

Research Article

Preparation of Carbon Molecular Sieves in Reducing Atmosphere for Selective O₂/N₂ Separation

Wi-Gwon Jang , Kwang-Guk Kim , Chol-Ryong Choe , Su-Il Kim* 

Institute of Chemical Engineering, Kim Chaek University of Technology, Pyongyang, DPR Korea

Abstract

Carbon Molecular Sieve (CMS) is an ultramicroporous carbonaceous adsorbent, which is widely used in the field of gas separation, especially for air nitrogen separation, as distinguished from activated carbon due to the uniformity of micropore distribution. The separation of nitrogen from air by pressure swing adsorption (PSA method) using carbon molecular sieve in a medium and small scale nitrogen production process is superior to the conventional air cold separation method. Carbon molecular sieves can be prepared from nuts, wood, polyethylene, polyimide, etc., but we used apricot seed husks as carbon substrates. We analyzed the effect of various factors on the properties of carbon molecular sieves prepared from a matrix of apricot seed husks with extremely low ash content of the matrix and high micropore volume to adsorb oxygen, and on this basis, we established a rational preparation process to prepare CMS for nitrogen gas separation. The properties of the carbon molecular sieves prepared under the optimum preparation conditions were 7.2mg/g oxygen equilibrium adsorption, 6.3mg/g adsorption for 1min and 32 selectivity, which were very good for nitrogen separation. The prepared carbon molecular sieve is highly selective and can be used as a very efficient adsorbent for nitrogen separation in air as well as a support for a highly efficient molecular sieve catalyst.

Keywords

Carbon Molecular Sieve (CMS), Equilibrium Adsorption Capacity, Separation

1. Introduction

CMS is a non-polar carbonaceous adsorbent with a kind of nanoscale ultra-micropores, consisting of fine carbon crystals and amorphous carbon, and has a highly developed pore structure and special surface properties. Because of this special microporous structure, CMS has a higher selectivity than activated carbon in adsorbing the desired component from a gas mixture [1]. The adsorption of gases can be broadly classified as chemical adsorption and physical adsorption. Chemical adsorption method has been widely used in the industry because of low cost, large adsorption, and high se-

lectivity [2]. Whereas, due to the highly corrosive to equipment, high energy consumption and large efficiency loss, chemical adsorption method still needs further improvement [3]. In contrast, solid adsorption exhibits favorable advantages including the absence of corrosivity, the low volatility and the low energy penalty [4]. Separation techniques based on the adsorption kinetics of gas molecules in porous materials such as CMS are used in many fields of the chemical industry and are more practical than conventional separation techniques such as cryogenic distillation or cryogenic adsorption [5].

*Corresponding author: si.Kim409@star-co.net.kp (Su-Il Kim)

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When gas molecules of different sizes are adsorbed on adsorbents with molecular-sized micropores, the activation energy required for the diffusion of gas molecules in the pores will be different, and consequently the diffusivity of molecules in the pores.

The use of CMS in gas separation processes is usually carried out in two packed bed adsorption columns, called pressure swing adsorption (PSA) [6]. Besides its use in separation processes, CMSs are widely used as catalysts and catalyst supports [7-10]. Several research groups have tried to prepare carbon molecular sieves to find efficient and economical techniques for gas separation processes.

Basically, carbon-based porous materials such as CMS can be produced by pyrolysis of various natural carbon materials such as coal, crust and wood [11-13] or by synthetic polymers such as polyvinylidene, polyimide, and phenol-formaldehyde resins [14-17]. It is generally known that the adsorption properties of carbon molecular sieves are affected by the nature of the precursor, the pyrolysis method, and the post-treatment conditions. Thus, although many studies have been carried out on the manufacture of CMS, the influence of each step of the production process on the product characteristics has not been systematically analyzed, but only limited to the analysis of individual steps. As a result, we did not identify a reasonable process parameter to produce CMS with an efficient characteristic structure. Therefore, in order to improve the technological properties of CMS, we chose new

matrix of apricot seed peel with extremely low ash content (less than 2%) and high microporous volume to adsorb oxygen, and hence, we confirmed the conditions of carbonization heat treatment, forming conditions, and pore control, which are suitable for the preparation of CMS for N₂/O₂ separation.

The CMS produced in this way is widely used in the chemical and metallurgical industries due to its rich source of raw materials and simple manufacturing.

2. Materials and Methods

2.1. Materials

The main raw material, apricot seed shell, benzene (99.9%), high temperature coal tar and coal pitch, the gases N₂ (99.998%) and O₂ (99.998%), thermosetting resin (novolac resin-401), the 5Å carbon molecular sieve were obtained from the Ponghwa Chemical Factory.

2.2. Preparation of CMS

The process of preparing a carbon molecular sieve for nitrogen separation from a raw material consists of five major steps, as shown in Figure 1.

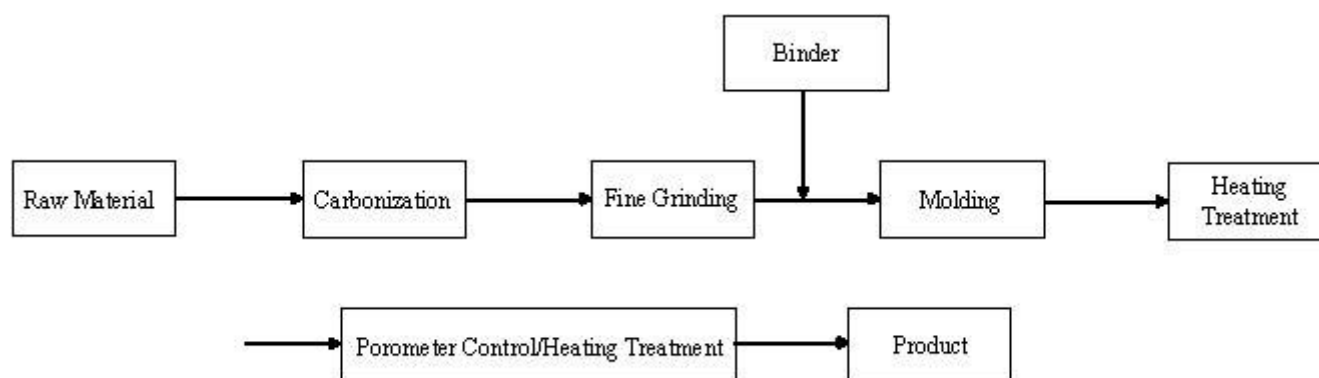


Figure 1. Process of CMS Manufacturing.

-Carbonization

Approximately 1kg of apricot seed shell, a carbon-containing raw material, was ground to reach a certain particle diameter and placed in a reducing atmosphere in a crucible. Then, the crucible was sealed and carbonized in a CWF-1200 muffle furnace with a thermostat.

-Fine Grinding

Using a conical ball mill (XMQ-240×90) with 500g of carbonaceous material, the particle size of the sample was determined to be less than 10μm. After a certain time of ball milling, the sample was sampled and the particle size distribution was measured using a LS-POP (VI) laser particle size

analyzer, and the milling was terminated when the desired particle size was reached.

-Molding

After milling, the raw material, the binder and the auxiliary binder were mixed in a certain proportion and then mixed. The homogeneously mixed material was molded into a Φ2mm×10mm cylinder in a screw extruder.

-Secondary Carbonization

Secondary carbonization is carried out in order to carbonize the binder in the forming step and to bond the carbon together to give a certain strength. After taking a certain amount of molded product into a reducing atmosphere crucible, it was

sealed and also subjected to secondary carbonization in a CWF-1200 muffle furnace.

-Porometer Control

Liquid-phase carbon deposition is performed to achieve a uniform pore diameter distribution. A pre-dried deposition precursor of 250g was placed in a vessel containing a certain proportion of benzene and tar, followed by soaking for 24h and drying. The dried sample was placed in a CWF-1200 muffle furnace for carbonization.

2.3. Characterization Evaluation of CMS

2.3.1. Adsorption Characterization Test

The experiments were carried out using a gravimetric adsorption capacity measurement apparatus as shown in Figure 2. In Figure 2, 1 and 2 are vacuum cokes, and the adsorption column consists of a transparent heat-resistant glass tube. A quartz spring is hung on the lid of the heat-resistant glass tube and a sample basket is hung on the end of the quartz spring.

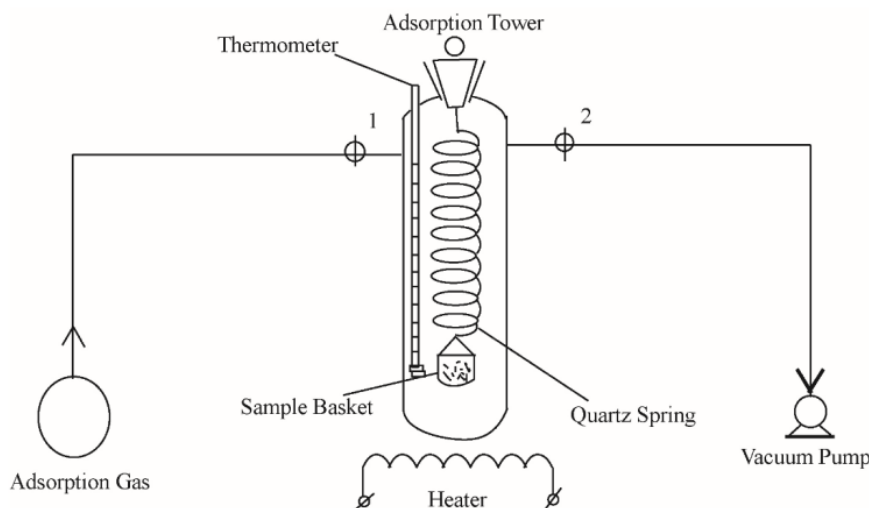


Figure 2. Gravimetric Adsorption Measuring Device.

In Figure 2, the adsorbent sample is first weighed about 0.5~1 g and put into the adsorption column with heat-resistant glassy material. Next, using Cock 1 and 2, the corresponding adsorbate gas as the gas inlet was fed into the adsorption column system and the system was replaced three times. After closing the cock 1 and opening the cock 2, the temperature inside the adsorption column system was heated and desorbed for 3h with a heater at 300°C under reduced pressure by a vacuum pump and then cooled to room temperature. The sensitivity of the quartz spring in the static adsorption measurement device is 21.1mg/mm, and the linear variation is measured with a KM-6 cassette meter with a measurement accuracy of ± 0.01 mm to determine the adsorption capacity of the adsorbent. The adsorption characteristics were plotted by closing the cock 2 and opening the cock 1 to provide the adsorbate gas to the adsorption chamber and measuring the adsorption amount over time.

From the adsorption characteristics of nitrogen and oxygen gases on the sample, the equilibrium adsorption (adsorption amount when the equilibrium adsorption state is reached, q_0), adsorption rate (adsorption amount for 1min, $q_{1\min}$), and selectivity (α) are calculated to compare the properties. In general, the amount of adsorption from the LDF model varies with time according to the following relationship:

$$q_t = q_{i0} \times (1 - \exp(-K_i t)) \quad (1)$$

Here

q_t : Instantaneous adsorption amount (mg/g) of component i .

q_{i0} : i -component equilibrium adsorption amount (mg/g).

K_i : Apparent mass transfer coefficient (1/s) of the i component.

t : Adsorption elapsed time (s).

Considering the data of the single-component gas adsorption measurements in Eq. (1) and the gravimetric adsorption rate measurement, the selectivity α for K_{O_2} and K_{N_2} is determined by the following equation:

$$\alpha = K_{O_2} / K_{N_2}$$

The adsorbate gas used in the experiments was pure, up to 99.998% oxygen or nitrogen gas.

2.3.2. Abrasion Rate Measuring Test

The wear resistance of the prepared samples was measured using a wear rate measuring device (36mm diameter, 300mm length cylindrical). For the measurement of magic rate, 100g of the molded heat-treated sample was put in the wear rate

measuring device and rotated at 54rpm for 45min. Then the sample was taken out and the weight (W) of the crushed material less than 1mm was measured.

Then, we find the wear resistance ratio (X , %) according to Eq. (2).

$$X = (100 - W) / 100 \quad (2)$$

2.3.3. PSA-N₂ Separation Performance Test

The most widely used carbon molecular sieve industry is air separation by pressure swing adsorption. Therefore, by performing air separation using a small variable pressure adsorption experimental setup, it is possible to observe in detail the performance of CMS and propose the best preparation method. The experimental setup is shown in Figure 3.

Operating conditions of the device are as follows:

Boost time $t_r=5s$, time of isostatic pressure $t_b=0.4s$, purge time $t_p=0.2s$, adsorption pressure $P_a=7atm$, adsorption half-cycle $t_a=58s$, adsorption column size $\Phi 26 \times 1500$ mm, adsorption column volume 0.8, adsorption column CMS

packing volume 500g (1 column).

The nitrogen generator experimental setup was:

Step 1 - 1 column: Boost, 2 column: Desorption

Step 2 - 1 column: adsorption, 2 column: blowing

Step 3 - 1 column: desorption, 2 column: booster

Step 4 - 1 column: blowing, 2 column: adsorption

Nitrogen production is carried out continuously in this manner. At the moment of reaching the set adsorption pressure, the nitrogen outlet valve of the adsorption column is opened, from which nitrogen is discharged into the nitrogen tank. Finally, the cycling step is the step up, the adsorption step, the blowing step, and the desorption step, depending on how the time of each step is set, the yield and purity of the product nitrogen will vary. These cycles are carried out alternately by two columns. The cycle step operation is controlled by a microcomputer. Generally, the adsorption time and swelling time are chosen to be equal, and the step-up time and desorption time are set equal. The oxygen concentration at the outlet of the PSA column was measured to determine the N₂ separation performance of CMS.

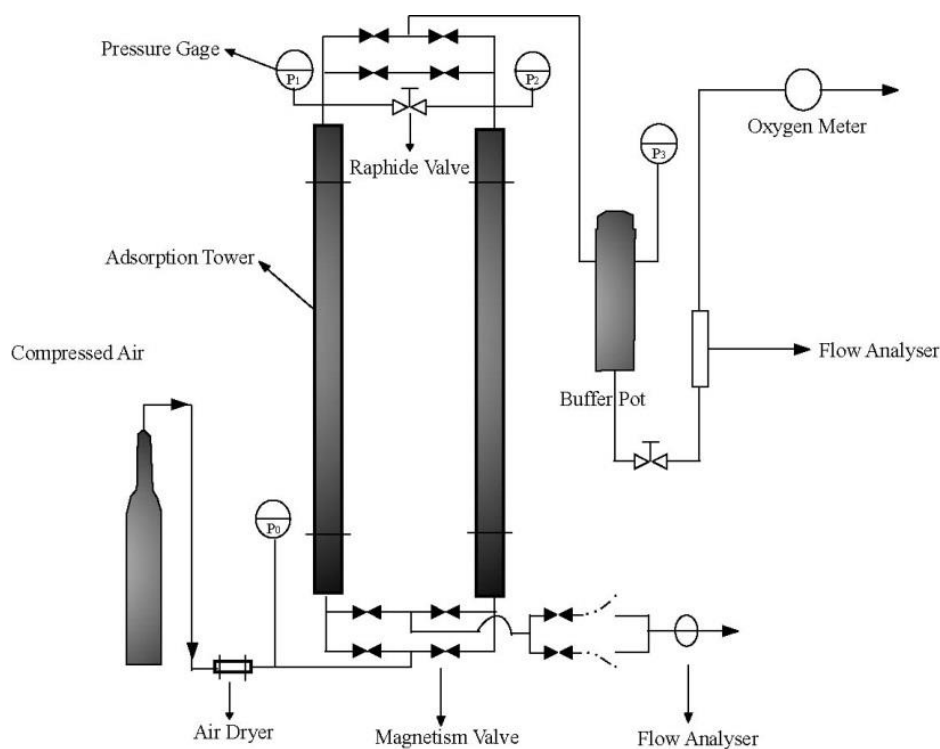


Figure 3. PSA Process Experimental Pilot Diagram.

3. Results and Discussion

3.1. Effect of Carbonization Condition

Carbonization was carried out in two stages. Up to temper-

atures below 400°C with a relatively high salt-dissolving fraction, the heating rate was fixed at $v=2^{\circ}C/min$, and the temperature range above 400°C was varied by varying the heating rate, the holding time and the holding temperature. The adsorption characteristics of carbides are shown in Table 1.

Table 1. Oxygen Adsorption Character according to Heat Treatment Condition.

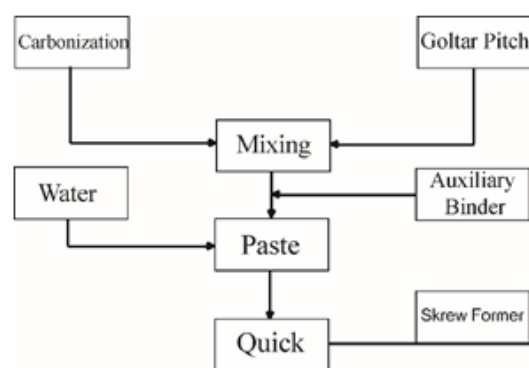
№	Retention Temperature/°C	Retention Time/h	Rate of Temperature Rise/°C·min ⁻¹	Density of Particles /g·mL ⁻¹	Balanced Adsorption Quantity	
					mL/g	mg/ mL
1	650	1	2	1.11	6.2	6.9
2	750	1	2	1.07	7.3	7.8
3	800	1	2	1.21	8.1	9.8
4	850	1	2	1.24	7.5	9.52
5	900	1	2	1.27	7.2	9.1
6	750	4	2	1.07	7.5	8.17
7	850	4	2	1.26	6.9	8.69
8	850	4	2	1.32	6.2	9.8
9	800	1	2	1.21	8.1	9.8
10	800	1	5	1.10	8.4	9.24
11	800	1	10	1.10	8.8	9.13

As shown in Table 1, when the heat treatment temperature is increased above 800°C, the amount of oxygen equilibrium adsorption decreases due to thermal shrinkage. It can be seen that the residence time at the heat treatment temperature $t=800^{\circ}\text{C}$ is longer than 1h, accompanied by the formation of micropores by pyrolysis and the reduction of micropore volume by thermal shrinkage, which leads to an increase in particle density and a decrease in oxygen balance adsorption. In addition, it can be seen that increasing the temperature-programmed rate from $v=2^{\circ}\text{C}/\text{min}$ to $10^{\circ}\text{C}/\text{min}$ increases the gravimetric oxygen adsorption capacity (mg/g), but with the overflow of the pyrolysis fraction per unit time, the micropore enlargement occurs and the volume-based adsorption capacity (mg/mL) decreases with decreasing particle density. Therefore, it can be seen that the optimum carbonization heat treatment condition is $t=800^{\circ}\text{C}$, $v=5^{\circ}\text{C}/\text{min}$ and 1h.

3.2. Effect of Molding Condition

Literature review and preliminary experiments confirmed that the optimum forming conditions were different depending on the ash content of the carbides. Therefore, an experimental investigation of the forming conditions suitable for

apricot seed-based carbides was carried out and a reasonable forming method was determined. The forming process of the milled carbides is shown in Figure 4.

**Figure 4.** Molding process of Milling Carbide.

After grinding the carbides to less than $10\mu\text{m}$, the thermo-setting resin components were mixed with different contents of 10~30% for molding, heat treatment and wear rate measurements are shown in Table 2.

Table 2. Abrasion Rate Determination Result (1).

№	Carbide Quantity /g	Heating Temperature/°C	Fabric percentage/%	Abrasion Rate/%
1	1	800	10	60
2	1	800	15	63

№	Carbide Quantity /g	Heating Temperature/°C	Fabric percentage/%	Abrasion Rate/%
3	1	800	20	65
4	1	800	25	69
5	1	800	30	72

As shown in Table 2, it can be seen that the wear resistance of the molded heat-treated materials is very low, and therefore, thermosetting resin alone cannot be used as the main binder. The abrasive wear rate was measured with a wear rate measuring de-

vice after the formation heat treatment by mixing the pitch and thermosetting resins into the carbides in an organic solvent. According to the results of previous studies, the pitch content for carbides was 10%. The experimental results are shown in Table 3.

Table 3. Abrasion Rate Determination Result (2).

№	Carbide+Pitch (10%)/g	Heating temperature/°C	Fabric Percentage/%	Abrasion Rate/%
1	1	800	5	93
2	1	800	10	96
3	1	800	15	98.5
4	1	800	20	99.8

As shown in Table 3, the highest wear resistance was observed when the thermosetting resin was molded and heat treated by 20% mixing. From these results, it was confirmed that the thermosetting resin can be used as an auxiliary binder and the optimum binder dosage is 10% pitch and 20% thermosetting resin.

3.3. Effect of Secondary Heat Treatment

The second heat treatment condition was set at a heating rate $v=5^{\circ}\text{C}/\text{min}$, holding time $\tau=1\text{h}$ and holding temperature $t=800^{\circ}\text{C}$. The single-component gas adsorption rate characteristics of the compacts after heat treatment are shown in Table 4 below.

Table 4. One Component Gas Adsorption Velocity Character of Modeling After Heating Treatment.

Adsorbate	Heat Treatment Time/h								
	0.5	1	2	3	5	10	15	20	25
$\text{O}_2/\text{mg}\cdot\text{g}^{-1}$	7.2	7.75	8.44	8.55	9	9.01	9.02	9.02	9.03
$\text{N}_2/\text{mg}\cdot\text{g}^{-1}$	2.08	3.32	4.43	5.68	6.37	7.34	7.35	7.356	7.36

Adsorption Temperature: 20°C , Adsorption Pressure: 0.1MPa

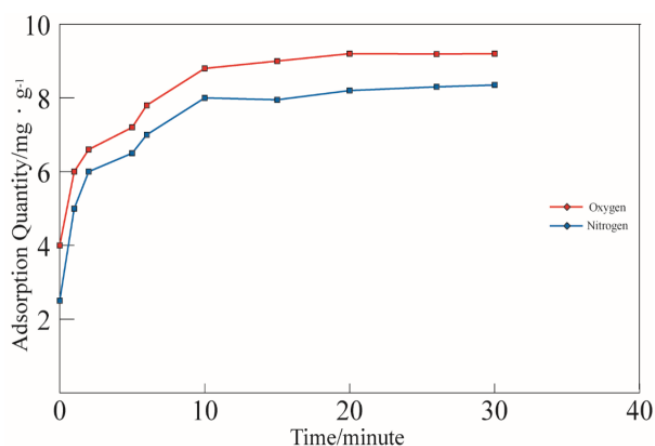
From the experimental results in Table 4, it can be said that after heat treatment, the granular carbides have enough properties as precursors for PSA- N_2 . First, the rate at which the adsorption equilibrium of oxygen is reached is very fast. The adsorption rate, which is a technical indicator of CMS for PSA- N_2 , exceeds 80% of the equilibrium oxygen uptake by

$q_{021\text{min}}=7.75\text{mg/g}$.

3.4. Effect of CMS According to Porometer Control Condition

The experimental results of N_2 and O_2 adsorption on the

molded heat-treated samples by mixing 20% thermosetting resin are shown in Figure 5.



(Molding Heat Treatment using 20% thermosetting resin as an auxiliary binder).

Figure 5. N_2 , O_2 Adsorption Characterization of Molded Heat Treatment Product.

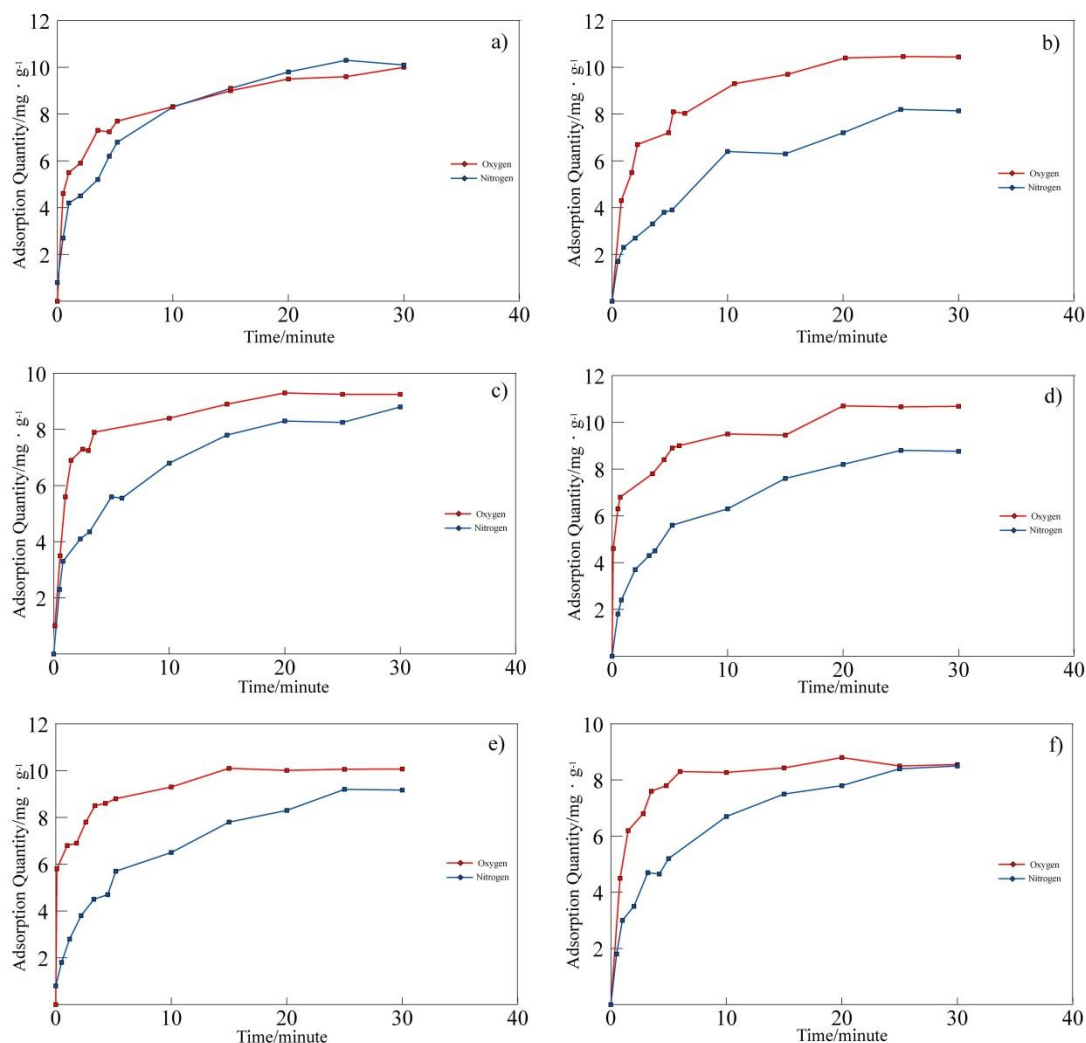


Figure 6. Adsorption Characteristics of Porometer-Controlled Samples.

a. Tar 33.3%, Benzene 66.7%, b. Tar 35.7%, Benzene 64.3%, c. Tar 38.5%, Benzene 61.5%, d. Tar 41.6%, Benzene 58.4%, e. Tar 43.5%, Benzene 56.6%, f. Tar 45.4%, Benzene- 54.6%

As shown in Figure 5, it can be seen that the molded heat treatment sample has a pore control due to the absence of oxygen adsorption selectivity to nitrogen with a 1min adsorption of 5.78mg/g of oxygen and a 1min adsorption of nitrogen of 4.93mg/g. Pore control of the molded heat-treatment products prepared under the conditions of No. 4 in Table 3 was carried out by immersing 1g of the molded material in a mixture of different amounts of tar and organic solvent for 24h and then annealing at 400°C. The results of the adsorption rate measurements of oxygen and nitrogen gas for six samples with controlled porosity at different compositions

of the pore control are shown in Figure 6.

From Figure 6a to f, it can be seen that the case of d) has the highest 1 min adsorption selectivity of oxygen to nitrogen and the equilibrium adsorption amount of oxygen. Of course, a change in tar composition results in an optimum dosage. Considering the adsorption characteristics of CMS prepared according to the heat treatment conditions, such as heat treatment temperature and residence time of molded heat-treated compacts incorporated using a pore regulator consisting of 41.6% tar and 58.4% organic solvent, are shown in Table 5.

Table 5. Adsorption Character of Manufactured CMS.

No	Heat Treatment Time/°C	Retention Time/h	Oxygen Balanced Adsorption Quantity/ mg·g ⁻¹	Rate of Oxygen Adsorption/mg·g ⁻¹	Selectivity
1	400	1	5.7	4.6	12
2	500	1	6.3	5.7	17
3	600	1	7.9	6.8	22
4	700	1	8.9	7.3	18
5	800	1	7.2	6.3	32
6	600	2	8.2	7.1	15
7	650	2	8.9	7.2	20
8	750	2	9.1	7.5	18

Table 5 shows that at 700°C, the equilibrium adsorption amount and adsorption rate are the highest, while at 800°C, the equilibrium adsorption amount and adsorption rate are low, but the selectivity is the highest.

3.5. Results of PSA-N₂ Separation Performance of the Manufactured CMS

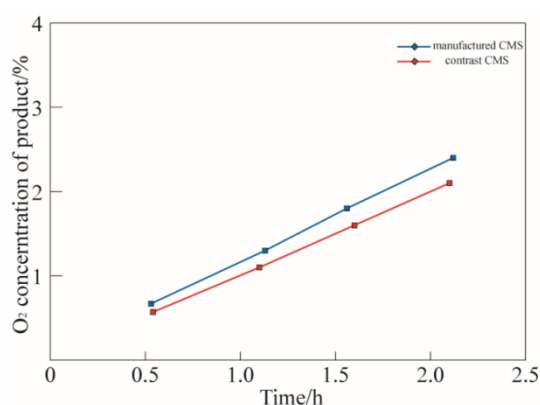


Figure 7. Change of O₂ concentration in product gas with space velocity.

The as-prepared seeded CMS was packed in a laboratory PSA-N₂ separator and the separation performance was tested. On the other hand, comparative separation experiments with CMS for comparison under the same conditions were carried out. The experimental results are shown in Figure 7.

As shown in Figure 7, the CMS made from apricot seed peel reached close to the CMS performance used for comparison with the nitrogen concentration of 99.2% at the spatial rate $S_V=0.5$ and 99% at $S_V=1$, approaching the time to reach the product concentration equilibrium. As above, we have determined the operating conditions and CMS performance of a PSA device capable of preparing nitrogen with a concentration of 99% for an adsorption pressure of $P_a=7$ atm and an adsorption half-cycle of $t_a=58$ s with a CMS made from apricot seed peel. From the above experimental results, we can see that the adsorption properties and wear resistance of our own CMS are much superior.

4. Conclusions

The process and method of preparation of CMS based apricot seed husk were newly established and the nitrogen purity of the PSA-N₂ separator was over 99% at $S_V=1$ using

this sorbent. The process of preparing carbon molecular sieves using apricot seed shells has five major steps.

The main operating conditions of the process are as follows:

Carbonization heat treatment conditions: holding temperature $t=800^{\circ}\text{C}$, heating rate $v=5^{\circ}\text{C}/\text{min}$, holding time $\tau=1\text{h}$.

Suitable binder dosage: 10% pitch and 20% thermosetting resin.

Secondary heat treatment conditions: holding temperature $t=800^{\circ}\text{C}$, heating rate $v=5^{\circ}\text{C}/\text{min}$, holding time $\tau=1\text{h}$.

The composition of the pore controller: 41.6% tar and 58.4% organic solvent.

Temperature of final heat treatment of molded heat-treated products: Holding temperature $t=800^{\circ}\text{C}$, heating rate $v=5^{\circ}\text{C}/\text{min}$ and holding time $\tau=1\text{h}$.

The carbon molecular sieve thus obtained showed very good performance for nitrogen separation, with oxygen balance of 7.2mg/g, 1min adsorption of 6.3mg/g and selectivity of 32.

Abbreviations

CMS Carbon Molecular Sieve
PSA Pressure Swing Adsorption

Conflicts of Interest

The authors declare no conflicts of interest.

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