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Magnetite impregnation effects on the sorbent properties of activated carbons and biochars



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ARTICLE INFO

Article history: Received 3 October 2014 Received in revised form 6 December 2014 Accepted 9 December 2014 Available online 17 December 2014

Keywords: Magnetic sorbents Activated carbon Biochar Wastewater treatment Soil and sediment remediation

ABSTRACT

This paper discusses the sorbent properties of magnetic activated carbons and biochars produced by wet impregnation with iron oxides. The sorbents had magnetic susceptibilities consistent with theoretical predictions for carbon-magnetite composites. The high BET surface areas of the activated carbons were preserved in the synthesis, and enhanced for one low surface area biochar by dissolving carbonates. Magnetization decreased the point of zero charge. Organic compound sorption correlated strongly with BET surface areas for the pristine and magnetized materials, while metal cation sorption did not show such a correlation. Strong sorption of the hydrophobic organic contaminant phenanthrene to the activated carbon or biochar surfaces was maintained following magnetite impregnation, while phenol sorption was diminished, probably due to enhanced carbon oxidation. Copper, zinc and lead sorption to the activated carbons and biochars was unchanged or slightly enhanced by the magnetization, and iron oxides also contributed to the composite metal sorption capacity. While a magnetic biochar with $219 \pm 3.7 \text{ m}^2/\text{g}$ surface area nearly reached the very strong organic pollutant binding capacity of the two magnetic activated carbons, a magnetic biochar with $68 \pm 2.8 \text{ m}^2/\text{g}$ surface area was the best metal sorbent. Magnetic biochars thus hold promise as more sustainable alternatives to coal-derived magnetic activated carbons.

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1. Introduction

Activated carbon is one of the most widely used sorbent materials for the purification of water (Sontheimer et al., 1988), air (Cheremisinoff, 2002), soil (Hale et al., 2012a, Vasilyeva, et al., 2001) and sediment matrices (Cho et al., 2012, Ghosh et al., 2011, Zimmerman et al., 2004). While powdered activated carbon has better sorption kinetic properties than granular activated carbon (Ahn et al., 2005), the separation of powdered activated carbon from cleaned environmental matrices is challenging. This is especially true for the novel soil and sediment remediation applications (Ghosh et al., 2011), where powdered activated carbon recovery from slurries of soil or sediment particles cannot be achieved with traditional coagulation/flocculation/clarification processes. Magnetic activated carbon (Faulconer et al., 2012, Kakavandi et al., 2013, Mohan et al., 2011, Ngarmkam et al., 2011, Oliveira et al., 2002, Yang et al., 2008) or magnetic biochar (Chen et al., 2011, Zhang et al., 2007) may provide an alternative route to the recovery of pollutant-loaded sorbents from cleaned environmental matrices. Magnetic activated carbon has been effectively used for organic and inorganic pollutants removal from wastewater (Castro et al., 2009, Oliveira et al., 2002) and also in the mining industry (Rossier et al., 2009).

Environmental impact analysis shows that locally produced biochar provides a more sustainable alternative to the use of coal-derived activated carbon (Sparrevik et al., 2011), but biochar will not necessarily have equivalent sorption properties (Hale et al., 2012b). The relative performance of magnetic activated carbon and magnetic biochar is therefore of great interest. Magnetic activated carbons and biochars have higher production costs, and may have changed sorbent properties, in comparison with the unaltered activated carbons and biochars. Better understanding of potential tradeoffs between the desirable attribute of magnetism and its side-effects on the pollutant binding capacity of magnetic sorbent materials is also required to properly assess the economic sense and technical feasibility of various treatment applications. The aim of this study was therefore a detailed characterization of magnetite impregnation effects on the sorbent properties of two types of commercial activated carbons (one produced from coal and one produced from coconut shells, which may be also described as an 'activated biochar') and two types of commercial biochars (both made from mixed wood at 500 °C, one produced in Romania, and one produced locally in the UK and marketed as sustainable and organic). A series of adsorption experiments were conducted to compare the adsorption ability of these two activated carbons and two biochars with and without magnetite impregnation for different pollutant classes comprising hydrophobic organic contaminants (phenanthrene), more hydrophilic organic contaminants (phenol), and positively charged bivalent heavy metal pollutants (Pb²⁺, Cu²⁺, and Zn²⁺) which may have electrostatic interactions with negative surface charges of the sorbents. To the best of our knowledge the present study offers the first comparison of magnetite impregnated activated carbons and biochars synthesized with the same procedures.

2. Materials and methods

2.1. Materials

Two kinds of commercial activated carbon were investigated, one produced from Coconut shells by Norit, and one produced from anthracite coal by Calgon (Calgon Filtrasorb 400) and obtained from Chemviron (Lancashire, United Kingdom). Two kinds of commercial biochar were obtained from Romchar (Harghita, Romania), and Oxford Biochar Ltd. (Dorset, UK), respectively. According to the manufacturers, both biochars were made from mixed wood chips with a maximum pyrolysis temperature of 500 °C, and the UK biochar was described by the producer as sustainable and organic. The sorbents were labelled CocoAC, CoalAC, Bio, and OrgBio, respectively. CocoAC, CoalAC, Bio and OrgBio were ground with a ceramic mortar to realize a <64 μ m size distribution.

Phenanthrene stock solution (5000 μ g/mL in methanol) was bought from Supelco (Bellefonte, PA, USA). Metal salts (CuCl₂·2H₂O and ZnCl₂) were obtained from BDH Laboratory Supplies (Poole, England) and Pb(NO₃)₂ from Merck (Darmstadt, Germany). Iron salt FeSO₄·7H₂O was obtained from BDH Laboratory Supplies (Poole, England) and FeCl₃·6H₂O was obtained from VWR (Lutterworth, UK). Aqueous phenol solution (30 g/L) was obtained from VWR (Lutterworth, UK). Ethanol with >99.8% purity was obtained from VWR (Lutterworth, UK). A phenanthrene and a deuterated phenanthrene calibration standard in methanol were obtained from Supelco (Bellefonte, PA, USA), hexane (for pesticide residue analysis) and acetone (for HPLC analysis) were bought from Sigma–Aldrich (St. Louis, MO, USA).

2.2. Preparation of magnetite impregnated AC and biochar

Magnetic activated carbon and biochar were prepared by a chemical precipitation technique as presented in Supporting information. This method is based on a technique used in previous studies (Castro et al., 2009, Oliveira et al., 2002), but with a few modifications. Briefly, 2.5 g AC or BC, 1.83 g FeS- $O_4 \cdot 7H_2O$ and 3.33 g FeCl₃ $\cdot 6H_2O$, and 100 mL de-ionized water were combined in a beaker. The mixtures were stirred and heated to 65 °C and then cooled by stirring to below 40 °C. 5 M NaOH solution was added drop wise to raise the pH to 10-11 and precipitate the iron hydroxides. The mixtures were stirred for 1 h and then rested overnight. The supernatants were removed, and the precipitates were washed and rinsed with de-ionized water onto a coffee filter, then rinsed with ethanol and dried at 80 °C in an oven. The magnetic carbon materials were labelled as MagCocoAC, MagCoalAC, MagBio, and MagOrgBio, respectively.

2.3. Sorbent characterization

The size distributions were measured by a laser diffraction method using a Malvern MasterSizer 2000 (Malvern, UK) which can measure particle size between 20 nm and 2 mm. The specific surface area (SSA), pore volume, and average pore size of the samples were determined by gas (N_2) adsorption/

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