



Efficient adsorption of carbon dioxide and methane on activated carbon prepared from glycerol with potassium acetate

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Abstract

In the context of global warming and the energy crisis, emissions to the atmosphere of greenhouse gases such as carbon dioxide (CO₂) and methane (CH₄) should be reduced, and biomethane from landfill biogas should be recycled. For this, there is a need for affordable technologies to capture carbon dioxide, such as adsorption of biogas on activated carbon produced from industrial wastes. Here we converted glycerol, a largely available by-product from biodiesel production, into activated carbon with the first use of potassium acetate as an activating agent. We studied adsorption of CO₂ and CH₄ on activated carbon. The results show that activated carbon adsorb CO₂ up to 20% activated carbon weight at 250 kPa, and 9% at atmospheric pressure. This is explained by high specific surface areas up to 1115 m²g⁻¹. Moreover, selectivity values up to 10.6 are observed for the separation of CO₂/CH₄. We also found that the equivalent CO₂ emissions from activated carbon synthesis are easily neutralized by their use, even in a small biogas production unit.

Keywords Glycerol · Activated carbons · Carbon dioxide · Methane · Adsorption

Introduction

The year 2022 was the year of the United Nations Ocean Conference that remind us that global mean sea levels increased at an average of 4.5 mm per year between 2013 and 2021, due to ice sheets melting at an increasing rate and also that ocean absorbing nearly 23% of CO₂ generated by human activity, and when it does, chemical reactions take place, acidifying the seawater. This puts marine environments at risk and, the more acidic the seawater becomes, the less CO₂ it can absorb (UN 2022). Besides attempts to avoid CO₂ formation, carbon capture and storage and carbon capture and utilization technologies have been developed to mitigate the effects of CO₂ in the atmosphere (Mikulčič et al. 2019; Rodin et al. 2020).

Besides CO₂, CH₄ also has a marked greenhouse effect, in fact, the effect of CH₄ is considered to have 84 times the warming power of CO₂ in 20 years' timescale (EDF

2022). This makes nearly 25% of the present global warming driven by CH₄. Biogas, particularly landfill gas, is composed of a mixture of gases but the major components are CH₄ (45–60%) and CO₂ (40–55%) (Jaramillo and Matthews 2005). In some developing countries, landfills are the single largest source of man-made CH₄ emissions. Therefore, purifying landfill gases to obtain high grade CH₄ for energy producing, for example to use in vehicles, is the best alternative to avoid its release into the atmosphere. Additionally, recent events in world geopolitics have highlighted the importance of both, the judicious use and the local production of CH₄.

Among the possibilities for CO₂ capture or separation, adsorption on porous materials emerges as an important technology since the separation of CO₂ is the most energy consuming step, resulting in a high cost (Haszeldine 2009). Various types of porous materials were studied for the capture and separation of CO₂, as reviewed elsewhere (Kumar et al. 2020; Karimi et al. 2022). Between the various families of adsorbents, activated carbons due to abundant sources, hydrophobic character, and lower heat of adsorption are considered excellent candidates (Karimi et al. 2022). Furthermore, since activated carbons can be obtained from biomass their use can give a contribution to net-zero emissions and/or, when obtained from industrial wastes, their use can contribute to the circular economy (Karimi et al. 2022). In this way,

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one of the byproducts formed during biodiesel production is glycerol, 10–20% of the total volume (Quispe et al. 2013).

Glycerol is a low added product whose accumulation is causing environmental issues. Previous studies with activated carbons from glycerol concerned mainly liquid-phase applications and catalysis (Batista, 2022). Here we show that it is possible to prepare activated carbons adsorbents from glycerol with adequate properties for using in gas-phase applications, namely in CO₂ capture and in the CO₂/CH₄ separation of landfill gases.

Experimental

Materials preparation

The chemical activation of the char was made by the acid carbonization of glycerol. Various chemical activation agents can be used such as KOH as reviewed elsewhere (Gao et al. 2020). Nevertheless, the materials prepared with KOH also have low density which is not the best characteristic for gas adsorption and, additionally, KOH is corrosive and not environmental friendly (Zhang et al. 2019). Therefore, in the present work, the chemical activation agent used was CH₃COOK (Aldrich, 99%) since this specie is reported to improve the density of the activated carbon and also to promote the formation of surface oxygen species (Zhang et al. 2019). The detailed experimental description of the activation process for sample preparation is given in Supporting Information—Section S1. The activated carbons (AC) were identified by the mass ratio of char:activating agent. Three mass ratios were used 1:1, 1:0.75 and 1:0.5, accordingly, the samples were labeled AC_ratio_1:1, AC_ratio_1:0.75 and AC_ratio_1:0.5, respectively.

Materials characterization

Activated carbons were characterized by evaluating the surface area, pore size distribution and porous volumes by nitrogen adsorption at −196 °C; Chemical analysis; determination of the pH at the point of zero charge-pH_{pzc}, Infra-red spectroscopy; X-ray diffraction; Thermogravimetry and Apparent tap density. The methodologies for the characterization of the samples by each one of the above-mentioned techniques are described in detail in Supporting Information—Section S2.

CO₂ and CH₄ adsorption

The adsorption of CO₂ and CH₄ was measured at 25 °C and up to 300 kPa in a custom-made volumetric apparatus, constructed in stainless steel and equipped with a pressure transducer and a vacuum system that allows a vacuum better than

10^{−2} Pa. This apparatus was previously described in detail in the literature (Pires et al. 2014). During experiments, the temperature was kept constant with a thermostatic water bath. Before every experiment, the samples were outgassed at 300 °C for 2 h.

Results and discussion

Physical characterization

The yield in the preparation of the char was 30%. On the basis of the mass of the char, the yield of the activation process was between 58 and 67% for the samples AC_ratio_1:1, and AC_ratio_1:0.5, respectively. The materials are amorphous as denoted by the broad peaks in the X-ray diffractograms—Supporting Information, Section S3 where the diffractions from the (002) and (001) plans attributed to the disorder graphite near 25 and 43 = 2θ°, respectively, are noticed (Joshi et al. 2021). An estimation of the crystallinity can be made by a method described in the literature (Zuo et al. 2009). This method gives crystallinity values of 11, 10 and 5% for AC_ratio_1:1, AC_ratio_1:0.75 and AC_ratio_1:0.5, respectively.

The tap density of the activated carbons (Table 1) decreases as the proportion of the activating agent (CH₃COOK) increases, as a consequence of a more developed porosity. The tap-density values for the activated carbons in Table 1 are higher than those reported when KOH is used as an activating agent for the same type or for different types of chars which has important implications since low density adsorbents are difficult to handle in practice (Batista et al. 2022; Li et al. 2019).

The amounts adsorbed (n_{ads}) versus the relative pressure p/p^0 , where p^0 is the saturation pressure of nitrogen at −196 °C, are given in Fig. 1a These isotherms are characteristic of microporous materials, as confirmed also by the micropore-size distributions from the density-functional

Table 1 Relation of mass to tap volume (tap density); Brunauer–Emmett–Teller (BET) specific surface area (A_{BET}) and microporous volumes (V_{micro}). Activated carbons (AC) with higher density are favored as adsorbents but the density decreases increasing the ratio of the activating agent. Additionally, high surface areas and porous volumes are desirable and these improve with the increase of the ratio of the activating agent which was CH₃COOK

Sample	Tap density (g cm ^{−3})	A_{BET} (m ² g ^{−1})	V_{micro} (cm ³ g ^{−1})
Char	0.64	< 10	–
AC_ratio_1:0.5	0.74	155	0.06
AC_ratio_1:0.75	0.63	663	0.25
AC_ratio_1:1	0.58	1115	0.42

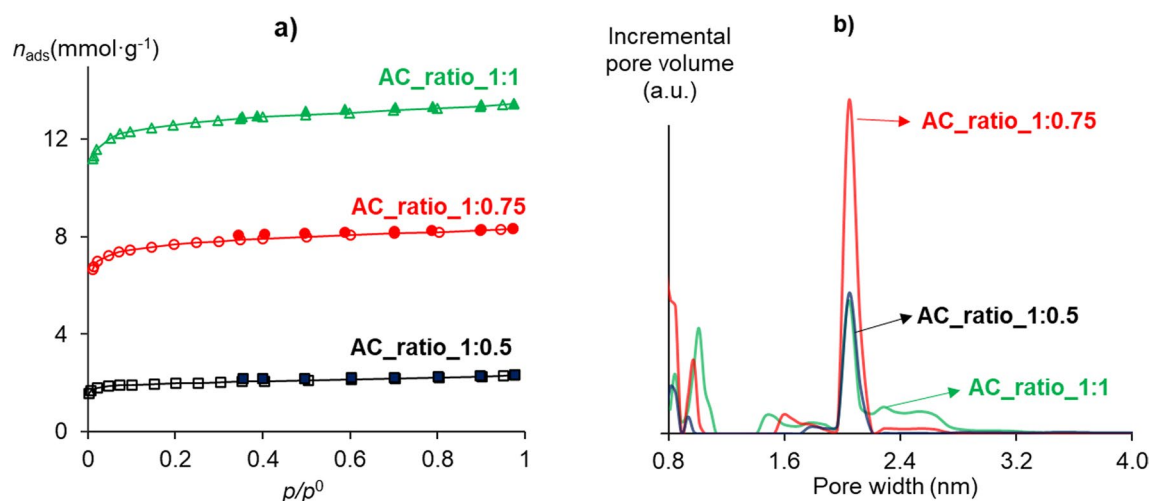


Fig. 1 **a** Nitrogen adsorption isotherms at $-196\text{ }^{\circ}\text{C}$, where the closed points are desorption points, and **b** pore size distributions for the prepared activated carbons (a.u. stands for arbitrary units). The samples

theory—Fig. 1b. The micropore-size distributions are bimodal, more details on the micropore volumes in Supporting Information—Section S2.

Specific surface areas and micropore volumes are given in Table 1. The char has only a residual surface area and does not present microporosity. The specific surface area, increases from 155 to $1115\text{ m}^2\text{ g}^{-1}$ for AC_ratio_1:0.5 and AC_ratio_1:1, respectively, due to the increase in the proportion of the activating agent. This increase is also reflected in the microporous volumes and the bimodal micropore-size distributions are common for chemically activated carbons (Gauden et al. 2007). It was shown that a char from glycerol waste can be activated with a CH_3COOK , producing materials with high surface area and density but using a more sustainable and less corrosive substance than other more common activating agents (Batista et al. 2022).

Chemical characterization

As denoted by the pH_{pzc} values in Table 2, the materials have a surface of acidic nature, usually related to oxygen surface species.

The surface chemistry of the materials was also studied by FTIR. The spectra are presented in Supporting Information—Figure S4, where the band near 3400 cm^{-1} is due to the O–H stretching and the bands in the region $2900\text{--}2800\text{ cm}^{-1}$ are attributable to asymmetric and symmetric C–H stretching vibrations in aliphatic $-\text{CH}$, $-\text{CH}_2$ and $-\text{CH}_3$ and those at 1620 cm^{-1} to a highly conjugated hydrogen bonded C=O (Pradhan and Sande 1999; Ryu et al. 2002). The band near 1400 cm^{-1} is attributable to the asymmetric stretching of S=O and also to oxygenated carbon species of $-\text{COOH}$ type and the broadband centered at

present highly rectangular isotherms, denoting developed microporosity, and the micropore-size distributions are bimodal with the majority of micropores having widths near 2 nm

Table 2 pH at point of zero charge (pH_{pzc}) and chemical composition (C, H, S and O in %). The % of O was estimated from: $\text{O} (\%) = 100 - \text{C} (\%) - \text{H} (\%) - \text{S} (\%)$. The characterization of oxygen and sulfur groups in the adsorbents is important because these will interact differently with the CO_2 and CH_4 molecules since, although both molecules have relatively similar polarizability values CO_2 , contrarily to CH_4 , has a significative quadrupole moment. These different interactions influence the selectivity of the adsorbents for the CO_2/CH_4 separation

Sample	pH_{pzc}	C (%)	H (%)	S (%)	O (%)
Char	1.84	64.18	3.76	12.55	19.03
AC_ratio_1:0.5	2.08	77.02	1.92	7.01	14.05
AC_ratio_1:0.75	2.09	78.49	1.79	6.62	13.10
AC_ratio_1:1	2.13	73.85	1.67	1.93	22.55

1100 cm^{-1} to various types of oxygenated carbon species such as alcohol, phenol and ether bridges between rings (Shin et al. 1997). All these oxygen and sulfur groups can influence the CO_2 and CH_4 adsorption.

CO_2 and CH_4 adsorption

The adsorption isotherms of CO_2 and CH_4 at $25\text{ }^{\circ}\text{C}$ in the activated carbons are presented in Fig. 2 a, b, respectively. For the same pressures, the amounts adsorbed are higher for CO_2 than for CH_4 as is usually the case for activated carbons and other types of materials. This is in-line with the physical properties of CO_2 and CH_4 , namely the higher polarizability and permanent quadrupole of CO_2 that enhance its interactions with the surface, and the differences in the critical temperature of both molecules, which is higher for CO_2 (Poomisitiporn et al. 2016).

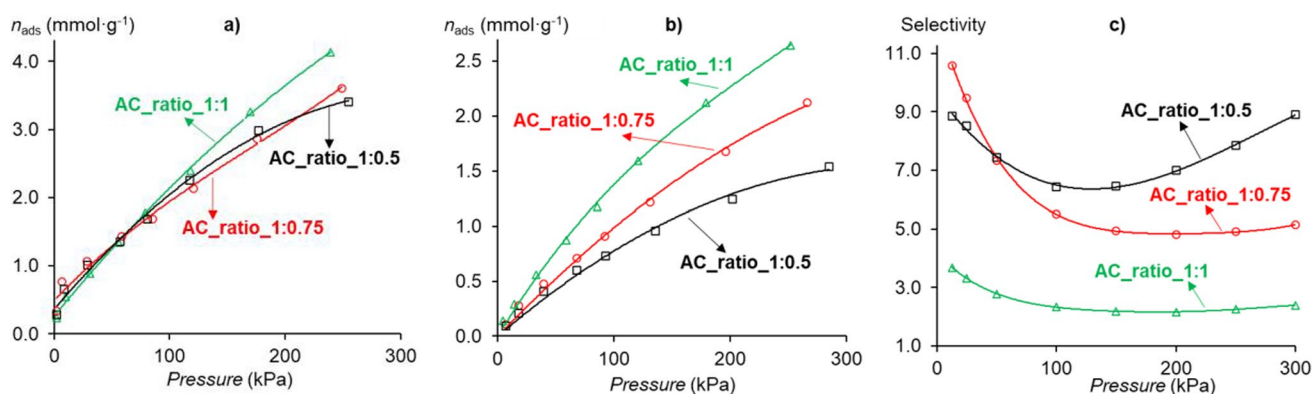


Fig. 2 Isotherms of adsorption at 25 °C **a** CO₂ and **b** CH₄. The amounts adsorbed of CH₄ are in-line with the specific surface area, but this is only partially the case for CO₂, due to the additional effect of the surface chemistry of the materials. The selectivity val-

ues, estimated from the Ideal Adsorbed Solution Theory, in function of the pressure are also presented in **c**. The activated carbons AC_ratio_1:0.5 and AC_ratio_1:0.75, present high selectivity values

The sequence in the amounts adsorbed in Fig. 1 is in-line with the specific surface areas of the materials in the case of CH₄ but this is only partially the case for CO₂, particularly for the lowest pressures where the effect of the surface chemistry of the materials is more noticed. There is a relatively high range of values in the literature for the amounts adsorbed of CO₂ in activated carbons for conditions of pressure and temperature similar to those used in this work. Nevertheless, for instance in activated carbons prepared from biomass/biochar, the values for CO₂ adsorption range from 0.65 to 3.5 mmol g⁻¹—Table S6, indicating that the values in the present work approach the upper limit of this range, which is also similar to values found for commercial activated carbons (Karimi et al. 2022). In the case of CH₄ the amounts adsorbed are lower than those found for most of the commercial activated carbons as reviewed in the literature, although that review was centered on materials for CH₄ capture and not for the CO₂/CH₄ separation (Choi et al. 2016). The amounts of CH₄ adsorbed in Fig. 2b are, however, comparable to those found in activated carbons prepared from industrial wastes or from carbon fibers (Alcañiz-Monge et al. 2009). The materials can be regenerated and reused as illustrated with the case of CO₂ adsorption in the AC_ratio_1:0.75 material—Supporting Information, Section S5.

The selectivity of the prepared activated carbons was estimated from the ideal adsorbed solution theory (IAST) and details are given in Supporting Information—S7, Simon (2016). The selectivity in function of the pressure—Fig. 2c shows that the values are always higher for the materials obtained with less amount of activating agent. In fact, except for the lowest pressures, the AC_ratio_1:0.5 material presents the highest selectivity values. Even if the reasons for this result are not entirely clear, we might consider that the explanation relies essentially on the differences in the CH₄ adsorption. In fact, if we consider the CO₂ adsorption

(Fig. 2a) the isotherms are not strongly different from which other, due to the combined effects of porosity and surface chemistry. Contrarily, the isotherms of CH₄ (Fig. 2b) are considerably separated, and seem therefore to be more dependent on the porosity itself since, as above-mentioned CH₄ adsorption is much less susceptible to the changes in the surface chemistry. In Supporting Information—Table S6 literature values of selectivity for the CO₂/CH₄ system, obtained with activated carbons in experimental conditions comparable to the present work and similar methodologies of calculation, are given. From that table, and comparing with the results in Fig. 2c, it can be concluded that the selectivity values from this work, particularly for the AC_ratio_1:0.5 and AC_ratio_1:0.75 activated carbons, that show values between 6.4 and 10.6, are close to the upper range of the values found in the literature. It should be noticed that high values of selectivity are desirable but commercial processes exist for which the selectivity values are higher than 3 (Yang 2003). The significance of the equilibrium gas and adsorbed phase composition diagrams was discussed in the literature (Ruthven 1984). These diagrams, as predicted by the IAST, are given in Supporting Information—S8.

It is important to clarify the energy used and the respective equivalent CO₂ emissions (CO₂-eq) created in the formation of the adsorbent. This has been addressed detailed in the literature for various types of activated carbons (Lima et al. 2008; Hjaila et al. 2013; Alhashimi and Aktas 2017). One of the most conservative studies includes the energy used in all the steps, from the residues transportation to the preparation and drying of the char and the activated carbon, the total energy being 167 × 10³ MJ (47 × 10³ kWh) to produce 1 ton of activated carbon (Hjaila et al. 2013; Alhashimi and Aktas 2017). This amount of energy, assuming that all comes from fossil fuels, corresponds to 20 tons of CO₂-eq per ton of activated carbon (EPA 2022). If we consider even

a small biogas production unit (500 m³/day) that can deliver 88 kg of CH₄/hour per ton of adsorbent, and that this CH₄ is equivalent to 7.4 tons CO₂-eq/h, we conclude that the 20 tons of CO₂-eq produced in the preparation of a ton of adsorbent are compensated in a few hours of use, confirming the importance of avoiding CH₄ release to the atmosphere (Santos 2011; EDF 2022). With the same rationale, the regeneration/recycling of the adsorbent has only the relatively marginal impact of 1.2 ton CO₂-eq/t of adsorbent and it is easily neutralized (Alhashimi and Aktas 2017). In this way, the regenerated adsorbent can be used both for the CO₂/CH₄ separation or for CO₂ capture even if, by itself, its use only in CO₂ capture would not neutralize the CO₂-eq produced in the preparation of the adsorbent.

Conclusion

In this work it was shown that starting from glycerol, an available low added by-product from the biodiesel industry, and by using potassium acetate as an activating agent, activated carbons can be obtained with porosity and surface chemistry characteristics that make them valuable both, for CO₂/CH₄ separation with selectivity values up to 10.6 and for CO₂ adsorption, with amounts up to 3.6 mmol g⁻¹ at 298 K and 200 kPa. Additionally, the CO₂ emissions released during the manufacture of the adsorbent are largely compensated right after the first use cycle of the CO₂/CH₄ separation.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s10311-022-01556-0>.

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Author contributions BM experiments, report, data analysis; MB planning, experiments, data analysis, writing JP planning, data analysis, writing.

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Declarations

Conflict of interest The authors declare no competing financial interest.

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