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Efficient CO$_2$ adsorption using mesoporous carbons from biowastes

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Abstract

The mesoporous carbon nanomaterials obtained from waste onion peels confirmed the formation of mesoporous carbon nanospheres (CNSs) with an average pore diameter of 2 nm. The as obtained CNSs were studied to demonstrate their ability to adsorb the greenhouse gas CO$_2$ at different temperatures, 30, 40 and 50 °C. The results showed that the CNSs have high potential to adsorb CO$_2$ at these temperatures. Present work demonstrates the formation of sustainable, porous adsorbents by a simple waste to wealth based approach that is effective for the adsorption of CO$_2$.

Introduction

An uncontrolled, exponential increase in the level of CO$_2$ is considered to be the most serious potential threat as it creates severe environmental damages [1]. Currently, the prime priority of entire scientific community is to alleviate the unfettered release of CO$_2$ and to balance its concentration in atmosphere. This has evoked an interest in the scientific community to undertake all possible efforts to separate and capture the greenhouse gas using various techniques [2–4]. The usual techniques involve membrane separation [5], liquid solvent absorption/ionic liquids [6, 7], cryogenic approaches [8, 9], and adsorption on solid sorbents [10, 11].

Of various separation techniques, adsorption of CO$_2$ over solid sorbents, especially porous adsorbents has received considerable attention in terms of energy efficiency and cost effectiveness. Accordingly, the porous materials zeolites, silica, carbonaceous materials, molecular sieves, metal organic frameworks, nanoporous polymers, etc [12–14] have been investigated for their efficiency as potential CO$_2$ adsorbents. In recent years, the carbon-based materials having porous nature are extensively recommended as they exhibit diverse attractive features as listed; the pores in carbon materials ensure faster diffusion and extremely high permeability gas molecules compared to other solid sorbents [15]; possess inherent affinity towards CO$_2$ [16]; provide structural stability over a large number of cycles with insensitive to moisture [17]; require less energy for regeneration [18]; display tunable textural properties [17, 19]; and ensure low cost [20, 21].

Though, the porous carbon structures synthesized from chemical substrates have been earlier used as sorbents, the major challenge encountered is cost. Therefore, a low cost, stable and environmentally benign material is in quest. In this regard, the recent focus is driven on valorization of biomass/bio–waste materials to sustainable carbon nanomaterials that can serve the purpose [22–28]. It proposes an inexpensive method due to abundant availability of precursors and a simple procedure (one-step pyrolysis) of carbon nanomaterial synthesis such as carbonization [23], ammoxidation [24], hydrothermal [25, 26, 29], KOH activation [27], calcination [28], and pyrolysis [30]. Moreover, they offer large surface area and high pore volume which facilitates gas adsorption. Few reports are available, in this regard, where bio-derived carbonaceous materials have shown excellent ability to uptake CO$_2$, and the results are comparable with that of commercial adsorbents. Elisa et al reported the preparation of carbon materials from biomass. The sorption studies evaluated 1.3 mmol g$^{-1}$ of CO$_2$ at 25 °C [31]. In a similar study, the activated carbon produced from waste oil fly ash was used as CO$_2$ adsorbent by Saad et al [32]. The carbon material was used to capture CO$_2$ (74.5 mg g$^{-1}$) from a mixture of N$_2$ and CO$_2$, and the chemisorbed amount of the gas increased over a temperature range, 0–40 °C. Further, nitrogen doped carbon materials have been used as excellent
CO₂ sorbents [33–36] because the hydrogen bonding between the surface of carbon and CO₂ molecule is considered to facilitate adsorption process. A nitrogen doped activated carbon material is synthesized by Xing et al [37], using bean dreg of soya bean milk. These activated carbon materials were capable of adsorbing CO₂ up to 4.24 mmol g⁻¹. Similarly, the KOH activated porous carbon material obtained from celtuce leaves had an adsorption capacity of 6.04 and 4.36 mmol g⁻¹ at 0 and 25 °C, respectively [38].

The biomass derived carbon structures used as potential adsorbents, reported so far, demanded surface activation subsequent to their synthesis. In recent years, many reports were emphasizing the catalyst free, non-activated mesoporous carbons from various biowastes [39–43].

In this work, we have investigated the CO₂ adsorption capacity of CNSs derived from commonly produced bio-waste, onion peels. The detailed procedure of CNSs synthesis from onion peels by one-step pyrolysis has been reported earlier [17].

Methods

Sample preparation

Biowaste onion peel, the precursor used in this work was collected from market areas nearby. The waste material was washed thoroughly under tap water and completely dried in oven at 80 °C for 2 days. The parched waste was ground to fine powder and carried further for pyrolysis at different temperatures from 500 °C to 900 °C for one hour. The set temperature value was reached at a rate of 10 °C min⁻¹ and this process was carried out under nitrogen atmosphere at gas flow rate of 150 ml cm⁻². After pyrolysis, the carbonized material was rinsed with dil. HCl, followed by washing with distilled water and dried at 80 °C in oven for overnight [17]. The technique involved no usage of catalysts and activation agents, thereby producing CNSs by one-pot, environmental friendly procedure. Detail procedure is given in our earlier reports [17].

Characterization

As mentioned all the characterizations concerning mesoporous shape and size were already reported in our earlier paper [17] and here we repeat some experiments for the sake of clarity. The pyrolyzed products were analyzed by various characterization techniques. The spherical morphology of CNSs were captured using field emission scanning electron microscopy (FESEM, JEOL JSM-7100F) operated at 15 kV and elemental composition was demonstrated by energy dispersive x-ray spectroscopy (EDS). X-Ray diffraction (XRD) patterns were studied from 10° to 60° using PANalytical-x-ray. The Raman spectroscopic analyses of CNSs were performed by New Xplora Plus V1.2 multiline, HORIBA Jobin Yvon confocal Raman spectroscope. N₂ adsorption-desorption experiments were performed to study the textural properties of CNSs surface area and porosity were demonstrated using BELSORP-max, Microtrac, Japan. CO₂ studies were also carried out using BELSORP-max, Microtrac, Japan. CO₂ adsorption and N₂ adsorption values were compared for the better understanding of the structures.

Results and discussion

Bio-waste derived CNSs

The biowaste was pyrolyzed at different temperatures such as 500, 600, 700, 800 and 900 °C; the corresponding products were coded as AC5, AC6, AC7, AC8, and AC9. The features of carbonized products were studied in detail by various characterization techniques. [17]. The outcome of the analyses have been discussed in supplementary (figures S1–S4 is available online at stacks.iop.org/MRX/7/015605/mmedia). The results of CNSs obtained by pyrolysis at 900 °C are mentioned here for illustration purpose.

The pyrolysis temperature was found to have an impact on overall features of the products formed. For the sake of understanding, the SEM and TEM images of AC9 are given in figures 1(a) and (b), respectively and figure 1(c) indicates corresponding histogram. The two prominent peaks appeared in Raman spectroscopic plot (figure 1(d)) at 1350 cm⁻¹ (G band) and 1396 cm⁻¹ (D band) indicated graphitic nature of AC9 CNSs with some extent of disorderness, respectively. The XRD analysis graph in figure 1(e) corroborated with Raman spectroscopic result. The sharp peaks at ~29° and ~44° stipulated crystalline/graphitic feature of AC9. Other peaks correspond to traces of metals present in the carbon matrix. The figure 1(f) is BET plot showing large surface area of AC9 with impressive pore size distribution curve inset.

The graph, figure 2(a), illustrated a direct relationship between carbon content of CNSs with pyrolysis temperature. The volatile matters in bio-material were removed to maximum extent when heated at higher temperatures; as a result, percentage of carbon in CNSs increased in parallel with temperature. The graph also showed reduction in the size of particles from AC5 to AC9 (figure 2(a)). The surface properties of onion peel derived CNSs were analysed using BET technique. The N₂ adsorptio-desorption curves of AC5-AC9 material has been given in figure 2(b). The graph, figure 2(c) variation of surface area and pore volume in parallel with temperature.
Therefore, in the series, AC5 exhibited the lowest value, whereas AC9 showed the highest surface area and pore volume. Further, BET also confirmed the formation of mesoporous structures with a pore diameter ~2 nm.

**CO₂ adsorption studies**
The CNSs obtained by one-pot pyrolysis of onion peel waste showed excellent surface area and mesoporous nature. Therefore, they were predicted to have the capacity to capture greenhouse gas, CO₂ effectively, at their
surface. CO₂ adsorption was studied using all the materials (AC5-AC9) in series at different temperatures, 30, 40 and 50 °C under absolute pressure from 0 to 1 atm.

Initially, CO₂ adsorption study using biowaste derived CNSs was conducted at 30 °C. The amount of gas captured by each material has been shown in figure 3. As expected, at low temperature (30 °C) CNSs had not shown remarkable adsorption (0.74 mmol g⁻¹ for AC5), because of their poor morphology, order, and surface properties (surface area 5.4 m²g⁻¹; pore volume 0.00 cm³g⁻¹). With high surface area (207 m²g⁻¹ and 550 m²g⁻¹) and pore volume (0.2 cm³g⁻¹ and 0.4 cm³g⁻¹), AC6 and AC7 were found to be efficient sorbents for CO₂ with captured amount 1.43 mmol g⁻¹ and 1.68 mmol g⁻¹, respectively. This result is higher than the amount of CO₂ adsorbed (1.42 mmol g⁻¹) by mesoporous organosilica nanotubes prepared by Wei et al [44]. An unexpected decrease in the adsorption of CO₂ (1.59 mmol g⁻¹ to 1.57 mmol g⁻¹) was observed for AC8 to AC9 materials. Though, AC9 had the largest surface area (929.9 m²g⁻¹) and maximum pore volume (2.3 cm³g⁻¹) in the series, it exhibited less efficiency to capture CO₂ compared to AC7 and AC8 (table 1).

The CNSs, AC5–AC9 were subjected to CO₂ capture studies at slightly elevated temperature. A similar trend was observed at 40 °C, where the carbonized materials formed at lower temperature (500 °C) possessed less capacity to hold CO₂ (0.91 mmol g⁻¹) at their surface; and AC6-AC9 exhibited 1.00 mmol g⁻¹—1.40 mmol g⁻¹. In the series, AC7 exhibited the highest amount (table 1) of as adsorbed with a value of 1.53 mmol g⁻¹. CO₂ adsorption at 50 °C followed the same trend as that of 30 and 40 °C; from AC5 to AC7, the CNSs showed increment in CO₂ capture (0.79—1.34 mmol g⁻¹), which later decreased from AC8 to AC9 (1.32—1.17 mmol g⁻¹). The CO₂ adsorption studies at different temperatures (30, 40 and 50 °C) put light upon the effect of structural and textural properties of onion peel waste derived CNSs on the greenhouse gas adsorption capacity. The plots (figure 3) were evident that CO₂ adsorption increased from AC5 to AC7 and then followed a reduction trend, at all the three temperatures. When relooked at the Raman spectroscopic plot, the intensity of D band (correspond to disorder) and value of I_D/I_G ratio was increased ≥800 °C. Therefore, the increment in the disorder in CNS matrix could be claimed reason for this trend as structural disorder causes distortion of pore channels resulting in hindrance to CO₂ molecules to permeate through [45].

Moreover, during adsorption process, the gas molecules interact with the surface of solid materials; both the walls in small pores influence adsorption. Therefore, in porous CNSs (AC8–AC9) as pore diameter decreased, interactions between pore walls and gas molecules would be lesser resulting in low adsorption of CO₂. Further,
lower amount of CO₂ was adsorbed at 50 °C compared to that at 30 and 40 °C. The result indicated effect of system temperature on the sorption process. When temperature was raised, kinetic energy of the gas molecules elevated, resulting in their escape to bulk free space; consequently, density of the adsorbed phase decreases with increase in temperature [46–48].

Conclusions

Novel mesoporous CNSs synthesized from onion peel waste at a series of temperatures, 500 °C–900 °C by a catalyst free, one-step pyrolysis technique showed the ability to adsorb CO₂ efficiently. The BET studies were evident for the large surface area and mesoporous nature of the carbonized products; the study also showed optimization of surface properties with the increment in pyrolysis temperature. The as-obtained porous CNSs were tested for their efficiency as adsorbents for CO₂ at ambient and elevated temperatures. The isotherms determined the highest amount of gas adsorbed at 30 °C (1.68 mmol g⁻¹) and with the elevation in temperature (40 °C and 50 °C), the gas sorption decreased (1.53 and 1.34 mmol g⁻¹, respectively). Therefore, the study confirmed suitability of bio-waste derived porous CNSs as potential adsorbents for CO₂.

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Table 1. N₂ adsorption–desorption values and amount of CO₂ adsorbed by AC CNSs at different temperatures, 30, 40 and 50 °C.

| Samples | BET (m² g⁻¹) | dₚ (nm) | Vₚ (cm³ g⁻¹) | 30 °C | 40 °C | 50 °C |
|---------|--------------|---------|--------------|-------|-------|-------|
| AC 5    | 5.4          | 2.0     | 0.00         | 0.74  | 0.91  | 0.79  |
| AC 6    | 207.0        | 3.1     | 0.16         | 1.43  | 1.00  | 1.07  |
| AC 7    | 550.0        | 2.6     | 0.37         | 1.68  | 1.53  | 1.34  |
| AC 8    | 692.0        | 2.5     | 0.43         | 1.59  | 1.40  | 1.32  |
| AC 9    | 929.9        | 2.3     | 0.54         | 1.57  | 1.40  | 1.17  |

dₚ—average pore diameter; Vₚ—total pore volume.
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