



Copper oxide-decorated porous carbons for carbon dioxide adsorption behaviors

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ABSTRACT

Copper oxide-loaded porous carbons (PCs) for high efficient carbon dioxide capture were prepared. Copper oxides were loaded onto porous carbons by a postoxidation method involving copper electroplated PCs at 300 °C in an air stream. Additionally, porous carbons were prepared from ion-exchangeable polymeric resin by a chemical activation method. The microstructure of the copper oxide/PCs was characterized by XRD, and the formation of copper oxides after the postoxidation process was confirmed by XPS. The carbon dioxide adsorption behaviors were evaluated by a PCT (pressure–composition–temperature) apparatus at 298 K and 1.0 atm. It was found that the presence of copper oxides significantly led to an increase in the carbon dioxide adsorption capacity of the carbons. Copper oxide nanoparticles have electron-donor features, resulting in the enhancement of adsorption capacity of carbon dioxide molecules, which have an electron acceptor feature.

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1. Introduction

It has become commonly accepted that carbon dioxide is one of the major greenhouse gases causing global warming. Moreover, carbon dioxide is often found in natural gas streams [1–3]. When combined with water, carbon dioxide creates carbonic acid, which is corrosive. Therefore, there is growing interest in developing technologies for the efficient adsorption or storage of large quantities of carbon dioxide. Commercial carbon dioxide capture technologies that exist today are very expensive and energy intensive. Improved technologies for the capture of carbon dioxide are necessary to achieve low energy penalties. Adsorption is considered to be among the promising technologies available for capturing carbon dioxide from flue gases [4–7]. The success of such an approach is strongly dependent on the use of suitable adsorbents with good CO₂ selectivity and adsorption capacity, resulting in large specific surface area and viable CO₂-friendly sites on adsorbent surfaces [7,8].

Porous materials, including activated carbons, zeolites, and alumina powders, are useful materials for gas adsorption and storage. Activated carbons, which are also known as porous carbons, have been widely used in separation, purification, and catalytic processes as well as in energy storage [9]. Porous carbons with a high specific surface area can be prepared from polymeric precursors [10] such as ion-exchangeable resins, and their pore structures can be controlled by the activation conditions.

It is also true that carbon dioxide molecules have a soft acidic nature [8,11]. This fact indicates that the presence of strong base functional groups such as amine groups can enhance the carbon dioxide adsorption capacity. According to a previous study by the authors, the presence of copper oxide on carbon materials can increase the adsorption capacity of the acidic adsorbate [12], indicating that copper oxides serve as an electron donor.

In the present study, copper oxide-loaded porous carbons were prepared for highly efficient carbon dioxide adsorption. Copper oxides were introduced via the postoxidation of copper-electroplated porous carbons, and the effects of the oxidation degree on the carbon dioxide adsorption capacity of the porous carbons were evaluated.

2. Materials and methods

2.1. Sample preparation

To prepare the as-received porous carbon sample, polystyrene (PSI)-based ion-exchangeable resins (SamYang Co., Korea) were used as a precursor. They were impregnated with KOH according to a 4.0:1 (KOH:resin) weight ratio according to the aforementioned study [10]. In this work, ultrapure N₂ gas was fed (50 cc/min) into a tubular furnace. The samples were heated at a rate of 5 °C/min and then activated for 1 h at 1000 °C. They were then labeled “as-received” (or porous carbons, PCs). The textural properties of the as-received samples are listed in Table 1.

Neat Cu/PCs were prepared by a copper-electroplating method [13]. The as-received were subjected to copper electroplating with

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Table 1

Textural properties of the as-received porous carbons, neat Cu-loaded porous carbons, and Cu₂O-loaded porous carbons as a function of the oxidation time.

	As-received	Neat Cu/PCs	Cu ₂ O-2/PCs	Cu ₂ O-5/PCs	Cu ₂ O-10/PCs
S_{BET}^a (m ² /g)	2100	1610	1590	1565	1510
V_{M}^b (cm ³ /g)	1.15	0.81	0.80	0.78	0.75
V_{T}^c (cm ³ /g)	1.22	0.90	0.89	0.88	0.86

^a Specific surface area (m²/g) (P/P_0 : 0.03–0.05) = $V_{\text{ads}} \times 6.02 \times 10^{23} \times 1.62 \times 10^{-19} / 22,400$.

^b Micropore volume (cm³/g) (t -plot; thickness range = 6–20) = $[13.9900 / \{0.034 - \log(P/P_0)\}]^{0.5}$.

^c Total pore volume (cm³/g) = $V_{\text{ads}} (P/P_0 = 0.995) \times (\text{molar volume of liquid N}_2 / \text{molar volume of gaseous N}_2)$.

a fixed treatment time of 5 s, and were termed the neat Cu/PCs. The plating bath was composed of CuSO₄ and H₂SO₄; the pH was 3.0 ± 1, the temperature was 25 ± 1 °C, and the current density was fixed at 70 A/m². The copper content of neat Cu/PCs, measured by AAS, was around 8.2 wt.%.

The Cu₂O/PC samples were prepared using a postoxidation method of the neat Cu/PCs samples. The oxidation temperature was fixed at 300 °C in an air stream, and the heating rate was 5 °C/min. The oxidation time was varied in the range of 2–10 min for the Cu₂O-2/PCs, Cu₂O-5/PCs, and Cu₂O-10/PCs. During the oxidation, the sum contents of copper and copper oxide were slightly increased (up to 10.5 wt.%) due to the oxidation (or loss) of carbon support.

2.2. XRD and XPS studies

Wide-angle X-ray diffraction (XRD) patterns of the neat Cu/PCs and Cu₂O/PC samples as a function of the oxidation time were obtained with a Rigaku Model D/MAX-III B diffraction meter equipped with a rotation anode using CuK α radiation ($\lambda = 0.15418$ nm).

The XPS spectra were collected using a MgK α X-ray source (1253.6 eV). The pressure inside the chamber was held below 5×10^{-8} Torr during the analysis. The C_{1s} electron binding energy was referenced at 284.6 eV, and a curve-fitting procedure was carried out using a nonlinear least-square curve-fitting program with a Gaussian–Lorentzian production function. For all elements, atomic concentrations were estimated based on comparisons of the integrated peak intensities normalized by the atomic sensitivity factors.

2.3. Textural properties and carbon dioxide adsorption

N₂ adsorption isotherms were measured using an ASAP 2010 (Micromeritics) at 77 K. The samples were degassed at 573 K for 12 h to obtain a residual pressure of less than 10^{−6} Torr. The amount of N₂ adsorbed on the samples was used to calculate the specific surface area by means of the BET equation [14,15]. The total pore volume was estimated to be the liquid volume of the N₂ at a relative pressure of approximately 0.995. The micropore volume was calculated using the t -plot method [16–18].

The carbon dioxide adsorption behaviors of the samples at 298 K and 1 atm were evaluated using a BEL-HP (BEL Japan). Before each analysis, the samples were degassed for 6 h at 573 K. The sample weight for each analysis was fixed at 1.0 g, and the temperature of the sample cylinder was maintained at 298 ± 0.05 K.

3. Results and discussion

Fig. 1 shows the XRD diffraction of neat Cu/PCs and Cu₂O/PC samples. In the neat Cu/PCs, metallic copper peaks were clearly found at $2\theta = 43$ and 50 and decreased as the oxidation time

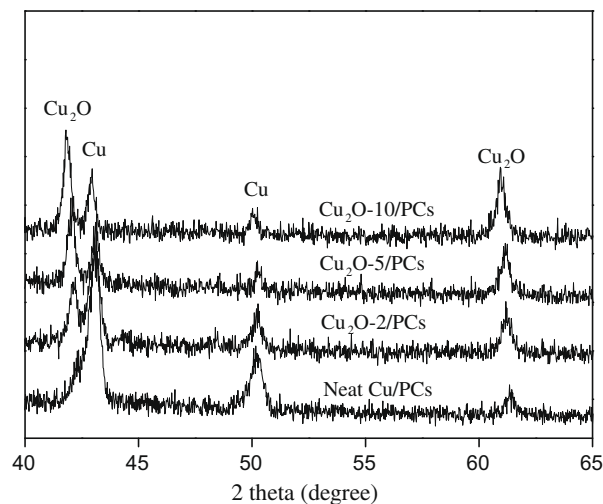


Fig. 1. XRD spectra of Cu₂O/PCs samples as a function of the oxidation time.

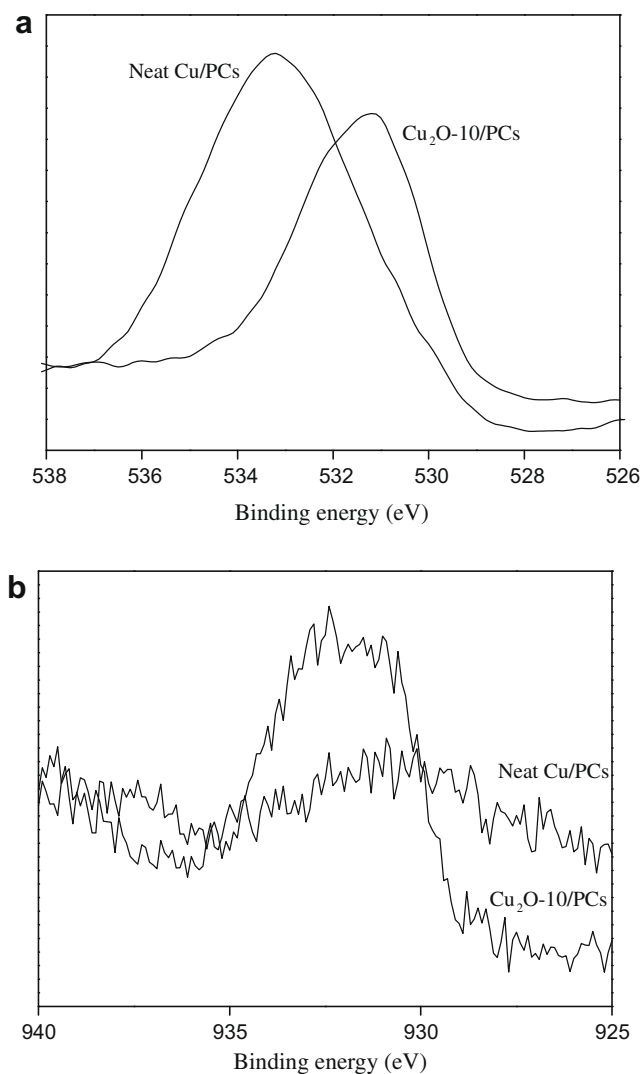


Fig. 2. High-resolution (a) O_{1s} and (b) Cu_{2p} peaks of neat Cu/PCs and Cu₂O-10/PCs samples.

increased. It is interesting to note that the Cu₂O peaks (at $2\theta = 42$ and 62) were increasingly enhanced as the amount of metallic copper decreased.

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