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Populus wood biomass-derived graphene for high CO₂ capture at atmospheric pressure and estimated cost of production

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ABSTRACT

In the present study, populus wood biomass (PWB), as a precursor, was first carbonized under a nitrogen atmosphere and the obtained carbon was used to prepare graphene via chemical activation using KOH. The graphene samples were used to study as adsorbents for CO₂ capture. The effect of different parameters, such as various KOH/C weight ratios (2:1–4:1 g/g), different heating temperatures (750–950 °C) and heating time (30–90 min), on the surface characteristics and CO₂ uptake capacity of the as-synthesized graphene was investigated. Various characterization techniques including elemental, TGA, BET, TEM, FT-IR and Raman were used. Under the experimental conditions of KOH/C impregnation ratio of 3:1, heating temperature of 850 °C and heating time of 60 min, the graphene sample showed a BET surface area of 1317.1 m²/g, total pore volume of 0.604 m³/g, up to 94% microporosity, and an average micropore diameter size of 1.84 nm. The measured uptake capacity of 7.2 mmol/g at a pressure of 1 bar and 293 K by sample G-3-850-60 was among the highest reported values in the literature data for carbon base materials. The isosteric heat of CO₂ adsorption onto the graphene samples were computed from the Clausius–Clapeyron equation, which was consistent with the physical nature of adsorption. Furthermore, these nano-adsorbents exhibited a high CO₂ adsorption capacity, thus proving to be good candidates for cost-effective CO₂ capture and storage for the downstream application on an industrial scale. In addition, the results of our laboratory investigations explore a process for scaling-up a complete flow diagram of graphene production from populus wood biomass and an economic evaluation of graphene production is estimated at a cost of about \$25 per kg.

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1. Introduction

Anthropogenic CO₂ emissions, the consequence of the combustion of fossil fuels, have resulted in an environmental concern due to an increased energy demand in a world marked by population growth and

a higher standard of living. The European Strategic Energy Technology Plan (SET-Plan) is aimed at decreasing, by 2020, greenhouse gas emissions by 20% and a further reduction to 50% by 2050. It is essential to advance technologies that will help us to use fossil fuels while decreasing the emissions of greenhouse gases. It has been recognized that the existing commercial CO₂ capture technology is costly and energy-consuming, and, hence, improved technologies for CO₂ capture seems

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necessary in order to achieve lower energy demands. Among such technologies, adsorption processes based on porous solids (e.g., carbon materials) can be excellent candidates in economic and environmental terms to alleviate CO₂ emissions compared to the precise technology of absorption processes based on basic solutions (Ali, 2010, 2012, 2014; Ali et al., 2012; Ali and Gupta, 2006; Casco et al., 2014; Choi et al., 2009; Guo and Lua, 2003). Utilizing highly porous solids as sorbents to capture CO₂ by using the pressure, temperature or vacuum-swing adsorption system seems to be a promising option (Sevilla and Fuertes, 2012; Shaarani and Hameed, 2011). In this regard, the different porous adsorbents such as the metal-organic frameworks (MOFs), activated carbon, zeolites, graphene, porous carbons or organic-inorganic hybrid sorbents have been targeted as potential alternatives for CO₂ capture (Shafeeyan et al., 2010). Graphene is known as a new nanomaterial with a single sheet of carbon atoms arranged in a hexagonal lattice. After the first report of synthesizing it by using the 'Scotch tape' approach in 2004 (Belmabkhout and Sayari, 2009), graphene has become an area of research attracting a lot of interest. Some enchanting characteristics of graphene, such as high thermal conductivity (~5000 W/mK) (Zhao et al., 2009), high theoretical specific surface area (2630 m²/g) (Ning et al., 2012; Wang et al., 2012), strong Young's modulus (~1 TPa) (Cazorla et al., 2011) and fast charge carrier mobility (~200,000 cm² V⁻¹ s⁻¹) have been reported. Graphene has been applied to isolate gases, recovery of solvents, and the elimination of organic pollution. Moreover, the application of graphene to natural gas storage can also be promising. Recent synthetic attempts have diverged in several different directions. For example, reduction of graphene oxide (Aaron and Tsouris, 2005), micromechanical cleavage (Srivastava et al., 2007), chemical vapor deposition (CVD) (Cazorla-Amorós et al., 1996), liquid-phase exfoliation (Mohr and Rao, 1999), chemical synthesis (Gauden and Wiśniewski, 2007) and carbon segregation (Ghosh et al., 2008) have been improved to generate single-layer graphene (SLG) or few-layer graphene (FLG) (Xu et al., 2012). CVD appears to be an interesting method for growth of graphene on copper foil because of its ability to produce a large-area synthesis of high quality graphene single-layer in comparison with mechanical and/or chemical treatments (Lee and Park, 2012). However, in this application, graphene represents low CO₂ adsorption resulting in a limitation of its performance. To improve the CO₂ uptake capacity, the fabrication of porous carbons with a pore structure that can adsorb CO₂ properly can overcome the problem (Heidari et al., 2014). In the present study, we demonstrate the CO₂ adsorption ability of graphene. The chemical activation has been conducted with KOH and the capacity has been assayed under different pressures at room temperature. Additionally, the regeneration of adsorbent was also studied.

2. Materials and methods

2.1. Biomass

Populus wood residue was collected from the Borujen County in the Chaharmahal and Bakhtiari province of Iran, which was utilized as precursor materials for the preparation of nano-adsorbent. This material was initially crushed, then dried at 70 °C for 72 h. The precursor was ground by ball milling to powder and sieved to acquire a particle size in the range of 0.4–0.8 mm. The ultimate analysis of populus wood biomass was done for elemental content (carbon, hydrogen, nitrogen and sulfur).

2.2. Preparation of graphene

The carbonization of powdered PWB was carried out under N₂ using a heating rate of 5 °C min⁻¹ for about 1 h. The carbonized samples were then blended with the KOH solution based on the KOH/C mass ratio of 2–4 (2 g KOH: 1 g carbonized PWB to 4 g KOH: 1 g carbonized PWB). The activation, in the presence of KOH as a chemical activating agent, was carried

out in an atmosphere of N₂ at a heating rate of 5 °C min⁻¹, activation temperature ranging from 750 to 950 °C, activation time ranging from 30 to 90 min and a constant nitrogen gas flow rate of 120 ml/min (0.198 kg/d). The prepared samples were carefully washed several times with 10 wt% of HCl to get rid of any inorganic salts, and then finally washed with distilled water until neutral pH. Ultimately the samples were dried in the oven at 110 °C for 12 h. The as-prepared graphene samples were denoted as G-R-T-t, where G was the abbreviation of graphene, R referred to the KOH/C weight ratio in g/g, T referred to the target activation temperature in °C and t stood for the activation time in minutes.

2.3. Characterization of materials

Textural characterization of graphene samples was examined by N₂ adsorption/desorption isotherms at 77 K using an automatic volumetric apparatus (Micromeritics ASAP 2020) BET surface area and porosity analyzer. Before the N₂ adsorption/desorption measurements, all samples were degassed at 200 °C in a vacuum for 3 h. The BET surface area was computed using the BET (Brunauer, Emmet and Teller) method from adsorption data in the range of relative pressures (P/P_0) 0.001–1. The total pore volume was calculated from the amount of N₂ gas (99.999% purity) adsorbed at P/P_0 of 0.99. The micropore method (MP) was used to achieve the micropore volume while the mesopore volume was ascertained by subtracting the micropore volume from the total pore volume. The mesopore size distribution was defined by using the Barret-Joyner-Halenda (BJH) method. FT-IR spectra of the graphene samples were acquired by Fourier transform infrared spectroscopy. The spectra were recorded from 400 to 4000 cm⁻¹ and KBr pellet was utilized as a reference sample. Furthermore, Raman microscope (Almega Thermo Nicolet Dispersive Raman Spectrometer) was used to characterize the functional groups on the surface chemistry of as-synthesized graphene. The chemical composition of the graphene samples was defined using CHNS/O elemental analyzer. The Elemental analyzer determined the nitrogen, carbon, sulfur, and hydrogen content of the samples. The oxygen content was then determined by subtracting the sum of the other components from 100. The transmission electron microscope (TEM) was used for supplementary observation of the morphology and textural features of as-synthesized graphene.

2.4. CO₂ adsorption measurements

The CO₂ adsorption capacity of the prepared samples was appraised using a static volumetric apparatus. The CO₂ adsorption experiments were carried out under pressures ranging from 1 to 10 bar and at temperatures of 273–333 K. The MATLAB software was used to determine the amount of CO₂ adsorbed by the graphene-based PWB samples using the Soave-Redlick-Kwong (SRK) formula. Adsorption/desorption cycles were being tested at 20 °C. The recyclability of the adsorption/desorption cycle was performed using 1 g of as-synthesized samples. All samples were degassed at 250 °C prior to the cyclic experiments. The adsorption/desorption cycle was repeated five times.

2.5. Adsorption isotherm

Adsorption isotherms are essential to outline the adsorbate and adsorbent interactions and are, therefore, helpful for opti-

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