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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, 300–600 and 600–1180 μm. The adsorbents were impregnated with two solvent mixtures of waste engine oil with kerosene and with thinner at a 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 the raw adsorbents. The acid treated CMS_(A-3) sample adsorbed 0.925 mol CO₂ g⁻¹ and 0.353 mol CH₄ g⁻¹. 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 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.

Keywords: adsorption capacity; carbon molecular sieve (CMS); impregnated adsorbents; acid treated adsorbent.

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, adsorption is currently

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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 the diameters of which are similar to those of the adsorbed particles. Therefore, these channels allow the passage of such particles with diameter 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 is in quite close connection with CMS.² CMSs are special classes of activated carbons that 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 an inert atmosphere.³ They can be prepared from 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 the size of the penetrating molecules, repulsive forces are dominant and the 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 the carbon structure in neutral environments, 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 seed,⁸ hazelnut shell, walnut shell,⁹ coconut shell,¹⁰ pomegranate seed,¹¹ *etc.*

In addition to agriculture, other polymeric and mineral materials, such as worn tires,¹² waste newspapers,¹³ coal,¹⁴ *etc.* might also be used as raw materials. CMSs have not only been used for adsorbing methane, carbon dioxide,¹⁵ carbon monoxide, and nitrogen,¹⁶ but also for separating multiple gas mixtures, such as carbon monoxide–hydrogen–methane,¹⁷ methane–carbon dioxide,¹⁸ and methane–nitrogen.¹⁹

In this study, the CMS was prepared from walnut shell followed by physical activation. Following the preparation of the CMS, it was granulated, acid treated, and impregnated by waste oil mixed with various solvents in 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

Walnut shells (5 kg) were used to prepare 300 g of CMS. First, 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 min to remove all organic compounds. In the final step, they were heated at 450–550 °C for 2–4 h 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.

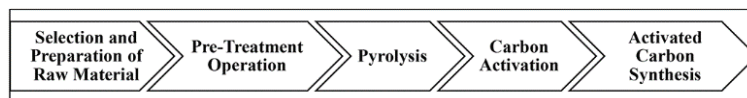


Fig. 1. An overview of the steps involved in the synthesis of activated carbon.²⁰

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

The following reactions were used to convert carbonized materials into gases using water vapor and carbon dioxide:



Finally, 300 g of CMS was obtained.

Division based on granulation

The material was granulated using the following standard ASTM E0011²¹ sieves to synthesize CMSs with meshes of 300–600–1180 μm.

Division based on pH

Group CMS – raw. Following the preparation of the CMS, its pH was measured according to ASTM D-3838²² instructions. 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 and then the solution was passed through a filter. The acidity, which was neutral (pH 6.5), was measured immediately.

Group CMS – acid treated. Acid treatment was performed to release the pores from sedimentary materials. CMS (100 g) was mixed with 100 ml of 0.1 M nitric acid solution and then placed under a hood for 2 h at 90 °C. The mixture was then 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 modification

Preparation of the modifying solution. A mixture of waste engine oil with kerosene, thinner, ethanol and benzene solvents was considered for modification. However, the mixture of waste engine oil with ethanol and benzene was not selected due to the inability to process and reduce the dimensions of the pores of the adsorbent. The waste engine oil was first passed through a filter and chemical treatment (physical separation) was performed. The kerosene and thinner solvents were separately mixed with waste oil at a mixing proportion of 1:1 and the resulting mixture was thoroughly stirred. Then, the mixture was kept at 25 °C for 30 min and then diluted and deposited. Finally, the deposited sludge was separated. Once the CMSs were granulated, they were acid treated and impregnated. The CMSs were dried at room temperature (25 °C) for 24 h. Then, the samples were placed in an oven at 125 °C for 2 h. Subsequently, the samples were heated at 10 °C min⁻¹ to 500 °C and calcined at this temperature for 1 h.

CMS modifying method. The prepared CMSs (1 g) were immersed in 50 ml of filtered solution for 1 h and then separated from the solution using filter paper.

Drying and calcination

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

TABLE I. Encoding of the acid treated samples

Sample code	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 of the impregnated samples; calcination temperature: 500 °C; calcination time: 1 h

Sample code	Used sample	Chemical treatment, proportion: 1:1
CMS _(R-2-K)	CMS _(R-2)	Oil waste:kerosene
CMS _(R-3-K)	CMS _(R-3)	Oil waste:kerosene
CMS _(R-2-T)	CMS _(R-2)	Oil waste:thinner
CMS _(R-3-T)	CMS _(R-3)	Oil waste:thinner
CMS _(A-2-K)	CMS _(A-2)	Oil waste:kerosene
CMS _(A-3-K)	CMS _(A-3)	Oil waste:kerosene
CMS _(A-2-T)	CMS _(A-2)	Oil waste:thinner
CMS _(A-3-T)	CMS _(A-3)	Oil waste:thinner

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 adsorption equilibrium is measured through the difference in pre and post-adsorption 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 that 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 a valve 1. By reading from the digital display and pressure gauge, the gas pressure in the tank is set to 2 bars through valves 1 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.

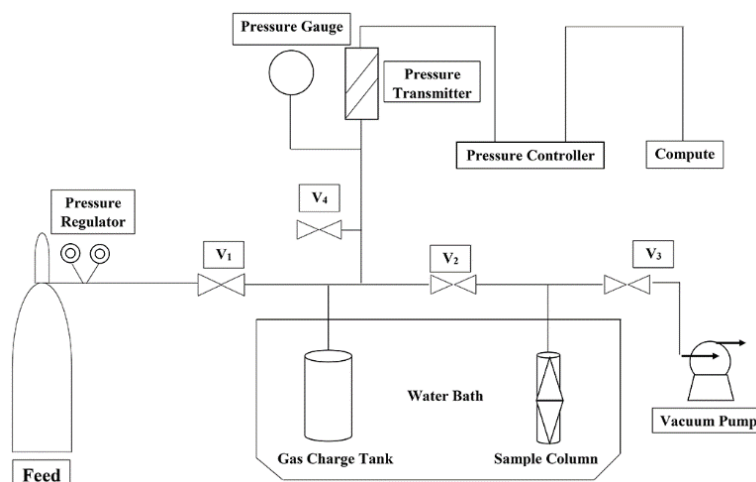


Fig. 2. Schematic view of the device for measuring the gas adsorption capacity using a constant volume-variable pressure (volumetric) method.

Measurement of CH₄ and CO₂ adsorption capacity

After exposing the whole system to a vacuum for 5 min, 2 g of an adsorbent sample was placed in the adsorption column and its temperature was increased to 100 °C for 5 min. using a heating jacket. Then the absorption column was again placed under vacuum to remove all the gases adsorbed 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 valve 1, the pressure of the gas tank was set to 2 bars by valves 1 and 4. After closing valve 1 and recording p_0 , the gas was allowed to occupy all of the available space 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 vol-

ume) that 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, examination of the adsorption for carbon dioxide and methane was commenced and the slope of the pressure variations with time were recorded until the pressure remained constant over time.

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 CMSs were characterized by Micromeritix3020 instrument using the BET method. First, a certain amount of the adsorbent sample was degassed at 200 °C for 2 h and then, desorption was performed in the presence of liquid nitrogen.

RESULTS AND DISCUSSION

In order to determine the effects of granulation, the number of moles of carbon dioxide and methane adsorbed by the adsorbent samples at a pressure of 2 bar at 25 °C were investigated and calculated using Eqs. (4) and (5) and Excel software:¹⁸

$$pV = nRT \quad (4)$$

$$n = \frac{Vp(p_0 - p_{1,2,3,\dots,n})}{RT} \quad (5)$$

where, p_0 is the equilibrium pressure, $p_{1,2,3,\dots,n}$ is the pressure at different times (atm), V is the volume (L), T is the temperature (K), R is the universal gas constant ($\text{L}\cdot\text{atm}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$), and n is the moles of adsorbed gas.

Granulation effects on the adsorption capacity of carbon dioxide and methane. The results indicated that in the group $\text{CMS}_{(A)}$ adsorbents, the adsorption capacity increased with increasing size, while in the group $\text{CMS}_{(R)}$ adsorbents, the adsorption capacity decreased with increasing size. The $\text{CMS}_{(A-3)}$ adsorbent had the highest adsorption capacity for methane and carbon dioxide, while the $\text{CMS}_{(A-2-T)}$ and $\text{CMS}_{(R-3-K)}$ adsorbents had the lowest adsorption capacity for carbon dioxide and methane, Tables III and IV.

TABLE III. Effects of granulation on the adsorption capacity for carbon dioxide and methane (mmol g^{-1}) by group $\text{CMS}_{(A)}$ adsorbents

$\text{CMS}_{(A-2)}$	$\text{CMS}_{(A-3)}$	$\text{CMS}_{(A-2-K)}$	$\text{CMS}_{(A-3-K)}$	$\text{CMS}_{(A-2-T)}$	$\text{CMS}_{(A-3-T)}$
CO ₂ adsorption capacity					
0.639	0.925	0.477	0.61	0.381	0.629
CH ₄ adsorption capacity					
0.343	0.353	0.191	0.248	0.114	0.343

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 the CMS_(R-3) adsorbent, the adsorption capacity for carbon diox-

TABLE IV. Effects of granulation on the adsorption capacity for carbon dioxide and methane (mmol g⁻¹) by group CMS_(R) adsorbents

CMS _(R-2)	CMS _(R-3)	CMS _(R-2-K)	CMS _(R-3-K)	CMS _(R-2-T)	CMS _(R-3-T)
CO ₂ adsorption capacity					
0.620	0.600	0.532	0.41	0.534	0.524
CH ₄ adsorption capacity					
0.315	0.276	0.114	0.095	0.165	0.162

ide and methane of the CMS_(A-3) adsorbent was increased by 54 and 28 %, respectively. However, the adsorption capacity for carbon dioxide and methane of the CMS_(A-2) adsorbent was only slightly increased compared to the CMS_(R-2) adsorbent. The reason for the significant increase in the adsorption capacity of CMSs could 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 gain more access into the pores, which would 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 (mmol g⁻¹)

CMS _(R-2)	CMS _(A-2)	CMS _(R-3)	CMS _(A-3)
CO ₂ adsorption capacity			
0.62	0.639	0.6	0.925
CH ₄ adsorption capacity			
0.315	0.343	0.276	0.353

Examination of the effects of impregnation on methane and carbon dioxide adsorption capacity

The results suggested that the 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 was acid 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 other adsorbents, those that were impregnated with waste engine oil and thinner had a higher adsorption capacity for carbon dioxide and methane as com-

pared to the adsorbents which were impregnated with waste engine oil and kerosene (as shown in Table VI).

TABLE VI. The results of the effects of modification of the adsorbent on its adsorption capacity for carbon dioxide and methane (mmol g^{-1})

CO ₂ adsorption capacity			CH ₄ adsorption capacity		
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

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 μm that had a higher adsorption capacity for carbon dioxide and methane compared to the other adsorbents had a larger BET special surface area compared to non-granulated, raw, acid treated CMS_(R) (as shown in Table VII).

Additional data related to adsorption capacities are given as Supplementary material to this paper.

TABLE VII. Textural properties of CMS_(R) and CMS_(A-3)

Sample	BET surface area, $\text{m}^2 \text{g}^{-1}$	Micro pore area $\text{m}^2 \text{g}^{-1}$	Micro pore volume $\text{cm}^3 \text{g}^{-1}$	External surface area $\text{m}^2 \text{g}^{-1}$
CMS _(R)	330	250	0.115	80
CMS _(A-3)	432	334	0.155	98

The performance of the prepared adsorbents are compared with those of other adsorbents with almost similar components and operating conditions are presented in Table VIII. The main purpose of the current investigation was to enhance 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 in similar works

Adsorbent	p / kPa	$T / ^\circ\text{C}$	Adsorption capacity, mmol g^{-1}		Reference
			CO ₂	CH ₄	
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

TABLE VIII. Continued

Adsorbent	p / kPa	T / °C	Adsorption capacity, mmol g ⁻¹		Reference
			CO ₂	CH ₄	
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

CONCLUSIONS

The results of the present study showed that the CMS that was synthesized from 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 an adsorbent increased its adsorption capacity for carbon dioxide and methane. The adsorption capacity for carbon dioxide and methane in the group CMS_(R) adsorbents (impregnated and acid treated CMSs that are not acid treated) decreased with increasing granulation size; while the adsorption capacity for carbon dioxide and methane in the group CMS_(R) adsorbents (impregnated and acid treated CMSs that were acid treated) increased with increasing the granulation size. The results showed that the modification reduced the adsorption capacity. In all samples, the adsorption capacity of the adsorbents was decreased after modification. The results of BET analysis indicated that the acid treated CMS_(A-3) with a granulation size of 600–1180 µm had higher 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 with the CMS_(A-3) sample (0.925 mmol g⁻¹ for carbon dioxide and 0.353 mmol g⁻¹ for methane).

SUPPLEMENTARY MATERIAL

Details of the adsorption capacity for carbon dioxide and methane to the adsorbents that were impregnated with waste engine oil and kerosene are available electronically from <http://www.shd.org.rs/JSCS/>.

ИЗВОД
ИСПИТИВАЊЕ АДСОРПЦИЈЕ УГЉЕН-ДИОКСИДА И МЕТАНА НА СИРОВИМ
УГЉЕНИЧНИМ МОЛЕКУЛСКИМ СИТИМА И МОДИФИКОВАНИМ ПОМОЋУ
ИСТРОШЕНОГ МОТОРНОГ УЉА

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Угљенично молекулско сито (CMS) је синтетизовано из љуске ораха, уз накнадну физичку активацију угљеничне компоненте. Испитана је адсорпција CO₂ и CH₄ на сировом адсорбенсу, као и на адсорбенсу третираном киселином, коришћењем две величине, од 300–600 μm и 600–1180 μm. Адсорбенси су импрегнирани истрошеним моторним уљем од керозена са разређивачем у односу 1:1 на 25 °C. Највиша вредност адсорпције CO₂ и CH₄ добијена је при погодној величини адсорбенса из групе (A), при чему су се боље показали они третирани киселином него сирови. Киселином третирани узорак (A-3) адсорбовао је 0,925 mol CO₂ g⁻¹ и 0,353 mol CH₄ g⁻¹. Према резултатима, смањење величине гранулације групе (R), адсорпциони капацитети CO₂ и CH₄ су се смањили, док је повећање гранулације код групе (A) увећало адсорпциони капацитет за CO₂ and CH₄. Штавише, киселином третирани адсорбенси увећали су адсорпциони капацитет за CO₂ and CH₄. Даља модификација умањила је адсорпциони капацитет код импрегнираних адсорбенса, због умањивања расположиве површине, запремине и величине пора адсорбенса.

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