



Influence of post-treatment strategies on the properties of activated chars from broiler manure [☆]



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HIGHLIGHTS

- Acid washing broiler litter activated biochars has no effect in copper ion adsorption.
- Acid washing litter activated biochars affects surface area, pH, ash and carbon contents.
- Manure type and activation conditions are determining factors in copper ion adsorption.
- Positive correlations found between copper uptake and concentration of certain elements.
- The elements (P, S) are likely present as negatively charged functional groups in the activated biochar.

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ABSTRACT

There are a myriad of carbonaceous precursors that can be used advantageously to produce activated carbons or chars, due to their low cost, availability and intrinsic properties. Because of the nature of the raw material, production of granular activated chars from broiler manure results in a significant ash fraction. This study was conducted to determine the influence of several pre- and post-treatment strategies in various physicochemical and adsorptive properties of the resulting activated chars. Pelletized samples of broiler litter and cake were pyrolyzed at 700 °C for 1 h followed by a 45 min steam activation at 800 °C at different water flow rates from 1 to 5 mL min⁻¹. For each activation strategy, samples were either water-rinsed or acid-washed and rinsed or used as is (no acid wash/rinse). Activated char's physicochemical and adsorptive properties towards copper ions were selectively affected by both pre- and post-treatments. Percent ash reduction after either rinsing or acid washing ranged from 1.1 to 15.1% but washed activated chars were still alkaline with pH ranging from 8.4 to 9.1. Acid washing or water rinsing had no significant effect in the ability of the activated char to adsorb copper ions, however it significantly affected surface area, pH, ash content and carbon content. Instead, manure type (litter versus cake) and the activation water flow rate were determining factors in copper ion adsorption which ranged from 38 mg g⁻¹ to 104 mg g⁻¹ of activated char. Moreover, strong positive correlations were found between copper uptake and concentration of certain elements in the activated char such as phosphorous, sulfur, calcium and sodium. Rinsing could suffice as a post treatment strategy for ash reduction since no significant differences in the carbon properties were observed between rinsed and acid wash treatments.

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1. Introduction

Copper is an essential micronutrient required by all living organisms for a variety of physiological and biochemical processes, however it can be potentially toxic to aquatic organisms when available in excess in the water (Martins et al., 2011). While there

are important natural sources of copper, multiple human activities (industries, agriculture, harbors) have considerably increased the input of this metal in estuarine and marine environments around the world (Morillo et al., 2005). Copper mining and smelting as well as electroplating industries are among one of the most common sources of copper pollution (Vijayaraghavan et al., 2004). Ionic copper, specifically, is highly correlated with toxicity (Borgmann, 1983). This heavy metal ion can also be found in drinking water through corrosion of plumbing systems. The removal and recovery of heavy metals from industrial and municipal wastewater is important for environmental protection and human health. The environmental Protection Agency, through their official list of

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priority contaminants has identified a maximum contaminant level for copper of 1.3 ppm in drinking water (U.S.E.P.A., 2011). A current review of the available methods for heavy metal removal from wastewater made by Fu and Wang (2011) includes chemical precipitation, ion-exchange, adsorption, membrane filtration, coagulation–flocculation, flotation and electrochemical methods. Removal of copper and other toxic metals from any contaminated water source via adsorption technology can be an effective remediation method when the right adsorbent is identified. In the U.S., 79% of total activated carbon production goes for liquid-phase applications with 37% and 21% of that going directly to industrial and municipal wastewater treatment respectively (Chemical Market Reporter, 2006). While the manufacture of activated carbons has been traditionally done using coal, coconut hulls or wood (Lima and Marshall, 2005a,c), the ability to utilize under-valued, inexpensive and readily available plant and agricultural residues as feedstock for activated carbon manufacture has understandable advantages.

As the raw materials obtained from agricultural wastes are easily and abundantly available, the cost of preparing the carbons is expected to be lower and provide a potentially inexpensive replacement of existing commercial adsorbents from non-renewable sources. Significant research advances have been made in this area in the last decade. Bailey et al. (1999) published a review summarizing available information on low-cost natural sorbents for heavy metals removal, which included materials such as bark, chitin/chitosan, dead biomass, seaweed/algae, clay, fly ash, peat moss, bone gelatin beads, modified wool and cotton, amongst others. Ioannidou and Zabaniotou (2007) published an updated review on the use of agricultural residues for the production of activated carbons. In their review, they listed more commonly and readily available residues, such as nutshells, fruit stones, sugarcane bagasse, soybean hulls, corn husks/cobs, grasses. Because the large majority of these organic residues is mainly composed of cellulosic compounds, they have an adequate amount of carbon to qualify them as a potential feedstock in activated carbon making. Lima and Marshall (2005a,c) demonstrated that broiler manure-based steam activated carbons were able to adsorb positively charged species such as copper, cadmium and zinc to a much larger extent than their plant based counterparts, such as carbons made from wood, coal or coconut shells, under the same processing conditions. Other studies reported on activated carbon and biochar production from animal manures (Shinogi and Kanri, 2003; Shinogi et al., 2003; Lima and Marshall, 2007; Qian et al., 2007; Qiu and Guo, 2010) or sewage sludge (Martin et al., 1996; Tay et al., 2001) and their ability to remove heavy metals (Fitzmorris et al., 2006; Zaini et al., 2009; Guo et al., 2010) or other compounds, i.e. organic compounds (Ahmedna et al., 2000; Rozada et al., 2003; Qian et al., 2007). Activated carbons sourced from agricultural residuals and produced via chemical activation have also been characterized (Johns et al., 1998; Koutcheiko et al., 2007). The large array of feedstock choices, pre-treatment options, activation conditions and post-treatment strategies can be extremely advantageous in producing activated carbons with specific desirable traits. In this process, however, the challenge is to determine cause–effect relationships between the original properties of the feedstock, the changes occurring during pyrolysis, activation and post-treatment technologies and the resulting characteristics of the activated carbon or char. Broiler manure as feedstock cannot be underestimated in light of the estimated U.S. production of 8.6 billion broilers in 2010 (U.S.D.A. N.A.S.S., 2011). In this study we utilize two types of broiler manure (litter and cake) and we look at different activation conditions as well as different post-activation treatments and report to their influence on the resulting activated char physical and chemical properties as it pertains to their ability to adsorb copper ions in laboratory prepared solutions.

2. Materials and methods

2.1. Sample preparation

Broiler litter and broiler cake samples were obtained from the USDA-ARS, Genetics and Precision Agriculture Research Unit (Mississippi State, MS). These two feedstock sources have been previously described (Lima and Marshall, 2005a,c) and their distinction results from the manure management practice called “de-caking” or removal of the fresh manure combined with some bedding material, feathers and spilled feed. As a result, producers only have to periodically add small quantities of fresh bedding, significantly reducing the frequency of complete clean-outs (Sistani et al., 2003). Litter is the older material that remains on the floor after de-caking. Samples (air dried to less than 15% moisture) were milled in a Retsch cross-beater mill (Glen Mills, Clifton, NJ) to less than 1 mm particle size and pelletized in a PMCL5 Lab pellet mill (CPM, Merrimack, NH) equipped with a 3/16 in (4.76 mm) die plate, to produce cylindrical pellets of 4.76 mm diameter and length of approximately 6.5 mm.

2.2. Production of activated chars and post-activation treatments

Pelletized samples were placed in a ceramic evaporating dish and inserted in a bench furnace (Lindberg/Blue M, Waterton, WI) equipped with a retort where they were pyrolyzed at 700 °C under nitrogen gas (flow rate of 1.6 L min⁻¹) for 1 h prior to steam activation. Steam activation involved injecting water at three flow rates (1, 3 or 5 mL min⁻¹) using a peristaltic pump, into the nitrogen gas flow entering the heated retort, for 45 min. Activated char samples were allowed to cool to room temperature overnight under a stream of nitrogen. At this point samples were either (a) used as is (unwashed; NW), (b) water rinsed with deionised water for 1 h and dried overnight at 80 °C (rinsed; R) or (c) acid washed with 0.1 M HCl for 1 h with two subsequent 1 h deionised water rinses and dried overnight at 80 °C (acid washed; W). Acid water washing/rinsing processes were carried out batch mode in a glass beaker at a 2% (w/v) ratio at 25 °C with the help of an overhead stirrer. Samples were sieved through 325 U.S. mesh sieve between rinses. With a mortar and a pestle, all dried activated char samples were reduced in size to between 0.42 mm and 1.0 mm (40 × 18 U.S. mesh) prior to all measurements and experiments.

2.3. Physical and chemical measurements: BET surface area, attrition, pH, ash content, ultimate analysis, elemental composition

Surface area measurements were obtained from nitrogen adsorption isotherms at 77 K using a Nova 2000 Surface Area Analyzer (Quantachrome Corp., Boynton Beach, FL). Specific surface areas (BET, Brunner–Emmett–Teller) were taken from adsorption isotherms using the BET equation. The micro pore size distributions were calculated using *t*-plots derived from the Nova 2000 software. Resistance to attrition, a measure of the activated char's ability to maintain physical integrity is defined as the particle size decrease due to breakage and was determined by agitating 3 g of activated char in a 250 mL Erlenmeyer flask for 15 min at 200 rpm in the presence of ten 5 g glass marbles. Percent attrition is the ratio between sample lost through a nominal opening of 0.425 mm (40 U.S. mesh screen) to initial weight. A Thermo Orion pH meter (Beverly, MA) was used to measure pH, where 0.5 g of sample was placed in 50 mL of deionized water, covered with parafilm, and allowed to equilibrate by stirring at 300 rpm for 72 h. Ash content in the activated char samples was determined by heating 1 g of sample to 650 °C under a flow of air for 6 h increments until no change in weight was observed and was

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