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Storage and separation of methane and carbon dioxide using platinum-decorated activated carbons treated with ammonia

Mohamed F Aly Aboud1,2,∗, Zeid A ALOthman1 and Abdulaziz A Bagabas4,∗

1 Sustainable Energy Technologies Center, College of Engineering, King Saud University, PO Box 800, Riyadh 11421, Saudi Arabia
2 Mining, Metallurgical and Petroleum Engineering Department, Faculty of Engineering, Al- Azhar University, Nasr City, Cairo, 11371, Egypt
3 Advanced Materials Research Chair, Chemistry Department, PO Box 2455, College of Science, King Saud University, Riyadh 11451, Saudi Arabia
4 National Petrochemical Technology Center (NPTC), Materials Science Research Institute (MSRI), King Abdulaziz City for Science and Technology (KACST), P. O. Box 6086, Riyadh 11442, Saudi Arabia

∗ Authors to whom any correspondence should be addressed.

E-mail: maboud@ksu.edu.sa, zaothman@ksu.edu.sa and abagabas@hotmail.com

Abstract

Activated carbon (AC) was treated with ammonia for nitrogen doping and then was further anchored with platinum nanoparticles through ultrasound-assisted impregnation method. Methane (CH₄) and carbon dioxide (CO₂) storage of the pristine and modified activated carbon were determined at ambient temperature. All the samples showed high storage capacities with preference towards carbon dioxide storage, which would be favorable for both energy and environmental applications. Ammonia treatment slightly improved the storage of both CH₄ and CO₂ for all the samples, which could be attributed to the little improvement in the micropore properties upon nitrogen doping. Platinum decoration had an opposite influence on both CH₄ and CO₂ storage, which might be due to the absence of metal encouraging storage mechanism such as gas dissociation or reconstruction on platinum’s surface in addition to the harmful effect of metal pore-blocking and higher density of metal particles.

Introduction

Natural gas, mainly composed of methane (CH₄) greater than 90%, became an alternative fuel due to its high resources that make it more economical compared to other fuels, especially with its high H/C ratio. In addition, it can be formed from renewable biomass resources [1]. The U.S. Department of Energy had set a goal in 2015 of 150 v/v that was revised to 180 v/v at 35 bar (the volume of gas at STP - 298 K and 0.1 MPa - per volume of the storage vessel and equivalent to 35 wt.% at 35 bar [2, 3]. Carbon dioxide (CO₂) may be present in biogas with a range between (25%–50%), based on the type of bio-digestion process and its efficiency [4]. Removal of carbon dioxide from biogas may enrich the methane percentage up to 90%, which increases its calorific value and will have a positive effect on its energy conversion [5].

Methane storage and distribution are usually performed under high pressure between 200–2450 bar or by using liquefaction techniques. Both of the previous techniques involve high cost due to the high energy requirements. In particular, for expensive multi-stage compression facilities, the single compression stage is limited to 500 Psi [6]. Furthermore, safety issues due to the special requirement for the storage tanks, especially if applied in-vehicle technology [7]. Chemical adsorption of methane over the surface area, as methane hydrates [8], is an alternative methane storage method with theoretical storage values that can reach up to 174 v/v. The problem with chemical adsorption storage, which makes this technique not suitable commercially, arises from the slow adsorption kinetics in addition to the difficulty of releasing methane back by reducing the pressure.

Carbon dioxide capture and storage (CCS) technologies are critical to reduce pollution and global warming and its removal from air is critical for the environment [9, 10]. Three technologies are used for the CO₂ capture:
oxy-fuel combustion, calcium looping, and amine solvents. However, they undergo different limitations such as high cost and usage of toxic materials with low stability [11].

Physical adsorption (physiosorption) storage method for both of methane and carbon dioxide on highly porous materials such as zeolites, metal-organic frameworks (MOFs), and activated carbons offer a lot of advantages that eliminate the disadvantage of other storage methods [12]. Activated carbon (AC) is among the strong candidates for gas storage because of their high stability, non-toxicity, small densities, recycling characteristics, low cost, and abundance [13–18]. Physiosorption within the high surface area adsorbents arises when an adsorbent contains pores that raise the potential field of force (Van der Waals) from their walls, causing interaction energy (adsorption) between the gas molecules and the adsorbent surface. The main factor that limits the gas storage on the adsorbent surface is the low energy of adsorption for most of the porous carbons [19].

Modifications of the graphitic skeleton may increase the gas uptake by introducing strong interaction sites on the carbonaceous surfaces or alter the chemical nature of carbonaceous matrix to form binding sites or centers on the carbon skeleton for gas capturing [20–36]. Introduction of nitrogen within the activated carbon was reported [37–39] to enhance the carbon dioxide storage capacity via the creation of strong electrostatic and non-electrostatic interactions with carbon dioxide. The presence of platinum within nitrogen modified activated carbon was demonstrated to decrease the nitrogen positive effect on carbon dioxide storage capacity due to the chemical interaction between platinum and nitrogen [40]. In this context, we examined Norit activated carbon and its modified forms by nitrogen, platinum, or by both of nitrogen and platinum for storage of carbon dioxide and methane greenhouse gases. We correlated the findings with the textural properties and the impact of the modifier.

### Experimental section

Activated carbon (Norit RX L5 Extra) from (CABOT) was used as the matrix and support for the decorated metals. The used metal precursor was hexachloroplatinic acid (H$_2$PtCl$_6$.xH$_2$O, 99%, WinLab, Wilfrid Smith, 39.96% min.). The activated carbons were divided into two groups: (i) first group was activated carbons, doped with nitrogen by treating them with ammonia (NH$_3$) gas at 850 °C and (ii) the second group was left without treatment. We followed the method of Mangun and his co-workers [41] who showed that treating of activated carbon with ammonia, an etching reagent within the skeleton of the activated carbon, was very successful method to introduce nitrogen within the activated carbons, where the textural properties increased with increasing temperature treatment from 400 °C to 800 °C. However, we used 850 °C, as NH$_3$-treatment temperature, due to a technical limitation in our furnace.

The ultrasound-assisted impregnation method was used to anchor 5.0 wt.% of platinum metal particles on the treated and the untreated activated carbons surfaces because of its simplicity and its large scale [42–45]. De-ionized water was used to dissolve the metal precursor and then was dispersed on the activated carbon (treated and non-treated) by using an ultrasonic bath to nominally form a 5.0 wt.% of metal-anchored carbonaceous materials. The mixture was left several hours on a hot plate under a hood with repetitive stirring for homogenization. The resulting metal salt- decorated AC was reduced under a hydrogen atmosphere at 350 °C for two hours.

The structure was investigated via x-ray diffractometry (XRD) by using a Rigaku MiniFlex 600, operated at 40 kV and 15 mA with CuK$_\alpha$ radiation. Sample morphology was studied with Secondary and LABE (low angle backscattered electron) scattering modes by using a field-emission scanning electron microscope (JEOL JSM-7600F), equipped with INCAx-act, oxford instrument for energy-dispersive x-ray spectroscopy (EDX). The textural properties were determined from nitrogen isothersms at 77 K by using a Micromeritics Gemini VII, 2390 analyzer, where each sample (∼ 0.2 g) was preliminarily degassed at 200 °C for 12 h. The specific surface area was estimated through different methods, but the present results and discussion relayed on the Brunauer–Emmett–Teller (BET) method. The total pore volume is based on the amount of gas adsorbed at P/P$_0$ = 0.9, while the micropore volume was derived from the Dubinin–Radushkevich (DR) equation. The elemental analysis for carbon, hydrogen, nitrogen, and sulfur (CHNS) was performed on ∼ 10 mg of the sample by using a PerkinElmer 2400 Series instrument. The methane and carbon dioxide storage measurements were performed by using the volumetric technique (Sievert’s method) by using high-pressure volumetric analyzer HPVVAII (Micromeritics) device that can reach up to 200 bars. The methane and carbon dioxide storage capacity was evaluated by measuring the sorption isotherms [pressure-composition-isotherms (PCI)] at 298 K up to 100 bars for methane and 5 bars for carbon dioxide, which was restricted to this low pressure value due to the instrument settings for carbon dioxide gas. The weight of the sample was at least 2.0 g to decrease the experimental errors as possible. Before any sorption measurements, the samples were degassed under vacuum at 200 °C for 12 h. Weight losses were measured after degassing and the degassed mass was used as the dry mass for the sorption process. The sample volume correction was based on skeletal density measurements corresponded to the excess values.
obtained by helium gas. Ultra-high purity grade (6N) gases were used in all the experiments. The storage measurements were repeated several times for validity and to check the hysteresis effect. All samples showed good repeatability. All the reported storage capacities were excess gas adsorption quantities and refer to the sample dry mass (i.e., degassed mass). The excess adsorption is defined as the difference in the amounts of gas-phase that will be present in the equivalent volume of the adsorbed phase in the presence and absence of adsorption [46].

Figure 1. X-ray diffraction (XRD) pattern of platinum-impregnated, ammonia-treated, activated carbon (Norit).

Figure 2. Scanning electron micrograph of platinum-impregnated, ammonia-treated activated carbon (Norit).
Results and discussion

Material characterization

Figure 1 displays the x-ray diffraction (XRD) pattern of the ammonia-treated activated carbon, anchored with platinum nanoparticles. The activated carbon showed no diffraction peaks due to its poor crystallinity. Sharp diffraction peaks were detected at 2θ° of 39.7° (111), 46.3° (200), 67.5° (220), 81.3° (311) and 85.6° (222), related to the occurrence of the fcc structure of platinum in agreement with the JCPDS card No. 03-065-2868.

Figure 2 shows the backscattered scanning electron micrograph of the ammonia-treated activated carbon, anchored with platinum nanoparticles. It revealed a layered structure of activated carbons with ∼75 nm average size sphere-shaped platinum nanoparticles, homogeneously spread over it. The corresponding EDX spectrum is shown in figure 3, where the Pt content was found to be ∼5.0 wt%, which was consistent with the value calculated, based on the metal salt addition. The nitrogen signal was not observed because its quantity was below the detection limit of the detector. According to the CHNS analysis, the nitrogen content was 2.0 wt.%.

Textural characterization

Figure 4 shows the nitrogen adsorption isotherms at 77 K of various activated carbon samples. The isotherms showed Type I, an indication of the microporous materials. Ammonia treatment did not affect the micropore properties, as confirmed by the stability of the micropore volume to around 0.4 cm³ g⁻¹ in addition to the small alteration in the micropore area for all the samples before and after treatment. On the other hand, the treatment enhanced the mesopores and macropores area, as verified by the enhancement of the total BET surface area for the ammonia-treated samples. The decorated samples showed lower surface areas due to the metal pore-
Table 1. Textural properties of pristine and ammonia-treated activated carbon (Norit) and both were decorated with Pt.

| Sample                                      | (BET) Surface area, (m² g⁻¹) | (t-plot) Micropore area (m² g⁻¹) | Total pore volume (cm³ g⁻¹) | Micropore volume (cm³ g⁻¹) |
|---------------------------------------------|------------------------------|----------------------------------|----------------------------|---------------------------|
| Activated carbon (Norit)                    | 1513 ± 20                    | 976.6 ± 10                       | 0.79 ± 0.1                 | 0.46 ± 0.1                |
| Activated carbon (Norit) treated with NH₃  | 1649 ± 20                    | 997.4 ± 10                       | 0.85 ± 0.1                 | 0.47 ± 0.1                |
| Activated carbon (Norit) decorated with Pt | 1473 ± 20                    | 906.1 ± 10                       | 0.79 ± 0.1                 | 0.43 ± 0.1                |
| Activated carbon (Norit) treated with NH₃ and decorated with Pt | 1508 ± 20                    | 914.5 ± 10                       | 0.79 ± 0.1                 | 0.43 ± 0.1                |
Figure 5. PCI (methane adsorption and desorption) at 25 °C of pristine and ammonia-treated activated carbon (Norit) and both were decorated with Pt. (a) Excess weight, (b) Excess Volume.

Table 2. Excess methane storage wt% at 298 K of the pristine and ammonia-treated activated carbon (Norit) and both were decorated with Pt.

| Sample                                           | Methane maximum adsorption (Wt.%) at R.T |
|--------------------------------------------------|------------------------------------------|
| Activated carbon (Norit).                        | 12.3 ± 0.1                               |
| Activated carbon (Norit) treated with ammonia.   | 12.6 ± 0.1                               |
| Activated carbon (Norit) and decorated with Pt.  | 11.3 ± 0.1                               |
| Activated carbon (Norit) treated with ammonia and decorated with Pt. | 11.7 ± 0.1 |

Figure 6. PCI (carbon dioxide adsorption and desorption) at 25 °C of pristine and ammonia treated activated carbon (Norit).
blocking effect, a negative effect of platinum metal decoration on activated carbons was also reported by Stadie et al.\cite{47}.

**Methane storage measurements**

Figure 5 (a) (Excess weight) and 5 b (Excess volume) show the methane adsorption and desorption at 298 K for the pristine and modified activated carbon samples. The PCI isotherms of methane indicated Langmuir Type I of IUPAC, an indication of monolayer adsorption and symptom of microporous materials. The methane capacity increased with increasing the pressure until it reached saturation around 50 bar.

The excess methane storage capacities in wt.% at both \( \sim 35 \) bar and at saturation pressure are summarized in table 2.

Ammonia treatment showed a slight improvement of methane storage of around 3.0 wt% for the nitrogen-doped samples. The slight enhancement in the methane storage could be attributed to the little improvement in the micropore properties after ammonia treatment. Deleterious impact on methane storage upon metal decoration could be due to metal pore-blocking and the higher density of the platinum-anchored activated carbon in comparison with the non-decorated sample.

Furthermore, the presence of platinum nanoparticles did not improve the methane storage because its dissociation on metallic surfaces, which facilitates its diffusion within the adsorbent pore structure, was rendered by its high activation barrier\cite{48}. In addition, the symmetry of the methane molecule would not allow for any dipole or quadrupole moments on metallic surfaces\cite{7}.

The highest saturated storage capacity of methane was achieved for the non-decorated, nitrogen-doped activated carbon sample with a value of 14.3 wt.\%, which was equivalent to 8.9 mmol g\(^{-1}\). It is in agreement with the linear relation reported by Sun et al.\cite{49} that relates the methane adsorption with the surface area where a value of 6 mmol g\(^{-1}\) was corresponding to a surface area of 1500 m\(^2\) g\(^{-1}\). Also, our methane storage values were comparable to the values reported by Policicchio et al.\cite{50} for their sample (HSCP 19) with a higher surface area of 1648 m\(^2\) g\(^{-1}\) and higher micropores volume of 0.6 cm\(^3\) g\(^{-1}\). Our methane storage values were also 7 orders of magnitude higher than the values reported by Kumar et al.\cite{51}. We showed methane adsorption values higher than those reported for activated carbons with adsorption capacities ranging between 4.9 wt. \% to 11.5 wt. \% reported by Yang et al.\cite{52} and also higher than the values of 10.8 wt.\% and 10.9 wt.\% reported by Quinn et al.\cite{6} for activated carbons with higher surface areas. Additionally, our methane storage values are higher than the 8.9 g g\(^{-1}\) wt.\% and 8.0 g g\(^{-1}\) wt.\% values reported for Metal-Organic Frameworks (COF-5) and (COF-10) samples with the corresponding surface area of 1670 m\(^2\) g\(^{-1}\) and 1760 m\(^2\) g\(^{-1}\), respectively, reported by Yagi et al.\cite{53}.

The Excess volumetric storage of the various samples is shown in figure 5(b). The volumetric storage of methane values are in the range of 100 c.c./g to 120 c.c./g at 10 bar and saturates to 160 c.c./g to 200 c.c./g around 50 bar. They are little higher than the reported methane storage values of 84.3 c.c./g and 96.5 c.c./g at 10
Table 3. Excess carbon dioxide storage wt.% at 298 K and 5.0 bar of pristine and ammonia treated Activated carbon (Norit) and both were decorated with Pt.

| Sample                                      | Carbon dioxide Excess capacity (wt.%) at R.T and 5.0 bar |
|---------------------------------------------|--------------------------------------------------------|
| Activated carbon (Norit).                   | 26.2 ± 0.1                                             |
| Activated carbon (Norit) treated with ammonia. | 27.7 ± 0.1                                             |
| Activated carbon (Norit) and decorated with Pt. | 25.2 ± 0.1                                             |
| Activated carbon (Norit) treated with ammonia and decorated with Pt. | 26.1 ± 0.1                                             |

Table 4. Carbon dioxide Excess capacity (wt.%) at room temperature for different activated carbons that went through different activation procedures and produced from different raw materials.

| Materials                  | Surface area (m² g⁻¹) | CO₂ uptake (mmol g⁻¹) | Reference |
|----------------------------|-----------------------|-----------------------|-----------|
| Activated carbon fibers    | 1247                  | 3.19                  | [60]      |
| Activated carbon           | 1320                  | 3.1                   | [61]      |
| Activated carbon           | 1445                  | 5.7                   | [62]      |
| Activated carbon           | 340                   | 6.0                   | [63]      |
| Activated carbon           | 2510                  | 5.2                   | [64]      |
| Activated carbon           | 1479                  | 5.5                   | [65]      |
| Activated carbon           | 418                   | 2.4                   | [66]      |

The carbon dioxide storage values lie within the reported values for methane storage for many activated carbons with similar textural properties reported by Park et al [56]. Hysteresis was observed for all the various samples which may be attributed to retaining some methane within the very narrow pore dimensions as cushion gases [6].

Carbon dioxide storage measurements

Figures 6 and 7 show the PCI isotherms for carbon dioxide adsorption and desorption of our various activated carbon samples at room temperature. The storage capacity of carbon dioxide progressively increases with increasing the pressure up to 5 bar. The sorption shape indicates type I isotherms symptom of microporous materials and monolayer adsorption.

Ammonia treatment showed slight improvement of carbon dioxide storage of around 5% for both decorated and non-decorated samples. The slight enhancement in the carbon dioxide storage could be attributed to the little improvement in the micropore properties upon ammonia treatment where the nitrogen effect could be marginal due its low content of ~2 wt.% [37].

Adverse influence on carbon dioxide storage upon metal decoration was found which may be attributed to the counter-effect of metal pore-blocking and its chemical interaction with nitrogen [40].

The storage capacity of carbon dioxide of our various activated carbon is ranging between 25.2 to 27.7 wt.% at around 5 bar that is equivalent to 5.7–6.3 mmol g⁻¹. Table 3 shows carbon dioxide excess capacity (wt.%) for our samples at room temperature and up to pressure around 5.0 bar.

Our modified and non-modified samples showed an average carbon dioxide storage value of 26 wt.% which is greater than their corresponding methane storage values that was in the range of 12 wt.%. The higher adsorption capacity of carbon dioxide compared to methane adsorption capacity will offer higher selectivity for CO₂ gas capture than CH₄ gas capture. Such preferentiality will propose big advantage when purifying methane from carbon dioxide and subsequently would increase the biogas calorific value. Additionally, improvement of CH₄ recovery can be achieved by injection of CO₂ into our Activated carbons, if they were used as methane adsorbents, to remove the retaining methane as cushion trapped gas [6, 57].

The carbon dioxide average storage value for our various Activated carbons is around ~ 6 mmol g⁻¹ which is higher than the 5 moles kg⁻¹ reported by Pelleranoa et al [58] of activated carbons with surface textural properties of 1468 m² g⁻¹ and micropore volume of 0.56 cm³ g⁻¹. Our carbon dioxide storage capacity was equal to the storage capacity of the Activated carbons used as preferential adsorption for CO₂ from CH₄/CO₂ mixture reported by Pino et al [59]. Our carbon dioxide storage values are greater than carbon dioxide storage values of a wide diversity of Activated carbons with same textural properties or even better as shown in Table 4.
Also, our metal decorated samples showed superior performance compared with the carbon dioxide adsorption values reported by Madzaki et al.\cite{67} who showed that decoration of activated carbon with cerium oxide was in the range of 0.856 mmol g$^{-1}$. In all the samples no hysteresis was observed which confirm the total reversibility of the stored carbon dioxide upon releasing the applied pressure at ambient temperatures.

**Conclusion**

High storage capacities of both methane and carbon dioxide storage were achieved for modified and non-modified activated carbons with extra preference towards carbon dioxide storage. This preferentiality could be used for biogas purification in addition to reducing pollution and global warming.

Ammonia treatment did not affect the micropore properties considerably. On the other hand, it enhanced the overall textural properties. Ammonia treatment showed a slight improvement of methane and carbon dioxide storage for all the samples that could be attributed to the little improvement in the micropore properties upon nitrogen doping.

Slight deleterious impact on both methane and carbon dioxide storage was found upon platinum decoration which may be endorsed to the absence of metal encouraging storage mechanism and the counter-effect of metal pore-blocking and the higher density of the platinum-anchored activated carbon compared with the non-decorated ones. The results of this study validate that improving the micropore properties is the main route to achieve a remarkable enhancement in the storage of both methane and carbon dioxide in carbonaceous materials via physisorption storage method.

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**Author contributions**

Mohamed F. Aly Aboud conceived and designed the experiments, performed all experiments and all data analysis; Mohamed F. Aly Aboud wrote the paper; Zeid A. Alothman participated in the methane and carbon dioxide storage measurements, data analysis, paper writing and corrections; Abdulaziz A. Bagabas participated in materials synthesis, the textural characterization, paper writing and corrections. All authors examined and approved the final manuscript.

**Conflicts of interest**

The authors declare no conflict of interest.

**ORCID iDs**

Mohamed F Aly Aboud [https://orcid.org/0000-0001-7434-0233](https://orcid.org/0000-0001-7434-0233)

Zeid A Alothman [https://orcid.org/0000-0001-9970-2480](https://orcid.org/0000-0001-9970-2480)

Abdulaziz A Bagabas [https://orcid.org/0000-0003-3590-7369](https://orcid.org/0000-0003-3590-7369)

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