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Short communication

Carbon dioxide capture on primary amine groups entrapped in activated carbon at low temperatures

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ABSTRACT

For purposes of selective CO₂ uptake under ambient conditions, a few number of primary amine groups are selected, including aminoclay (H₂N-CL), 3-aminopropyltriethoxysilane (APTES), and dopamine hydrochloride (DA). Coatings of both H₂N-CL into activated carbon (AC) show enhanced CO₂ adsorption capacity at 273 and 298 K and 1 atm, resulting in 3.069/1.950 mmol/g, compared to 2.872/1.824 mmol/g of pristine AC. However, APTES and DA coated AC at 273 and 298 K and 1 atm are reduced to 2.433/1.762 and 0.429/0.389 mmol/g. Particularly, the coating of H₂N-CL nanoparticles into AC exhibits enhanced selectivity of 8.8/18.7, compared to 7.6/15.9 in pristine AC for CO₂/N₂ at 273 and 298 K at 1 atm.

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Introduction

The emission of carbon dioxide (CO₂) (one of the greenhouse gases) into the atmosphere from large-scale anthropogenic point sources provides an impetus for research and development to reduce global warming and combat climate change [1–3]. Various methods for capturing and storing CO₂ have received substantial attention [4–6]; these methods are grouped mainly into the use of liquid amine and its derivative solutions (absorption), and impregnation of amine related materials into porous materials (solid adsorbents) on phase behavior focus [1]. Absorption processes are frequently employed in “wet-scrubbing” aqueous solutions, such as amine solutions and chilled ammonia, which are based on physical absorption [1]. In contrast, adsorption processes of capturing CO₂ from flue gas on solid media (for example, zeolites, activated carbons, organic–inorganic hybrids, hydrotalcites, calcium oxides, and metal organic frameworks (MOFs)) are operated by weak physisorption or strong chemisorption interactions [3].

There has been a shift in recent approaches for absorption processes due to toxicity, corrosion of equipment containers, solvent loss related to vaporization, and high energy consumption. The impregnation of amine related materials into solid media for selective post- or pre-combustion CO₂ capture is considered a promising alternative technology; focus is now on both increased CO₂ adsorption capacity under equilibrium conditions, and on fast CO₂ adsorption kinetics [7]. The main classes of these solid amine sorbents lead to chemical bonding of amines to the matrices or impregnation of supports into representative amine solutions (polyethyleneimine (PEI) and tetraethylenepentamine (TEPA)), to offer uniform distribution of amines, resulting in enhanced CO₂ sorption performance when compared to the usage of bulk amines [8–12].

Activated carbon (AC) was selected for purposes of this study, from amongst porous materials that could be used as immobilization substrates for amine materials. AC was selected for its low cost of raw materials and for the good yield obtained because of well-developed porosity [13]. Three primary amine types that differ in terms of unit or molecular structure (Fig. S1) were employed in this study: (i) water-solubilized aminoclay (H₂N-CL; [H₂N(CH₂)₃]₈Si₈Mg₆O₁₂(OH)₄) nanoparticles [14,15], composed of a magnesium-centered octahedral sheet with sandwiching silica-based tetrahedral sheets; organo-functionality of –(CH₂)₃NH₂ in H₂N-CL is

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through active sites in aqueous solution; (ii) 3-aminopropyltriethoxysilane (APTES; $\text{H}_2\text{N}(\text{CH}_2)_3\text{Si}(\text{OC}_2\text{H}_5)_3$) [16], which is used as a precursor for aminoclay synthesis and (iii) dopamine hydrochloride (DA; $(\text{HO})_2\text{C}_6\text{H}_3(\text{CH}_2)_2\text{NH}_2\cdot\text{HCl}$) [17], used to dissolve CO_2 in water in bicarbonate-based biominerals of mussels. After these primary amine materials were entrapped in AC, they were comparatively studied in terms of CO_2 adsorption profiles at low temperatures (273 and 298 K).

Experimental

Materials

See Supplementary Information, Text S1.

Preparation of aminoclay

Aminoclay ($\text{H}_2\text{N-CL}$) was prepared, as per the literature [14,15]. In brief, 8.4 g (~ 0.04 mol) of $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$ were injected in ethanol solution (200 mL). After these were fully dissolved through 5 min of stirring, 13 mL (~ 0.06 mol) of APTES were added into the magnesium chloride ethanolic solution, and stirred magnetically overnight. The precipitated white product, called $\text{H}_2\text{N-CL}$, was harvested by centrifugation (4000 rpm, 10 min). The obtained $\text{H}_2\text{N-CL}$ was then washed twice with pure ethanol and oven-dried at 50 °C for 24 h, in order to allow residual ethanol to evaporate from the $\text{H}_2\text{N-CL}$. Prior to its use, the dried $\text{H}_2\text{N-CL}$ was powdered by grinding with a pestle and mortar, and a stock solution was subsequently prepared using DI water and sonication for 10 min.

Preparation of CO_2 solid adsorbents

20 wt% of activated carbon (AC) was prepared by dipping activated carbon in 30% ethanol solution, and stirring for 1 h at room temperature. The solution was treated for 20 min with ultrasound at 40 kHz (Ultrasonics UC-05, Lab Companion, Korea). The ultrasound-irradiated activated carbon was air-dried overnight (henceforth denoted as AC).

The ultrasound-irradiated activated carbon (5 g) was dispersed in 1000 mL $\text{H}_2\text{N-CL}$ solution (10 g $\text{H}_2\text{N-CL}$: 100 mL ethanol–400 mL DI water) and vigorously stirred for 30 min at room temperature. Ultrasound irradiation was applied for 30 min, with the electrical energy input maintained at 100 W/cm². The solution was freeze-dried to obtain aminoclay-activated carbon powder (denoted as $\text{H}_2\text{N-CL-AC}$).

The AC (5 g) was immersed in 0.1 M APTES solution for 30 min. The APTES coated sample was air-dried overnight (denoted as APTES-AC).

The dopamine solution used for coating was prepared by dissolving dopamine hydrochloride at a concentration of 2 mg/mL in 10 mM of Tris-HCl buffer at pH 8.5. The AC (5 g) was submerged in the dopamine solution immediately following creation of the solution, remaining submerged for a period of 24 h. The dopamine-coated samples were air-dried overnight (denoted as DA-AC).

Gas sorption test

Micropore and mesopore structures of adsorbents were investigated by N_2 adsorption and desorption at 77 K using a volumetric sorption analyzer (ASAP 2020, Micromeritics, USA); this enabled analysis of pore parameters including BET (Brunauer, Emmett & Teller) surface area, pore size, and pore volume. The adsorption isotherms of CO_2 and N_2 were measured at 273 K and 298 K. The heat of adsorption was also calculated using a volumetric sorption analyzer. The moisture and CO_2 were removed from the samples by degassing at 353 K before the adsorption tests were performed. After the pre-treatment, the variation in weight of the samples was recorded.

Characterization of as-prepared adsorbents

See Supplementary Information, Text S1.

Results and discussion

CO_2 adsorption of $\text{H}_2\text{N-CL}$ powder produced using a milling method did not display suitable adsorption behavior (data not shown) at room temperature because of difficulty in accessibility of active sites of amine groups within $\text{H}_2\text{N-CL}$ layers. In $\text{H}_2\text{N-CL}$ powder for CO_2 adsorption and desorption, Moura et al. reported that the best adsorption of CO_2 was resulted from 363 K [18]. However, in aqueous solution, delaminated $\text{H}_2\text{N-CL}$ sheets exist in a few layers or in a single layer, enabling the effective use of the primary amine groups, i.e., water-soluble nanoparticles [15]. Thus, immobilization of $\text{H}_2\text{N-CL}$ as nanoparticle form into AC in aqueous solution can be conducted, along with immobilization of other primary amine materials, allowing for comparisons under low temperature conditions. Up to date, although $\text{H}_2\text{N-CL}$ was utilized as carbonization agent to produce calcium carbonate (CaCO_3) by CO_2 capture [19], but it did not investigate direct CO_2 capture at ambient condition yet. Fig. 1 shows the CO_2 adsorption

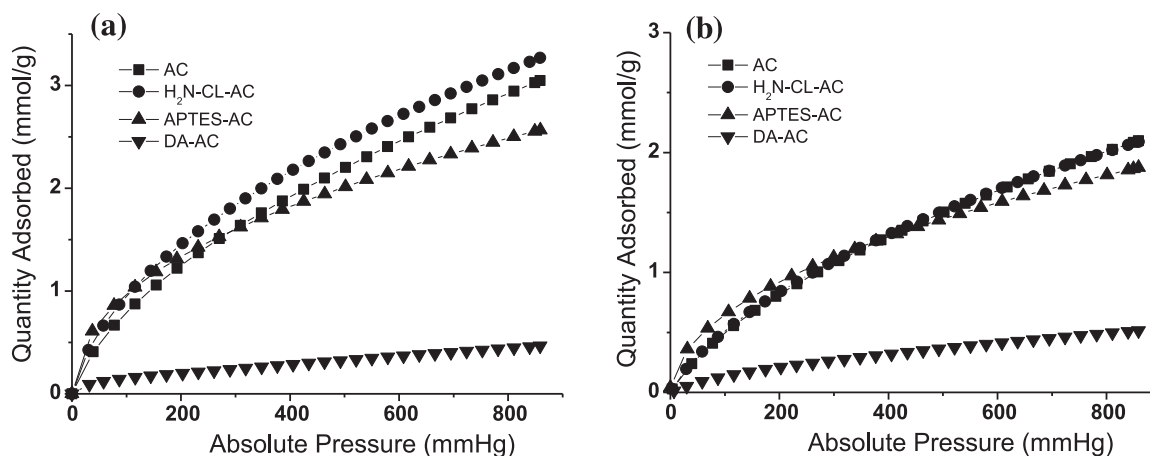


Fig. 1. CO_2 adsorption isotherms for adsorbents at 273 (a) and 298 (b) K.

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