Experimental study of CO₂ adsorption using activated carbon

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Abstract

The current study aimed to investigate the CO₂ adsorption isotherms of activated carbon (AC), prepared from olive waste via chemical activation by H₃PO₄. The textural parameters, the microstructural and morphological characterization of the AC were characterized by N₂ adsorption-desorption, SEM and XRD. The AC presented the best textural properties with a BET surface area of 1565 m² g⁻¹ and a micropore volume of 0.649 cm³ g⁻¹. The CO₂ adsorption was measured with a volumetric method at three various temperatures: 298 K, 308 K and 318 K. Different models, such as the Langmuir and Freundlich isotherms, were applied to model the CO₂ adsorption. Based on the correlation coefficient (R²), the Langmuir model provided a the best fit with the experimental data. Different thermodynamic functions, such as Gibbs free energy and entropy, were calculated using Van Hoff’s formulation. These thermodynamic parameters indicated that the CO₂ adsorption is an exothermic and spontaneous process. The results obtained in this study can serve as a benchmark while designing an adsorption-based gas storage system.

1. Introduction

CO₂ is classified as a greenhouse gas and a cause of climate change [1]. The International Panel on Climate Change (IPCC) estimates that the global carbon dioxide concentration level may rise to 390 ppmv, causing an increase in global average temperature of about 1.9 °C [2]. The CO₂ capture and storage (CCS) is one of the predominant strategies used to reduce carbon dioxide emissions in industrial sector. Various techniques have been studied to sequestrate CO₂ from fuel gases, such as adsorption [2], absorption [3] and membrane separation [4]. The adsorption process is considered more promising for capturing CO₂ in the context of CCS technologies [5]. Many solid adsorbents can be used for CO₂ capture, such as clay, zeolites, activated carbon and mesoporous silica. Among all these adsorbents, activated carbon is considered the most efficient due to its high porosity, large specific surface and high adsorption capacity [6]. The adsorption mechanism on AC has been used for separation and purification of gases and gas storage systems [6]. The development of these systems requires the determination of the adsorption isotherms and the thermodynamic parameters of the adsorbate-adsorbent. Consequently, several studies have investigated the CO₂ adsorption on different types of AC. Marta Sevilla, et al (2011) [7] showed that the chemical activation of porous carbon improves the textural parameters and increases the amount of adsorbed CO₂. Soodabeh Khalili, et al (2016) [8] compared CO₂ and CH₄ adsorption on AC. Their experimental data showed that the process of CO₂ adsorption is longer than that of CH₄. Activated carbon’s microporosity was measured by Maia, et al (2018) [9], who evaluated the adsorption isotherms and thermodynamic parameters by Tian-Calvet microcalorimeter. Skander Jribi, et al (2017) [10] performed CO₂ adsorption on activated carbon with a magnetic suspension balance and found useful for evaluating the performance of CO₂ adsorption mechanism. The CO₂ isotherm in activated carbon was measured by Vinod Kumar Singh, et al (2017) [11]. The results of the thermodynamic parameters showed that the CO₂ absorption on AC was a physisorption phenomenon. Tao Song, et al (2015) [12] found a maximum
efficiency of CO$_2$ adsorption on AC caused by chemical agents. Heidari, et al (2014) [13] proved that an activation agent affects the micropore volume and the surface, resulting in a high capture of CO$_2$. Zhang, et al (2010) [14] compared the CO$_2$ adsorption of zeolite 13X and activated carbon under different temperatures. Their results demonstrated that the amounts of CO$_2$ on the activated carbon were lower than that on the zeolites. Singh et al (2019) [15] compared the adsorption of CO$_2$ on AC using two activating agents. The results showed that the AC modified with K$_2$CO$_3$ presents a higher adsorption capacity compared with the AC treated with NaOH and NaNH$_2$. The effect of KOH and the impact of temperature on CO$_2$ adsorption on commercial activated carbon were studied by et al [16]. Kaur al (2019) [17] used the chemical activation of activated carbon for CO$_2$ capture. The results demonstrated that the adsorption process is exothermic.

The majority of studies that evaluated the CO$_2$ adsorption used commercial AC synthesized from non-renewable precursors [13, 18–22]. Khalili et al [23] concurred that AC should be formulated from renewable sources to guarantee the long-term sustainability of this industry. The use of renewable wastes, such as agro-industrial wastes, to generate AC is achievable since it overcomes the present problem of depletion of non-renewable precursors utilized for commercial AC production. In addition, the utilization of waste materials as raw precursors is advantageous due to the probability of reduced production cost, since such wastes are widely abundant in the country and can be freely acquired, or merely incur low amounts of money.

Based on the above-mentioned studies, olive waste was used as a raw material, in the current study, to prepare activated carbon using phosphoric acid (H$_3$PO$_4$) as an activation agent. The CO$_2$ adsorption isotherms in formulated AC were measured volumetrically at various temperatures (298, 310 and 320 K), and the experimental data were modelled with the Langmuir and Freundlich methods. The thermodynamic parameters of the CO$_2$ adsorption were used to describe the adsorption process.

2. Materials and methods

2.1. Preparation of activated carbon

Olive waste was used as a raw material to produce activated carbon. The acid activation of AC was carried out by H$_3$PO$_4$ according to Soleimani, et al (2008) [24] with some modifications. The olive waste was impregnated with 85% H$_3$PO$_4$ with a weight ratio of 1:1, and the mixture was heated in an oven at 100 °C for 24 h. The activated acid was then, washed with distilled water and NaHCO$_3$, respectively, to neutralize its acidity. Before drying at 600 °C, the sample was washed again with distilled water.

2.2. CO$_2$ capture capacity

The adsorption experiment was carried out through a Nova 4200e (Quantachrome) static volumetric analyser. Before the adsorption analysis was conducted, about 50 mg of the sample was degassed at 150 °C for several hours. The adsorption was then, measured at different temperatures: 25 °C, 37 °C and 47 °C.

2.3. Characterization

Various techniques were utilized to characterize the AC and to obtain useful information about its structure. The morphology was analysed by SEM with a FEI Q250 Thermo Fisher microscope. The functional group of adsorbents was evaluated by FTIR spectrometer (Bruker Vertex 70–RAM II). Using a Micromeritics 3-Flex analyser, the textural parameters and the isotherms of the AC were measured at 77 K and a relative pressure range of 0.1–0.99. In addition, the specific surface area was obtained from the N2 isotherm with the Brunauer–Emmett–Teller (BET) method. The mesopore size distribution was calculated with the Barrett–Joyner–Halenda (BJH) method. The Raman analysis was recorded on a Thermo-Fisher DXR 2 spectrometer; with a laser wavelength of 532 nm from a He-Ne laser at room temperature.

3. Modelling of adsorption isotherms

Different models were used to fit the experimental adsorption isotherms, such as Langmuir and Freundlich. According to the following equation (1), the Langmuir isotherm was employed to describe the monolayer adsorption into the homogeneous surface [25]:

\[ q = q_m \frac{kp}{1 + kp}, \]  

Where \( q_m \) is the maximum quantity of CO$_2$ adsorbed into activated carbon, \( k \) is a constant that describes the difference between the heat of adsorption at the first layer and the following layers, and \( p \) is the saturation pressure of CO$_2$. 

The Freundlich isotherm was used for multilayer adsorption and surface heterogeneity. This isotherm was expressed according to the following equation [26]:

\[
q = kP^{1/n}.
\]

(2)

4. Results and discussion

4.1. Chemical analysis

Table 1 sums up the analysis of the activated carbon. The sample had an elemental composition of about 56.4% carbon (C), 35.8% oxygen (O), 7.3% hydrogen (H), 0.4% nitrogen (N) and 0.1% sulphur (S). The high carbon and oxygen content in the activated carbon can be attributed to the decomposition of volatile compounds during the carbonization process.

| Element     | Composition (wt %) |
|-------------|--------------------|
| Carbon      | 56.4               |
| Oxygen      | 35.8               |
| Hydrogen    | 7.3                |
| Nitrogen    | 0.4                |
| Sulphur     | 0.1                |

4.2. XRD spectra analysis

The XRD diffraction of activated carbon is presented in Figure 1. The graph shows two broad peaks at 2θ of about 25° and 42°. The two peaks refer to (002) and (101) planes of crystalline hexagonal graphite lattice respectively (JCPDScardno.41-1487) [27, 28].

The whole surface of AC is constituted of graphite crystallites that decrease in scale, which can lead to enlargement or disordered internal structure, forming a larger specific surface area. The AC is composed of ultrafine particles with a porous structure. The lower the crystallinity, the higher the specific surface area.

4.3. Raman analysis

The Raman spectrum is implemented to provide information on the rotational energy or molecular vibration of the sample, indicating the degree of structural disorder [29]. Figure 2 shows the Raman spectrum of AC. The sample presents two peaks around 1350 cm\(^{-1}\) (D band) and 1596 cm\(^{-1}\) (G band). The D-band at the lower Raman shift is assigned to the disordered carbon (D-band) such as non-crystalline and defective structures. The G-band at the higher Raman shift is affiliated to the order–layered with sp2 hybrid carbon atom vibrations. Additionally, the intensity of the G-band peak is higher than the D-band peak, reflecting some order of graphitization [17].
4.4. Infrared spectra

The FTIR spectra used to understand the functional groups present in the activated carbon is illustrated in figure 3. The spectra showed an absorption band around 3400 cm\(^{-1}\) which can be attributed to the hydroxyl groups (\(-\text{OH}\)). The sharp bands at 2400 cm\(^{-1}\) and 1722 cm\(^{-1}\) are ascribed to the carboxylic acid [30]. The bands at 2900 cm\(^{-1}\) and 1590 cm\(^{-1}\) correspond to the C–H stretching vibration in the methyl group [31] and the C–C aromatic stretch bond, respectively. The wide band at 1100 cm\(^{-1}\) is assigned to the stretching mode of bonded hydrogen [32]. The two bands at 1510 and 1425 cm\(^{-1}\) are appointed to the C=C vibrations in aromatic rings, and the band observed at 1246 cm\(^{-1}\) can be affiliated to esters or phenol groups. The C–H bending in benzene vibrations appears at 895 cm\(^{-1}\).

4.5. SEM analysis

Figure 4 shows the SEM micrographs of the AC at different magnifications. The sample presents smooth areas characterized by a roughness structure resembling a series of parallel lines. The rough surface of the AC can be identified by a distinct roughness with oval patterns. The presence of the macropores was clearly noticeable within each oval section,. The AC gave rougher textures with heterogeneous surfaces and a greater variety of randomly distributed pores size.
4.6. Adsorption-desorption isotherm and BET surface area

Figure 5 presents the N2 isotherm and the pores size distributions of the adsorbent. The N2 isotherm shows that the studied AC can be classified as a type I isotherm [33]. According to the IUPAC classification, the adsorbent is essentially formed by micropores with a very narrow microporosity. The most adsorption of N2 happened in a relatively low pressure region due to the existence of available sites in the structure of the adsorbent [32]. The adsorbed gas increased slightly and reached an horizontal plateau at a higher pressure due to the occupation of sites by gas molecules.

The BJH pores size distribution in activated carbon is shown in figure 6. The BJH method reveals that the pores are narrowly distributed in AC and mainly composed of micropores. Table 2 summarizes the textural parameters of the AC. The results show that the evaluated sample has a significant porosity, with high
values of total pore volume $V_{\text{total}}$ ($0.676 \text{ cm}^3 \text{ g}^{-1}$), surface area $S_{\text{BET}}$ ($1565 \text{ m}^2 \text{ g}^{-1}$) and micropore volume ($0.649 \text{ cm}^3 \text{ g}^{-1}$).

### 4.7. Adsorption isotherms

Figure 7 demonstrates the CO$_2$ adsorption profile in activated carbon at several temperatures. The CO$_2$ adsorption capacity decreased with the increase of temperature due to the reduction in binding strength between the AC and the CO$_2$. The interaction between the adsorbent and the adsorbate occurs at different forces such as Van der Waals forces, dipole-dipole interactions and chemical bonding. At the beginning of the adsorption mechanism and at high temperatures, a large number of vacant pores are available on the surface of the AC and
The adsorbate enters in direct contact with the adsorbent and creates stronger Van der Waals forces between the adsorbent and the adsorbate. Consequently, the adsorption values decrease with increasing temperature. The decline observed in the amount of adsorbed CO₂ at high temperatures reflects that the adsorption mechanism is exothermic, with an occurrence of physisorption [34]. Furthermore, the CO₂ adsorption increased at a low pressure due to the presence of available sites for adsorption. In contrast, the adsorbed CO₂ decreased at a higher pressure given that most of the sites were occupied.

The experimental results of CO₂ adsorption in AC were fitted with the Langmuir and Freundlich models. The landmarks depict the experimental data while the solid lines are used to indicate the isothermal models employed in this study. Table 3 summarizes the correlation coefficients (R) and the predicted isotherm constants related to all isotherms. According to this table, the Langmuir model produced a good association fit, with R² values of about 0.99. As shown, the Langmuir constant (k_L) and Freundlich constant (k_F) decreased at high temperatures, proving physisorption behaviour. The decrease of the CO₂ adsorption capacity is related to the ‘Chatelier principle’, which indicates that endothermic desorption is favourable at raised temperatures.

Based on the Langmuir equation, the maximum CO₂ adsorption capacities (q_m) decreased with temperature; confirming the exothermic characteristic of adsorption. The nature of this process was defined from the separation factor (R_L) calculated by the following equation [16]:

$$R_L = \frac{1}{1 + K_L C_0},$$

where K_L and C_0 represent the Langmuir constant and the initial concentration, respectively. According to the value of R_L, the nature of adsorption was linear, favourable, unfavourable or irreversible [16]. As shown in

![Figure 7. CO₂ adsorption isotherms with (a) Langmuir model, (b) Freundlich model on activated carbon at 298, 310 and 320 K.](image)
Figure 8, the value of $R_L$ was within the range of 0 to 1, indicating that the adsorption process is favourable. The favorability of CO$_2$ adsorption can also be given by the Freundlich constant $'n'$. The $n$ values found in the current study oscillate between 1-2, proving the physisorption process. Moreover, the values of $1/n$ were less than 1 at different temperatures, providing a good adsorption intensity [18].

4.8. The thermodynamics of adsorption

The thermodynamic parameters of the adsorption process are standard Gibbs $\Delta G^\circ$, the enthalpy of reaction $\Delta H^\circ$ and the entropy $\Delta S^\circ$ [22]. Using equation (3), $\Delta H^\circ$ and $\Delta S^\circ$ were evaluated from the slope and intercepted by plotting $\ln(b)$ versus inverse temperature $(1/T)$ (figure 9). However, $\Delta G^\circ$ was calculated using equations (4) and (5).

$$\ln(b) = \left( \frac{\Delta S^\circ}{R} \right) - \left( \frac{\Delta H^\circ}{RT} \right)$$

(3)

$$\Delta G^\circ = -RT \ln(b)$$

(4)

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ$$

(5)

Where $R$ is the universal gas constant and $T$ is the temperature.

The thermodynamic parameters are shown in table 4. The $\Delta H^\circ$ value for the adsorbent was 3.57 kJ mol$^{-1}$. This suggests that the CO$_2$ adsorption was exothermic and physisorption in nature. Moreover, the positive value of $\Delta S^\circ$ indicates an expansion in disorder through the adsorption process. The sign of $\Delta G^\circ$ was negative, proving the spontaneous characteristic of the adsorption. To note, the $\Delta G^\circ$ values expanded with the increase of temperature, demonstrating that the adsorption phenomenon is favourable at lower temperatures.
The present work showed that the natural AC prepared from olive wastes has a comparable or considerably higher adsorption capacity compared with other types of AC (e.g., Norit X2, commercial AC; Algae; table 5). The textural characteristics and the microporous structures are found to play a dominant effect on CO2 adsorption at ambient temperature (physisorption) [35]. The microporous structure of the AC prepared from olive wastes allows a higher CO2 capture compared with mesoporous AC such as Algae and commercial AC. Nonetheless, according to table 5, the higher CO2 uptake permitted by bean grains despite the mesoporous structures are due to their physicochemical characteristics, which are mostly constituted of nitrogen, which enhances the CO2 adsorption through an acid-base interaction [18].

To note, several studies have been recently focused on using agricultural wastes as source of carbon such as palm kernel shell (PKS) [35], eggshell (ACES) [36] and modified palm kernel shell (CaO/PKS) [37]. The high void fraction and porous structure of these AC strongly support their adsorption behaviour. The large BET surface area of these AC (776.4 m² g⁻¹ for CaO/ACPKS), (273 m² g⁻¹ for ACES), 476.7 m² g⁻¹ for PKS) enhances the adsorption efficiency but it still lower than that of AC produced from olive waste (1565 m² g⁻¹).

**Table 4.** Thermodynamic parameters ΔH°, ΔS° and ΔG° for CO2 adsorption.

| Adsorbent | ΔH° (kJ mol⁻¹) | ΔS° (kJ mol⁻¹ K⁻¹) | ΔG° (kJ mol⁻¹) 298 K | ΔG° (kJ mol⁻¹) 303 K | ΔG° (kJ mol⁻¹) 318 K |
|-----------|----------------|-------------------|----------------------|----------------------|----------------------|
| Norit SX2 (peat) | 3.57 | 0.03 | -5.37 | -5.73 | -6.03 |

**Table 5.** Comparison of the CO2 adsorption capacity with different activated carbon.

| Adsorbent         | Adsorption capacity (mmol g⁻¹) | Porosity   | References |
|-------------------|-------------------------------|------------|------------|
| Norit SX2 (peat)  | 1.88                          | mesoporous | [18]       |
| Algae             | 1.39                          | mesoporous | [19]       |
| Bean dregs        | 4.24                          | mesoporous | [20]       |
| Coconut AC        | 1.79                          | microporous| [38]       |
| Commercial AC     | 1.5                           | mesoporous | [13]       |
| Our study         | 1.85                          | microporous|             |

5. Comparison with other activated carbons

The present work showed that the natural AC prepared from olive wastes has a comparable or considerably higher adsorption capacity compared with other types of AC (e.g., Norit X2, commercial AC; Algae; table 5). The textural characteristics and the microporous structures are found to play a dominant effect on CO2 adsorption at ambient temperature (physisorption) [35]. The microporous structure of the AC prepared from olive wastes allows a higher CO2 capture compared with mesoporous AC such as Algae and commercial AC. Nonetheless, according to table 5, the higher CO2 uptake permitted by bean grains despite the mesoporous structures are due to their physicochemical characteristics, which are mostly constituted of nitrogen, which enhances the CO2 adsorption through an acid-base interaction [18].

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6. Regenerability of activated carbon adsorbents

The capacity, the selectivity and the regeneration of adsorbents are necessary factors in practical industries. In our study, the cyclic adsorption-desorption performance of activated carbon was inspected to check its stability. As shown in figure 10, the AC presented an admirable cyclic CO₂ adsorption-desorption performance when both adsorption and desorption steps were applied executed.

7. Conclusion

The purpose of this paper was analyzing the mechanism of CO₂ adsorption in activated carbon. The AC was prepared from olive waste, activated by H₃PO₄ and characterized via SEM, XRD and N₂ adsorption-desorption method. The AC presented the best textural properties with a BET surface area of 1565 m² g⁻¹ and a micropore volume of 0.649 cm³ g⁻¹. The adsorption capacity of carbon dioxide was studied at three temperatures 298 K, 308 K and 318 K. The AC presented a good CO₂ adsorption capacity of around 1.85 mmol g⁻¹ CO₂ at 298 K compared with other adsorbents. To fit the experimental isotherm of CO₂ absorption, different models were used, such as the Langmuir and Freundlich models. The results showed that the Langmuir model gave the best fit to the practical results, with a regression coefficient (R²) of about 0.99. The thermodynamic parameters, such as standard Gibbs (ΔG°), enthalpy (ΔH°) and entropy (ΔS°), were calculated from the experimental data. These parameters proved that the CO₂ adsorption is spontaneous, exothermic and physisorption in nature.

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Data availability statement

No new data were created or analysed in this study.

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