**Study of Carbon Dioxide and Methane Adsorption on Carbon Molecular Sieves, Raw and Modified by Waste Engine Oil**

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*Abstract:* In this study, a carbon molecular sieve (CMS) was synthesized from walnut shells, followed by physical activation of the carbon content of the CMS. Adsorption of CO2 and CH4 onto raw and acid treated adsorbents were investigated using two sizes of 300-600 and 600-1180 microns. The adsorbents were impregnated with two solvent mixtures of waste engine oil with kerosene and with thinner with the proportion of 1:1 at 25°C. The highest adsorption for CO2 and CH4 was obtained at the suitable size of a group of CMS (A) adsorbents, with acid treated adsorbents being more efficient than raw adsorbents. The acid treated CMS (A-3) sample adsorbed 0.925 mole CO2/g and 0.353 mole CH4/g. The results indicated that by decreasing the granulation size of group CMS (R) adsorbents, the adsorption capacities for CO2 and CH4 were reduced while increasing the granulation size of group CMS (A) adsorbents resulted in an enhancement in the adsorption capacity for CO2 and CH4. Moreover, acid treated adsorbents achieved enhanced adsorption capacity for CO2 and CH4. Further modifications have reduced the adsorption capacity for CO2 and CH4 in impregnated adsorbents, due to a decrease in surface area, pore volume and pore size of the adsorbent.

RUNNING TITLE: CO2 AND CH4 ADSORPTION BY CMS

INTRODUCTION

Nowadays, adsorption and separation processes play a major role in various industries, such as chemical, oil, gas, petrochemical, and biochemical plants. With the advancement of adsorbents and process cycles, the adsorption is currently used as an important and essential process in gas separation in various industries.1

Carbon molecular sieves (CMSs) are adsorbents that perform the separation based on molecular dimensions, which are interesting both theoretically and empirically. In their carbon structure, they have narrow channels whose diameters are close to the diameter of the adsorbed particles. Therefore, these channels allow the passage of those particles that their diameter is as large as the openings of the channels and thus, larger molecules would not be stopped from the passage. The use of CMS in gas separation processes has been known for decades. Chemical processes have become increasingly interested in the development of low-cost gas separation processes and gas separation technology in quite close connection with CMS.2 CMSs are special classes of activated carbons which may exhibit several advantages such as excellent and sharp selectivity for the adsorption of planar molecules, higher hydrophobicity, higher resistance to both alkaline and acidic media, and higher thermal stability at high temperatures under inert atmosphere.3 They can be prepared by a variety of precursors such as polymers, coconut shells, wood, bituminous carbon, and other lignin cellulosic materials .4

In this mechanism, smaller particles have high potential and selectivity. CMSs have narrow pores with a diameter size near to that of particles in the gas mixture. In these narrow channels, the energy of the interaction between carbon and gas molecules involves repulsive and diffusive interactions. When the channels are much narrower than that of penetrating molecules, repulsive forces are dominant and molecules need activation energy to pass through the channels.5

Compared to other structures of molecular sieves such as zeolites, CMSs are among the most desirable candidates in the adsorption and separation of gases due to typical advantages such as high hydrophobicity, suitable thermal and chemical resistance against acids and bases, the stability of carbon structure in a neutral environment, and low fabrication costs.6, 7

Having the above features as well as being cheap and abundant, agricultural wastes are the most suitable raw materials for producing activated carbon, among which are the following raw materials: Bagasse, rice bar, cardamom core, olive stone, almond shell, apricot stone, cherry stone, grape seed8, hazelnut shell, walnut shell9, coconut shell10, pomegranate seed11, etc.

In addition to agriculture, other polymeric and mineral materials such as worn tires12, waste newspapers13, coal14, etc might also be used as the raw materials.CMSs have not only been used for adsorbing methane, carbon dioxide15, carbon monoxide, and nitrogen16, but also for separating multiple gas mixtures like carbon monoxide-hydrogen–methane17, methane–carbon dioxide18, and methane–nitrogen19.

In this study, the CMS was prepared by a walnut shell followed by the physical activation. Following the preparation of the CMS, it was granulated, acid treated, and impregnated by waste oil mixed with various solvents with the proportion of 1:1. Then, the adsorption capacity of prepared samples for carbon dioxide and methane was measured under the same conditions.

EXPERIMENTAL

*Steps for Preparing the CMS using Walnut Shell*

5 kg of the walnut shells were used to prepare 300 g of CMS. First of all, the walnut shells were wiped, crushed, and heated at 110 °C in order to remove moisture from the material. they were then heated at about 300 °C for 60 minutes to remove all organic compounds. In the final step, they were heated at 450-550 °C for 2-4 hours to be converted into charcoal. All the above mentioned procedures were performed in the absence of air.

In this research, the physical method was selected and performed for carbon activation. Carbon was activated in two steps in a rotary furnace at 700-900 °C in the presence of water vapor and carbon dioxide:

1. at 700-800 °C in the presence of water vapor

2. at 850-900 °C in the presence of water vapor

All preparation steps are presented in Fig.1.

By consuming some of the existing carbon and also removing the bitumen-like materials remained from the pyrolysis phase, these reactions cause the opening, better expansion, and formation of internal connection between carbon pores. As a result, the internal area of the pores has increased significantly.

The following reactions are used to convert carbonized materials into the gas by using water vapor and carbon dioxide:

 C + H2O ↔ CO2 + H2 (1)

 C + CO2 ↔ 2CO (2)

 CO + H2O ↔ H2 + CO2 (3)

 Finally, we obtained 300 g of CMS.



Fig.1. An overview of the steps involved in the synthesis of activated carbon20

*Division based on Granulation*

The material was granulated by following standard ASTM E001121 sieves to synthesize CMSs with meshes of 300-600-1180 microns.

*Division based on pH*

*Group CMS-* *Raw*

Following the preparation of the CMS, we first measured its pH according to the ASTM D-383822 instruction. First, distilled water acidity was measured and then 100 ml of distilled water was added to the beaker containing 10 g of activated carbon. A gentle turbulence was created in the beaker using a magnetic stirrer; then the solution was passed through a filter. The acidity was measured without delay, which was neutral (pH=6.5).

*Group CMS –* *Acid treated*

Acid treatment was performed to release the pores from sedimentary materials. 100 g of CMS was mixed with 100 ml of a nitric acid solution of 0.1 M concentration and was then placed under a hood for 2 hours at 90 °C. Then, the mixture was cooled and washed several times with distilled water. Next, the mixture was filtered and the acidity of the solution was measured immediately, which was acidic (pH 4.5).

*Division based on modifying*

*Preparation of the modifying Solution*

A mixture of waste engine oil with Kerosene, thinner, ethanol and benzine solvents was considered for modification. However, the mixture of waste engine oil with ethanol and benzine was not selected due to the inability to process and reduce the dimensions of the adsorbent`s pores. First of all, the waste engine oil was passed through a filter and chemical treatment (physical separation) was performed. The Kerosene and thinner solvents were separately mixed with waste oil with a mixing proportion of 1:1 and the resulting mixture was stirred thoroughly. Then, the mixture was kept at 25 °C for 30 minutes. After that, it was diluted and deposited. Finally, the deposited sludge was separated. Once the CMSs were granulated, they were acid treated and impregnated. To dry the CMSs, they were exposed to room temperature of 25 °C for 24 hours.

Then, the samples were placed in an oven at 125 °C for 2 hours. Afterward, the calcination operation was carried out at 500°C with a temperature rate of 10 °C/ min for 1 hour.

*CMS modifying Method*

1 g of the prepared CMSs was immersed in 50 ml of the filtered solution for 1 hour and then, the CMSs were separated from the solutions using a filter paper.

*Drying and Calcination*

*Encoding*

Adsorbent samples were acid treated, impregnated and encoded based on the granulation (Tables I, II).

Table I. Encoding the acid treated samples

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Sample Cod | U.S. STD Sieve No. | Particle Size, µm | pH | Calcination |
|  |  |  |  | Temperature,°C | Time, h |
| CMS(R) | - | - | 6.5 | - | - |
| CMS(R-2) | 30-50 | 300-600 | 6.5 | 500 | 1 |
| CMS(R-3) | 16-30 | 600-1180 | 6.5 | 500 | 1 |
| CMS(A-2) | 30-50 | 300-600 | 4.5 | 500 | 1 |
| CMS(A-3) | 16-30 | 600-1180 | 4.5 | 500 | 1 |

Table II. Encoding the impregnated samples

|  |  |  |  |
| --- | --- | --- | --- |
| Sample Cod | Used Sample | Chemical TreatmentProportion, 1:1 | Calcination |
| Temperature, °C | Time, h |
| CMS(R-2-K) | CMS(R-2) | Oil Waste: Kerosene | 500 | 1 |
| CMS(R-3-K) | CMS(R-3) | Oil Waste: Kerosene | 500 | 1 |
| CMS(R-2-T) | CMS(R-2) | Oil Waste: Thinner | 500 | 1 |
| CMS(R-3-T) | CMS(R-3) | Oil Waste: Thinner | 500 | 1 |
| CMS(A-2-K) | CMS(A-2) | Oil Waste: Kerosene | 500 | 1 |
| CMS(A-3-K) | CMS(A-3) | Oil Waste: Kerosene | 500 | 1 |
| CMS(A-2-T) | CMS(A-2) | Oil Waste: Thinner | 500 | 1 |
| CMS(A-3-T) | CMS(A-3) | Oil Waste: Thinner | 500 | 1 |

*Reactor tests*

*Description of Gas Adsorption Method and the Measurement Device*

Weighted and volumetric methods are two common approaches for measuring the adsorption equilibrium. In the weighted method, the adsorption equilibrium is examined based on the measurement of weight changes in the adsorbent sample during adsorption; while in the volumetric method, the absorption equilibrium is measured through the difference in pre and post-absorption gas content.

 In the present study, the adsorption of carbon dioxide and methane by the synthesized adsorbents was investigated using a gas adsorption measurement device which was designed and made based on the volumetric method. A schematic view of the device is shown in Fig. 2. In this device, a tank was used for the adsorbed gas, and another tank was used as an adsorption column. Furthermore, a water-ice bath was used to adjust the temperature. By adjusting the regulator and opening the capsule valve, the gases enter the gas tank through valve1. By reading from the digital display and pressure gauge, the gas pressure in the tank is set to 2 bars through valves1 and 4. All parts of the device, such as pipes, valves, fittings, gas tanks and absorption columns, are made of steel and have a maximum pressure tolerance of about 40 bars.

*Measurement of CH4 and CO2 Adsorption Capacity*

First of all, the whole system was exposed to a vacuum for 5 minutes. 2 g of adsorbent sample was placed in the adsorption column and its temperature was increased to 100 °C for five minutes with the help of a heating jacket. Then the absorption column was again placed under vacuum to remove all the adsorbed gases by the adsorbent. Next, the temperature of the absorption column was reduced to 25 °C using a water-ice bath container. By closing valves 2 and 4 and opening valve1, the pressure of the gas tank was set to 2 bars by valves1 and 4. After closing valve1 and writing down P0, the gas was allowed to occupy all of the space available between valves 1, 2, and 4 by opening valve 2. From this moment on, changes in the gas pressure with time inside the system were recorded in a computer and continued until reaching the equilibrium state (P1). In order to determine the adsorbed content, a control test was performed with Helium and that part of the pressure drop that occurs after the expansion of the gas in the system (Dead volume) and was not related to gas adsorption was deducted from the total observed pressure drop in each test. The adsorbent volume was also measured and deducted from the volume of the system.

After all the above steps, we started to examine the adsorption for carbon dioxide and methane and recorded the measurements of the slope of the pressure variations according to time, until the constant pressure was maintained over time.



Fig. 2. Schematic view of the device for measuring gas adsorption capacity using constant volume-variable pressure (volumetric) method

*Characterization of the Adsorbents*

*Analysis of BET Specific Surface Area of the CMS Adsorbent Selected in terms of Adsorption Capacity*

The surface area, pore volume and pore size of the synthetic CMS were characterized by Micromeritix3020 instrument using the BET method. First, a certain amount of the adsorbent sample was degassed at 200 °C for 2 hours and then, desorption was performed in the presence of liquid nitrogen.

RESULTS AND DISCUSSION

In order to determine the effects of granulation in this study, we investigated and calculated the number of moles of carbon dioxide and methane adsorbed by the adsorbent samples at the 2 bar pressure and 25 °C, with the help of equations 4, 5, and 6 and using EXCEL software.18

p × V = n × R × T (4)

 (5)

 Where, p0 is the equilibrium pressure, p1, 2, 3,…, n is the pressure at different times (atm), V is the volume (Liter), T is the temperature (K), R is the universal gas constant (Liter·atm·K-1·mol-1), and n is the number of adsorbed gas moles.

*Granulation effects on the Adsorption Capacity of Carbon Dioxide and Methane*

The results indicated that in group CMS (A) adsorbents, the adsorption capacity has increased with increasing size, while in group CMS (R) adsorbents, the adsorption capacity has decreased when the size increases, CMS (A-3) adsorbent has the highest amount of adsorption capacity for methane and carbon dioxide, CMS (A-2-T) and CMS (R-3-K) adsorbents have the lowest adsorption capacity for carbon dioxide and methane. (As shown in Table III, IV)

Table III. Results of granulation effects on the Capacity of Adsorption for Carbon Dioxide and Methane by group CMS (A) adsorbents

|  |
| --- |
| CO2 adsorption capacity, mmol g-1 |
| CMS(A-2) | CMS(A-3) | CMS(A-2-K) | CMS(A-3-K) | CMS(A-2-T) | CMS(A-3-T) |
| 0.639 | 0.925 | 0.477 | 0.61 | 0.381 | 0.629 |
| CH4 adsorption capacity, mmol g-1 |
| CMS(A-2) | CMS(A-3) | CMS(A-2-K) | CMS(A-3-K) | CMS(A-2-T) | CMS(A-3-T) |
| 0.343 | 0.353 | 0.191 | 0.248 | 0.114 | 0.343 |

Table IV. Results of granulation effects on the Capacity of Adsorption for Carbon Dioxide and Methane by group CMS (R) adsorbents

|  |
| --- |
| CO2 adsorption capacity, mmol g-1 |
| CMS(R-2) | CMS(R-3) | CMS(R-2-K) | CMS(R-3-K) | CMS(R-2-T) | CMS(R-3-T) |
| 0.62 | 0.6 | 0.532 | 0.41 | 0.534 | 0.524 |
| CH4 adsorption capacity, mmol g-1 |
| CMS(R-2) | CMS(R-3) | CMS(R-2-K) | CMS(R-3-K) | CMS(R-2-T) | CMS(R-3-T) |
| 0.315 | 0.276 | 0.114 | 0.095 | 0.165 | 0.162 |

*Effects of Acid Treatment of Granulated CMSs on the Adsorption Capacity of Methane and Carbon Dioxide*

The results showed that acid treated samples, as compared to raw (non-acid treated) ones, have a higher adsorption capacity for carbon dioxide and methane. As compared to CMS (R-3) adsorbent, the adsorption capacity for carbon dioxide and methane in CMS (A-3) adsorbent has increased by 54% and 28%, respectively. However, the adsorption capacity for carbon dioxide and methane in CMS (A-2) adsorbent had only a slight increase compared to CMS (R-2) adsorbent. The reason for a significant increase in the adsorption capacity of CMSs can be the granulation size in the acid treatment process. This is because coarser granules have larger channels that make it possible for the acidic solution to get more access into the pores, this will result in more pores to be released from sediments (as shown in Table V).

Table V. Effects of acid treatment of granulated CMSs on their Adsorption Capacity for Methane and Carbon Dioxide

|  |
| --- |
| CO2 adsorption capacity, mmol g-1 |
| CMS(R-2) | CMS(A-2) | CMS(R-3) | CMS(A-3) |
| 0.62 | 0.639 | 0.6 | 0.925 |
| CH4 adsorption capacity, mmol g-1 |
| CMS(R-2) | CMS(A-2) | CMS(R-3) | CMS(A-3) |
| 0.315 | 0.343 | 0.276 | 0.353 |

*Examining the Effects of impregnation on Methane and Carbon Dioxide Adsorption Capacity*

The results suggested that acid treated adsorbents have a higher adsorption capacity compared to the *impregnated* ones. In comparison to CMS (A-2-T),with a granulation size of 300-600 µm, which wasacid treated and *impregnated* with waste engine oil and thinner, CMS (A-2-K) with a granulation size of 300-600 µm, which was acid treated and *impregnated* with waste engine oil and Kerosene, had a higher adsorption capacity for carbon dioxide and methane. In the rest of the adsorbents, those that were *impregnated* with waste engine oil and thinner had a higher adsorption capacity for carbon dioxide and methane as compared to the adsorbents which were *impregnated* with waste engine oil and kerosene (as shown in Table VI ~~6~~).

Table VI. The results of the effects of modifying the adsorbent on its adsorption capacity for carbon dioxide and methane

|  |  |
| --- | --- |
| CO2 adsorption capacity, mmol g-1 | CH4 adsorption capacity, mmol g-1 |
| CMS(A-2-T) | CMS(A-2-K) | CMS(A-2) | CMS(A-2-T) | CMS(A-2-K) | CMS(A-2) |
| 0.381 | 0.477 | 0.639 | 0.114 | 0.191 | 0.343 |
| CMS(A-3-K) | CMS(A-3-T) | CMS(A-3) | CMS(A-3-K) | CMS(A-3-T) | CMS(A-3) |
| 0.61 | 0,629 | 0.925 | 0.248 | 0.343 | 0.353 |
| CMS(R-2-K) | CMS(R-2-T) | CMS(R-2) | CMS(R-2-K) | CMS(R-2-T) | CMS(R-2) |
| 0.532 | 0.534 | 0.620 | 0.114 | 0.165 | 0.315 |
| CMS(R-3-K) | CMS(R-3-T) | CMS(R-3) | CMS(R-3-K) | CMS(R-3-T) | CMS(R-3) |
| 0.410 | 0.524 | 0.600 | 0.095 | 0.162 | 0.276 |

*The result of BET Special Surface Area Analysis*

The results of BET test indicated that acid treated CMS (A-3) sample with a granulation size of 600-1180 microns that had a higher adsorption capacity for carbon dioxide and methane compared to the other adsorbents, has a larger BET special surface area compared to non-granulated, raw, acid treated CMS (R) (as shown in Table VII).

Table VII. Textural properties of CMS (R) and CMS (A-3)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| samples | BET surface area,m² g-1 | Micro pore area,m² g-1 | Micro pore volume,cm³ g-1 | Externalsurface area, m² g-1 |
| CMS(R) | 330 | 250 | 0.115 | 80 |
| CMS(A-3) | 432 | 334 | 0.155 | 98 |

To investigate the priority of prepared adsorbents, the adsorbents with almost similar components and operating conditions are listed in Table VIII. The main purpose of the current investigation is enhancing the adsorption capacity. According to Table VIII, among all listed prepared adsorbents, CMS (A-3) has the highest adsorption capacity.

 Table VIII. Performance of the four adsorbents and some other similar works.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Adsorbent | *p* / kPa | *t*/ °C | Adsorption capacity, mmol g-1 | Reference |
| CO2 | CH4 |  |
| CMS | 198.9 | 25 | 2.527 | ..... | 16 |
| CMS S-6 | 40 | 27 | 0.352 | 0.412 | 24 |
| CMS | .... | 25 | 1.15 | 0.95 | 25 |
| CMS | 206 | 25 | ..... | 1.41 | 26 |
| CMS-I | 200 | .... | 1.15 | ...... | 27 |
| CMS-III | 200 | ..... | 0.85 | .... | 27 |
| CMS-3K-12 | 292.5 | .... | 2.364 | .... | 28 |
| C-102 | 100 | 25 | 0.003 | .... | 29 |
| CMS(A-2) | 200 | 25 | 0.639 | 0.343 | Current work |
| CMS(A-3) | 200 | 25 | 0.925 | 0.353 | Current work |
| CMS(A-2-K) | 200 | 25 | 0.477 | 0.191 | Current work |
| CMS(A-3-K) | 200 | 25 | 0.610 | 0.248 | Current work |

CONCLUSION

The results of the present study showed that the CMS, which was synthesized by walnut shells, was capable of adsorbing carbon dioxide and methane at a pressure of 2 bar and a temperature of 25 °C. This CMS adsorbs more carbon dioxide (with a molecular diameter of 0.33 nm) than methane (with a molecular diameter of 0.38 nm).

The results suggest that the acid treatment of adsorbent has increased its adsorption capacity for carbon dioxide and methane. The adsorption capacity for carbon dioxide and methane in group CMS (R) adsorbents (*impregnated* and acid treated CMSs that are not acid treated) was decreased with increasing the granulation size; while the adsorption capacity of carbon dioxide and methane in group CMS (R) adsorbents (*impregnated* and acid treated CMSs that were acid treated) has increased with increasing the granulation size. The results showed that the modification has reduced the adsorption capacity. In all samples, the adsorption capacity of the adsorbents has decreased after modification. The results of BET analysis have indicated that the acid treated CMS (A-3), with a granulation size of 600-1180 microns, had more surface area, volume, and pore size as compared to non-granulated, non-acid treated and acid treated CMS (R). The highest adsorption capacity for carbon dioxide and methane was obtained for CMS (A-3) sample. (0.925 mmol/g for carbon dioxide and 0.353 mmol/g for methane).

SUPPLEMENTARY MATERIAL

Details of adsorption capacity of carbon dioxide and methane as compared to the adsorbents which were impregnated with waste engine oil and kerosene in Figs 3-10, are available electronically from http://www.shd.org.rs/JSCS/.

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