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Oxygen and Nitrogen Enrichment in Air by Cycling Zone Adsorption

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Summary

Enrichment of N_2 and O_2 in air has been obtained by cycling zone adsorption in the double zone standing wave mode. The solid adsorbent is molecular sieve type 5 A. The maximum separation factor obtained in these experiments is 10.6 for oxygen.

INTRODUCTION

The principles of cycling zone adsorption were first described in 1969 (*1*) as a result of the developments in parametric pumping. Cycling zone adsorption has the advantage of being a continuous separation process in contrast to the batchtype process of parametric pumping.

The reported separations by cycling zone adsorption (*1*) were all applied to ideally diluted liquid or gas mixtures. The diluted mixture is separated by the cycling adsorption–desorption process which yields two phases: one containing more and one containing less of the minor component. In the present communication, cycling zone adsorption is demonstrated for a system of practical importance, viz., N_2 and O_2 in air.

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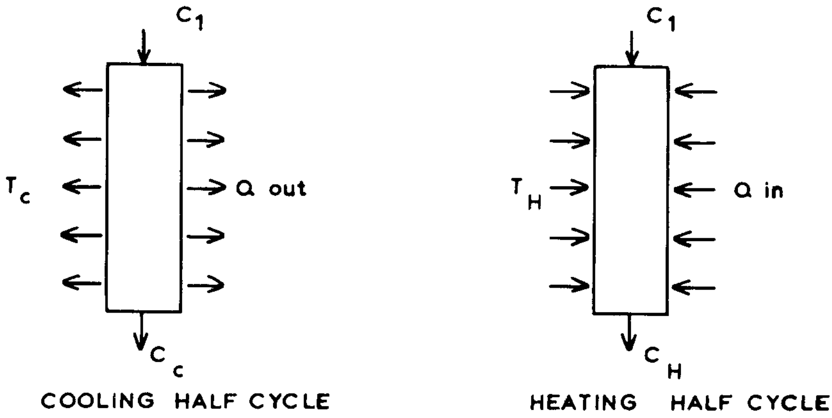

FIG. 1. Single zone standing wave operation of cycling zone adsorption.

BASIC ASPECTS

Figure 1 shows the principle of cycling zone adsorption applied to air separation. Air, having an oxygen concentration C_1, is fed into a bed of molecular sieve particles. The column is held at a constant low temperature T_C. The molecular sieve preferentially adsorbs nitrogen from the entering gas, so the effluent gas is richer is oxygen (C_C).

After some time the adsorbent is saturated and the oxygen concentration in the effluent gas decreases to C_1. Then the temperature is raised to T_H. The molecular sieve desorbs more nitrogen than oxygen. The oxygen concentration in the product gas decreases further to C_H. When the desorption is complete the oxygen concentration increases to C_1, the temperature is lowered to T_C, and the process is repeated. The process has an optimum cycle time analogous to the optimum flow-termperature cycle time in parametric pumping. The situation shown in Fig. 1 is called the single zone standing wave operation of cycling zone adsorption (*1*).

At constant pressure the changes in adsorption cause flow fluctuations in the effluent gas stream. These fluctuations limit the selection of the rate of inflow when a single zone system is used. Therefore, two zones in series were used, Zone I at T_C, Zone II at T_H, and conversely. With this design the outflow fluctuations are partially cancelled.

APPARATUS

The two zones consist of jacketed glass columns (15 × 0.8 cm i.d.) packed with 45–60 mesh Linde molecular sieve type 5 A (Fig. 2). The molecular sieve was prevoiusly activated by heating during 24 hr at 400°C in a stream of nitrogen. The feed of dry air (from a pressure

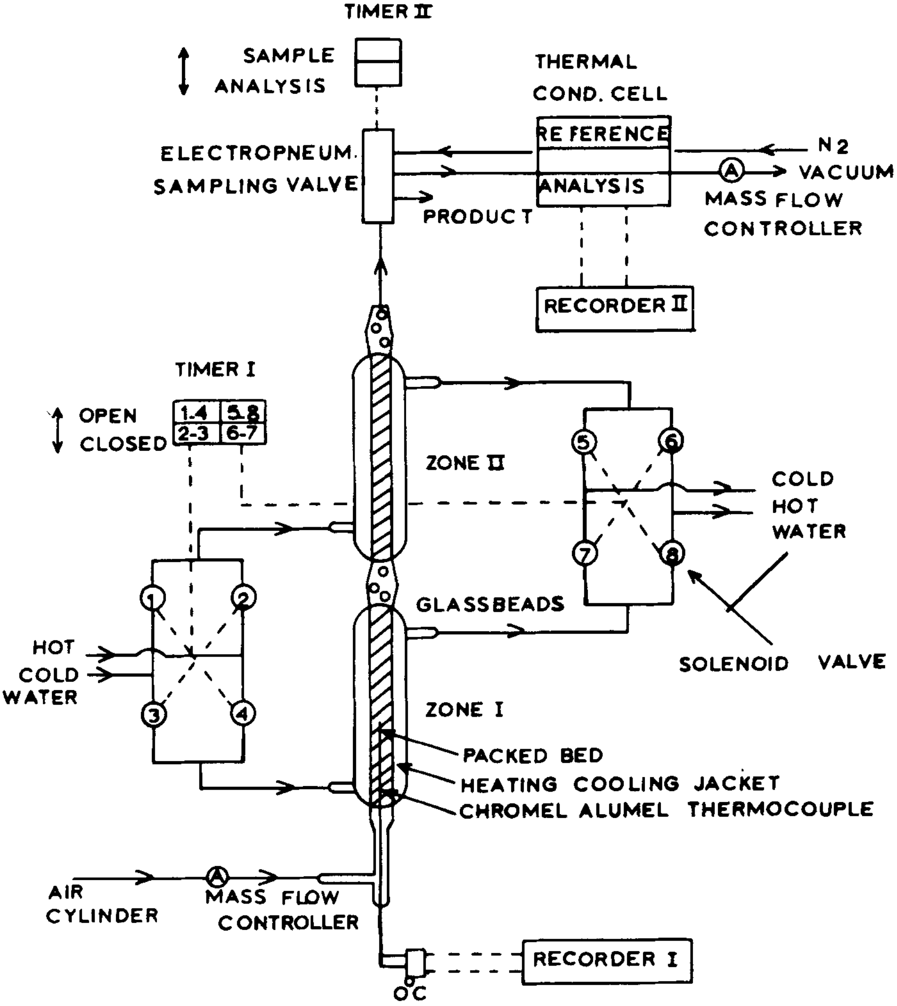

FIG. 2. Double zone cycling zone adsorption apparatus.

cylinder) is kept constant by a mass flow controller (Brooks, Type 8743). A dual cam recycling timer (Industrial Timer Corp.) is used in connection to two sets of 4 solenoid valves for the alternating heating and cooling of each zone. An electropneumatic sampling valve (Loenco, Type L-208-6 with actuator) connected to a second recycling timer takes samples of the effluent gas of the second zone at regular time intervals. The amount of oxygen in the sample is measured gas chromatographically with the aid of a thermistor thermal conductivity cell (Gow, Mac, Type 10–470). The temperature in the first zone is monitored by a chromel–alumel thermocouple.

EXPERIMENTAL PROCEDURE AND RESULTS

The feed of air is kept constant at 10 ml/minute (20°C, 1 atm). The temperature of the zones is cycled at 1 cycle in 6 min (Fig. 3). The

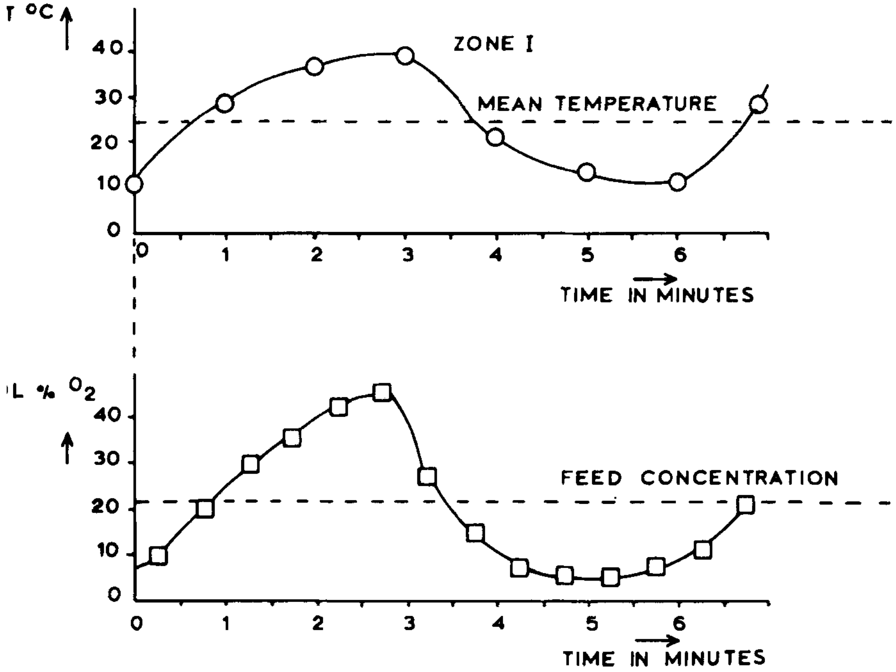

FIG. 3. Temperature cycle in Zone I and oxygen concentration cycle in effluent gas.

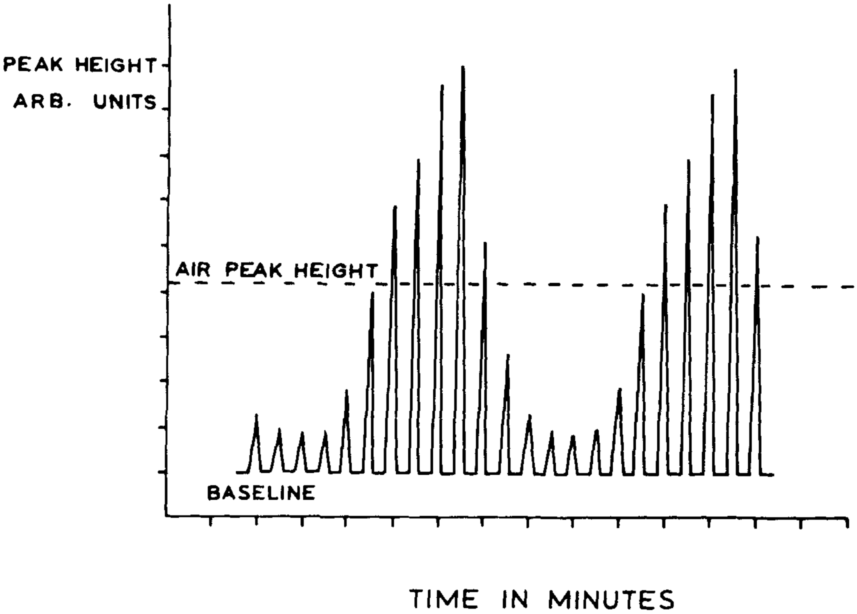

FIG. 4. Chromatographic oxygen peaks from product samples.

minimum temperature T_C is 10.2°C, the maximum temperature T_H 38.7°C. For the analysis, constant volume samples of about 0.20 ml (20°C, 1 atm) of effluent gas are taken every 30 sec. The recorded peak height is observed to be directly proportional to the oxygen percentage of the sample, as it should be theoretically. The height of an air peak is taken at 21 vol % oxygen as a standard. Figure 4 shows the trace of Recorder II during the separation. The obtained separation factor S, defined as the ratio of the maximum oxygen concentration to the minimum oxygen concentration, is 10.6 under these conditions.

The percentage of oxygen during one temperature cycle is shown in Fig. 3.

DISCUSSION

The differences between this separation of a mixture of two components having comparable concentrations and the separation of very diluted mixtures studied before (*1*) are twofold. The adsorption of gas during the cooling half cycle and the desorption during the heating half cycle cause pronounced flow fluctuations in the effluent gas. A double zone separator was used to reduce these effects. Neverethless, the fluctuations were too high for continuous analysis of the product flow, since the thermal conductivity cell is sensitive to flow fluctuations.

The second problem originates from the fact that the nitrogen and oxygen adsorption isotherms on molecular sieve 5 A are not linear at high concentrations. Thus the characteristic velocity of propagation of concentration waves through the zones is not only a function of the temperature and flow velocity, as is the case in ideally diluted systems (*1*, *2*), but also of the concentration itself. This situation is analogous to the description of retention times in gas–solid chromatography. With linear adsorption isotherms the retention time is independent of the sample size, whereas curved isothersm cause a dependency of the retention time on the sample size (*3*). The influence of these differences on the optimum cycle time is under study.

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