is continuously discharged without interruption during the process cycle.

The primary pressure equalization is effective for keeping the adsorption front of nitrogen in the one column short through the supply of an oxygen-rich gas from the product end of the other column, thereby increasing the oxygen concentration of the resultant gas. The purging wherein the oxygen-rich gas from the product end of the other column is passed through one column during the vacuum desorption of nitrogen in the other column, is effective for depressurizing the partial pressure of nitrogen in the column so as to increase the desorption effect and, simultaneously, for shortening the time for vacuum desorption so as to reduce the power cost. Furthermore, the secondary pressure equalization is effective for further recovering a slight amount of the oxygen-rich gas remaining in the column from which the oxygen-rich gas has been almost completely discharged out, and for eliminating the temperature nonuniformity in the column generated due to adsorption and desorption.

This process will now be explained in detail with reference to Fig. 4.

- In this operation, an oxygen-rich gas is continuously discharged while the nine steps described hereinafter are successively repeated. The operating time of each step can be optionally controlled by a timer.

Step 1

- Valve 1A is opened and pressurized air 20, from which water mist has been removed in a mist separator 21 is introduced to column A at the bottom, i.e., the feed end, thereof. Simultaneously, valves 2A and 2C are

opened and column A is communicated through manifold 18 with column C which is being pressurized to the predetermined pressure by pressurized air at the preceding stage (step 9) and is already discharging the product oxygen-rich gas through the opened valve 12C. The oxygen-rich gas is introduced to column A at the product end thereof from the top, i.e., the product end, of column C, to equalize the pressures between columns A and C (primary pressure equalization). During this period, the flow rate of the oxygen-rich gas derived from column C is controlled by valves 6A and 6C. On the other hand, valves 4B and 5 are opened and column B is depressurized while being evacuated by the vacuum pump 19.

Step 2

Valves 2A and 12C are closed and valve 12A is concurrently opened, and the product gas is discharged from column A. While delivering the product gas, column A is supplied with pressurized air through valve 1A, so that the pressure in column A is progressively increased. On the other hand, valve 9 is opened and an oxygen-rich gas is introduced at the product end of column B from the product end of column C through valve 3B which has been already opened at step 9. The introduced oxygen-rich gas flows countercurrently through column B to purge the interior thereof, while it is being discharged out of the system by the vacuum pump 19 (purging). Column C is further depressurized while delivering the oxygen-rich gas to column B. The feed rate of the oxygen-rich gas from column C is controlled by valve 17.

Step 3

While delivering the product gas, column A is supplied with pressurized air and is further pressurized until a predetermined highest pressure is reached. Valves 2C, 3B, 5, and 9 are closed and valves 3C and 10 are newly opened. The oxygen-rich gas remaining in the

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upper portion of column C is introduced at the feed end of column B, to equalize the pressures between columns B and C (secondary pressure equalization). The feed rate of the oxygen-rich gas from column C is controlled by valve 11.

The oxygen-rich gas from column C may be introduced at the product end of column B by maintaining valve 3B opened, in place of opening valve 10.

Step 4

Valves 1A and 4B are closed and valve 1B is newly opened, and pressurized air is introduced to column B at its feed end. Valves 2A and 2B are concurrently opened and an oxygen-rich gas is introduced to column B at its product end from the product end of column A, to equalize the pressures between columns A and B (primary pressure equalization). During this period, the flow rate of the oxygen-rich gas derived from column A is controlled by valves 6A and 6B. In the other hand, valve 10 is closed and valves 4C and 5 are opened, and column C is depressurized while being evacuated by the vacuum pump 19.

Step 5

Valves 2B and 12A are closed and valve 12B is concurrently opened, and the product gas is discharged from column B. While delivering the product gas, column B is supplied with pressurized air through valve 1B, so that the pressure in column B is progressively increased. On the other hand, valve 9 is opened and an oxygen-rich gas is introduced at the product end of column C from the product end of column A. The introduced oxygen-rich gas flows counter-currently through column C to purge the interior thereof, while it is being discharged out of the system by the vacuum pump 19 (purging). Column A is further depressurized while delivering the oxygen-rich gas to column C. The feed rate of the oxygen-rich gas from column A is controlled by valve 17.

Step 6

While delivering the product gas, column B is supplied with pressurized air and is further pressurized until a predetermined highest pressure is reached.

- 5 Valves 2A, 3C, 5, and 9 are closed and valves 3A and 10 are newly opened. The oxygen-rich gas remaining in the upper portion of column A is introduced at the feed end of column C, to equalize the pressures between columns A and C (secondary pressure equalization). The feed rate
10 of the oxygen-rich gas from column A is controlled by valve 11.

Step 7

- Valves 1B and 4C are closed and valve 1C is newly opened, and pressurized air is introduced to column C at
15 its feed end. Valves 2B and 2C are concurrently opened and an oxygen-rich gas is introduced to column C at its product end from the product end of column B, to equalize the pressures between columns B and C (primary pressure equalization). During this period, the flow rate of the
20 oxygen-rich gas derived from column B is controlled by valves 6B and 6C. On the other hand, valve 10 is closed and valves 4A and 5 are opened, and column A is depressurized while being evacuated by the vacuum pump 19.

Step 8

- 25 Valves 2C and 12B are closed and valve 12C is concurrently opened, and the product gas is discharged from column C. While delivering the product gas, column C is supplied with pressurized air through valve 1C, so that the pressure in column C is progres-
30 sively increased. On the other hand, valve 9 is opened and an oxygen-rich gas is introduced at the product end of column A from the product end of column B. The introduced oxygen-rich gas flows counter-currently through column A to purge the interior thereof, while
35 it is being discharged out of the system by the vacuum pump 19 (purging). Column B is further depressurized while delivering the oxygen-rich gas to column A.

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The feed rate of the oxygen-rich gas from column B is controlled by valve 17.

Step 9

While delivering the product gas, column C is
5 supplied with pressurized air and is further pressurized until a predetermined highest pressure is reached. Valves 2B, 3A, 5, and 9 are closed and valves 3B and 10 are newly opened. The oxygen-rich gas remaining in the upper portion of column B is introduced at the feed end of column A, to equalize the pressures between columns A and B (secondary pressure equalization). The feed rate of the oxygen-rich gas derived from column B is controlled by valve 11.

In Fig. 4, reference numeral 22 represents the
15 effluent of the product oxygen-rich gas, and reference numeral 23 represents the effluent of the separated nitrogen.

The secondary pressure equalization in steps 3, 6 and 9 may be carried out by introducing the oxygen-rich
20 gas derived from columns C, A and B at the product ends of columns B, C and A, respectively, as illustrated in step 3 of Fig. 5C.

As will be understood from the above description, the above-mentioned nine steps are repeatedly carried
25 out in such a manner that steps 1 through 3 form one unit and the phase of each column is varied in a column-to-column relationship. Figures 5 and 6 are diagramatic views illustrating these three basic steps (steps 1 through 3).

30 As described hereabove, in accordance with the process of the present invention, the pressure in each column is constantly varied and is not maintained at any certain value even for a moment. This is realized by effecting a mutual exchange of oxygen between the
35 columns, which is effective for reducing the amount of oxygen discharged out of the system as much as possible, to increase the oxygen yield. Furthermore, even if the

highest pressure of adsorption and the vacuum pressure to be reached at the pressurizing side and the vacuum side, respectively, are kept to a high level range, the incessant variation of the pressure in each column

- 5 reduces the average pressure and the average vacuum pressure as much as possible, to decrease the power cost.

Purging is an indispensable operation for stably obtaining the product gas having a high concentration of oxygen. In controlling the highest purging amount, it is not particularly necessary to know its absolute amount. This control may be effected on the basis of a reduction in the pressure in the column delivery purging oxygen (this pressure reduction is represented as purge Δp). The purge Δp is usually in the range of from 0.1 to 1.0 kg/cm². The purging amount is determined in such a manner that the highest oxygen concentration and recovery per maximum pressure of adsorption, maximum vacuum pressure to be reached, and optimum cycle time can be attained.

The factors for evaluation of the efficiency of the process for producing an oxygen-rich gas by pressure swing adsorption include the oxygen concentration of the resultant oxygen-rich gas and the recovery thereof. As another factor, there may be mentioned a ratio of the amount of an adsorbent, e.g., molecular sieve, to the amount of oxygen produced, per unit time. In the following examples, this factor is referred to as a bead size factor (BSF) and the unit is expressed in terms of (kg·MS)/(t·O₂/d) [(kg molecular sieve)/(ton·100% O₂/day)]. The BSF is a factor relating to the process and the quality of the molecular sieve. However, where molecular sieves of the same quality are used, the BSF serves as a factor for evaluating the process. As is apparent from the above-mentioned unit, it is deemed that the lower the BSF, the better the process.

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The present invention will be explained in more detail by the following examples.

Example 1

Three adsorption columns each having an inner diameter of 81 mm and a height of 2.5 m were filled, respectively, with 9.14 kg of an adsorbent consisting of Molecular Sieve 13X beads each having a size of 8 to 12 mesh. The operational conditions comprised a highest adsorption pressure of $0.5 \text{ kg/cm}^2\text{G}$, a vacuum pressure to be achieved of 200 Torr, and a purge Δp of 0.2 kg/cm^2 . The cycle time was changed per experimental number. Continuous operation was carried out according to the above-mentioned process (the process shown in Figs. 2 and 3, i.e., the six-step cycle). The results obtained are shown in Table 1.

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Table 1

Experiment No.	Feed rate of air (Nm ³ /h)	Flow rate (Nm ³ /kgMS·h)	Amount of enriched oxygen (Nm ³ /h)	Oxygen concentration (%)	Oxygen yield (%)	BSF (kg·MS)/(t·O ₂ /d)
341-1	4.20	0.153	0.441	95.3	47.8	1898
342-1	3.31	0.120	0.364	92.9	48.8	2360
353-3	2.56	0.093	0.286	95.2	50.9	2932

Note:

Cycle time, Experiment No. 341-1 Primary pressure equalization 15 seconds, purging 35 seconds
 342-1 Primary pressure equalization 15 seconds, purging 45 seconds
 353-3 Primary pressure equalization 20 seconds, purging 60 seconds

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Example 2

The same operations as those described in Example 1 were repeated, except that the operational conditions were changed to: a highest adsorption pressure of 0.5 kg/cm²G, a vacuum pressure to be achieved of 360 Torr, and a purge Δp of 0.27 kg/cm². The cycle time was changed per experimental number. Continuous operation was effected. The results obtained are shown in Table 2.

Table 2

Experiment No.	Feed rate of air (Nm ³ /h)	Flow rate (Nm ³ /kgMS·h)	Amount of enriched oxygen (Nm ³ /h)	Oxygen concentration (%)	Oxygen yield (%)	BSF (kg·MS)/(t·O ₂ /d)
372-3	3.43	0.125	0.344	95.0	45.5	2440
383-3	4.30	0.156	0.468	93.1	48.4	1833
384-1	3.50	0.127	0.379	94.2	48.7	2235

Note:

Cycle time, Experiment No. 372-3 Primary pressure equalization 15 seconds, purging 26 seconds
 383-3 Primary pressure equalization 15 seconds, purging 18 seconds
 384-1 Primary pressure equalization 15 seconds, purging 25 seconds

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Comparative Example 1

The same operations as those described in Example 1 were repeated, except that the operational conditions were changed to: a highest adsorption pressure of 5 0.5 kg/cm²G, a vacuum pressure to be achieved of 200 Torr, and a purge Δp of 0 kg/cm² (no purging carried out). The cycle time was changed per experimental number. Continuous operation was effected. The results obtained are shown in Table 3.

10 In these experiments, the oxygen concentration was less than 85% and the oxygen yield was 1/2 times or less that of Example 1.

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Table 3

Experiment No.	Feed rate of air (Nm^3/h)	Flow rate ($\text{Nm}^3/\text{kgMS}\cdot\text{h}$)	Amount of enriched oxygen (Nm^3/h)	Oxygen concentration (%)	Oxygen yield (%)	BSF ($\text{kg}\cdot\text{MS}) / (\text{t}\cdot\text{O}_2/\text{d})$
387-2	2.99	0.109	0.350	62.8	35.0	3625
388-3	2.00	0.073	0.141	84.3	28.4	6768

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Comparative Example 2

The same operations as those described in Example 1 were repeated, except that the depressurizing was effected by blow down due to the self-pressure (i.e., the vacuum pump was kept stopped) instead of carrying out the vacuum suction. The cycle time was changed per experimental number. In this manner, continuous operation was effected. The results obtained are shown in Table 4.

- 5
- 10 The resultant product gas exhibited a remarkably low concentration of oxygen and inferior yield.

Table 4

Experiment No.	Feed rate of air (Nm^3/h)	Flow rate ($\text{Nm}^3/\text{kgMS}\cdot\text{h}$)	Amount of enriched oxygen (Nm^3/h)	Oxygen concentration (%)	Oxygen yield (%)	BSF ($\text{kg}\cdot\text{MS}) / (\text{t}\cdot\text{O}_2/\text{d})$
396-2	3.07	0.112	0.80	28.3	35.2	3555
396-5	2.16	0.079	0.349	36.8	28.4	6220
398-2	1.32	0.048	0.137	39.9	19.8	14,653

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Example 3

- Three adsorption columns each having an inner diameter of 81 mm and a height of 2.5 m were filled, respectively, with 9.1 kg of an adsorbent consisting
- 5 of Molecular Sieve 13X beads each having a size of 8 to 12 mesh. The operational conditions comprised a highest adsorption pressure of $2.0 \text{ kg/cm}^2\text{G}$, a vacuum pressure to be achieved of 160 Torr, and a purge Δp of 0.2 kg/cm^2 . The cycle time was changed per experimental number.
- 10 Continuous operation was carried out according to the above-mentioned process (the processes shown in Figs. 5 and 6, i.e., the nine step-cycle). The results obtained are shown in Table 5.

Table 5

Experiment No.	Feed rate of air (Nm^3/h)	Flow rate ($\text{Nm}^3/\text{kgMS}\cdot\text{h}$)	Amount of enriched oxygen (Nm^3/h)	Oxygen concentration (%)	Oxygen yield (%)	BSF ($\text{kg}\cdot\text{MS})/(\text{t}\cdot\text{O}_2/\text{d})$
301-1	3.33	0.121	0.432	95.0	58.9	1944
312-1	2.15	0.078	0.302	94.3	63.3	2804
313-2	4.33	0.157	0.548	93.0	56.2	1566

Note:

Cycle time, Experiment No. 301-1 Primary pressure equalization 13 seconds, purging 40 seconds, secondary pressure equalization 20 seconds

312-1 Primary pressure equalization 13 seconds, purging 80 seconds, secondary pressure equalization 25 seconds

313-2 Primary pressure equalization 11 seconds, purging 17 seconds, secondary pressure equalization 25 seconds

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Comparative Example 3

The same operations as those described in Example 3 were repeated, except that the purging step was omitted from the above-mentioned process (i.e., valve 9 was kept
5 closed). The results obtained are shown in Table 6.

According to this process, it was impossible to increase the oxygen concentration to not less than 91.8%, and the oxygen yield was remarkably low as compared with Example 3.

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Table 6

Experiment No.	Feed rate of air (Nm ³ /h)	Flow rate (Nm ³ /kgMS·h)	Amount of enriched oxygen (Nm ³ /h)	Oxygen concentration (%)	Oxygen yield (%)	BSF (kg·MS) / (t·O ₂ /d)
231-4	2.33	0.085	0.174	91.3	32.5	5042
242-5	3.26	0.118	0.106	91.7	14.2	8211

Note:

Cycle time, Experiment No. 231-4 Primary pressure equalization 13 seconds, vacuum desorption time 70 seconds, secondary pressure equalization 13 seconds

242-5 Primary pressure equalization 13 seconds, vacuum desorption time 25 seconds, secondary pressure equalization 20 seconds



Comparative Example 4

The same operations as those described in Example 3 were repeated, except that the purging was carried out at normal pressure without effecting vacuum suction (i.e., the vacuum pump was kept stopped), and the purge Δp was changed from 0.2 kg/cm^2 to 0.8 kg/cm^2 . The cycle time was the same as described in Example 3. A purge Δp of 0.8 kg/cm^2 indicated the optimum value obtained as a result of the preliminary experiments. The results obtained are shown in Table 7.

According to this process, it was possible to increase the oxygen concentration to 94.0 but the oxygen yield was 1/2 times or less that of Example 3.

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Table 7

Experiment No.	Feed rate of air (Nm^3/h)	Flow rate ($\text{Nm}^3/\text{kgMS}\cdot\text{h}$)	Amount of enriched oxygen (Nm^3/h)	Oxygen concentration (%)	Oxygen yield (%)	BSF ($\text{kg}\cdot\text{MS}) / (\text{t}\cdot\text{O}_2/\text{d})$
152-1	3.10	0.113	0.174	94.0	25.2	4885
163-3	3.39	0.124	0.250	92.6	32.6	3445
162-1	4.77	0.174	0.247	90.0	22.2	3586

(409)

Example 4

The same operations as those described in Example 3 were repeated, except that the highest adsorption pressure and the vacuum pressure to be achieved were changed to 0.5 kg/cm²G and 200 Torr, respectively. The cycle time was changed per experimental number. Continuous operation was effected. The results obtained are shown in Table 8.

Table 8

Experiment No.	Feed rate of air (Nm ³ /h)	Flow rate (Nm ³ /kgMS·h)	Amount of enriched oxygen (Nm ³ /h)	Oxygen concentration (%)	Oxygen yield (%)	BSF (kg·MS) / (t·O ₂ /d)
261-2	2.23	0.081	0.245	95.0	49.8	3426
262-1	2.71	0.098	0.303	94.7	50.5	2784

(51)

Comparative Example 5

The same operations as those described in Example 3 were repeated, except that the highest adsorption pressure was changed to $0.5 \text{ kg/cm}^2\text{G}$, and the desorption
5 was carried out by blow down due to the self-pressure without effecting vacuum suction, and no purging was carried out. The cycle time was changed per experimental number. Continuous operation was effected.

The oxygen concentration could not be increased to
10 more than 28%, and the oxygen yield was remarkably decreased.

The results obtained are shown in Table 9.

Table 9

Experiment No.	Feed rate of air (Nm ³ /h)	Flow rate (Nm ³ /kgMS·h)	Amount of enriched oxygen (Nm ³ /h)	Oxygen concentration (%)	Oxygen yield (%)	BSF (kg·MS) / (t·O ₂ /d)
298-2	1.34	0.049	0.29	24.0	24.8	11,480
299-3	0.57	0.021	0.05	27.5	11.5	58,120

CLAIMS:

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1. A process for producing an oxygen-rich gas wherein three adsorption columns each filled with a bed of adsorbent capable of selective adsorption of nitrogen are used, and a gas mixture containing oxygen and nitrogen is
- 5 flowed through the adsorption columns to cause the nitrogen contained in the gas mixture to be adsorbed on the adsorbent, thereby removing the nitrogen from the gas mixture, comprising carrying out successively the following steps, in a first adsorption column:
- 10 (1) introducing a pressurized gas mixture to the first adsorption column at its feed end and simultaneously introducing an oxygen-rich gas derived from a second adsorption column to the first column at its product end, to equalize the pressures between the first
- 15 and second columns;
- (2) introducing the pressurized gas mixture to the first column at its feed end to cause the nitrogen contained in the gas mixture to be selectively adsorbed on the adsorbent contained therein and
- 20 simultaneously discharging an oxygen-rich gas from the product end thereof, to increase the pressure in the first column;
- (3) stopping the introduction of the pressurized gas mixture and introducing a part of the
- 25 oxygen-rich gas being discharged from the product end of the first column to a third adsorption column at its product end, to equalize the pressures between the first and third columns;
- (4) discharging the oxygen-rich gas from
- 30 the product end of the first column to introduce the gas to the second column at its product end, thereby purging the interior of the second column;
- (5) evacuating the first column at its feed end to depressurize it; and
- 35 (6) introducing an oxygen-rich gas derived from the third column to the first column at its



(A) product end while the first column is evacuated at its feed end, to purge the interior of the first column; wherein, during the operation of the first column, the above-mentioned process cycle is carried out in each of
5 the second and third adsorption columns while the phase is being changed.

2. A process as claimed in claim 1, wherein the gas mixture is air.

10 3. A process as claimed in claim 1, wherein the absorbent is selected from synthetic zeolites and natural zeolites.

4. A process as claimed in claim 1, wherein an oxygen-rich gas having an oxygen concentration of at least 90% is produced.

15 5. A process as claimed in claim 1, which further comprises the steps of: after step (4), further discharging the oxygen-rich gas from the product end of the first column to introduce the gas to the second column at its feed end or product end, thereby equalizing the
20 pressures between the first and second columns; and, after step (6), introducing the oxygen-rich gas derived from the third column to the first column at its feed end or product end, to equalize the pressures between the first and third columns.

25 6. A process as claimed in claim 5, wherein the gas mixture is air.

7. A process as claimed in claim 5, wherein the absorbent is selected from synthetic zeolites and natural zeolites.

30 8. A process as claimed in claim 5, wherein an oxygen-rich gas having an oxygen concentration of at least 90% is produced.

9. A process for producing an oxygen-rich gas according to Claim 1 substantially as herein described.

