



CO₂ activation promotes available carbonate and phosphorus of antibiotic mycelial fermentation residue-derived biochar support for increased lead immobilization



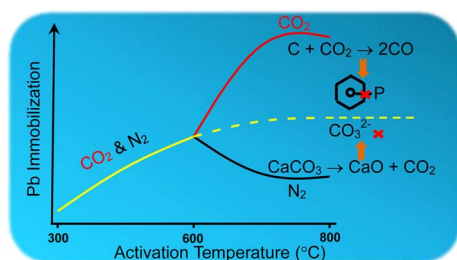
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GRAPHICAL ABSTRACT



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ABSTRACT

Recycling of antibiotic mycelial fermentation residue (AR), a hazardous waste material, into high-performance heavy metal remediation material is an emerging research hotspot. The inorganic composition of biomaterials represents their capacity to immobilize heavy metals. In this study, to improve the Pb immobilization capacity, lincomycin mycelial fermentation residue (LR, a type of AR) was pyrolyzed in CO₂ or N₂ gas under different temperatures to adjust the carbon and phosphorus composition of its biochar. Results indicate that both activation temperature and gas type can significantly influence the transformation of LR carbon and phosphorus-containing groups. At high temperatures, the activation gas significantly controlled changes in carbonate and available phosphorus; however, this process was less successful at low temperatures. CO₂ gas clearly inhibited the degradation of carbonate, but promoted consumption of the carbon matrix and released organism-combined phosphorus. Results suggest that CO₂-activated biochar at 700 °C exhibited the strongest Pb immobilization capacity of 454 mg/g in aqueous solution and the highest Pb immobilization rate of 60% in soil, due to its more sites (carbonate and available phosphorus) for Pb stabilization. This study provides an effective method for transforming waste AR into a high-performance material for metal stabilization.

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1. Introduction

Antibiotics are widely used in the treatment of both human and poultry, and China has become the largest antibiotic producer, accounting for 70% of the global market [1–3]. AR material, an emerging solid pollutant, is generated during the production of antibiotics; since 2008, AR has been classed as hazardous waste in China due to the fact that AR contains many toxic substances, including traces of antibiotics [3,4]. Therefore, the treatment of AR has become a subject of growing concern and scientific interest [5–8].

Recently, pyrolysis techniques have been successfully applied in the treatment of biomaterial to produce biochar [9–11]. Importantly, pyrolysis can effectively eliminate the antibiotics loaded in the AR, decreasing the potential antibiotics risk of AR-derived biochar [2]. Biochar is rich in carbon element and ash (e.g., carbonate and phosphate), and is widely used for heavy metal immobilization [12–14]. It has been well documented that available ash components represent the metal immobilization capacity of biochar via the precipitation reaction. Therefore, it is imperative to increase the content of carbon and phosphorus matters within biochar to increase its metal immobilization ability [15,16].

It is widely reported that biochar's available ash components for metal immobilization are significantly influenced by activation temperature [17]. For example, carboxyl is easily decomposed to carbonate at low activation temperatures (< 400 °C) and then carbonate is further degraded to CO₂ at high activation temperatures (> 500 °C) [18]. The available phosphorus is also strongly affected by activation temperature. Generally, high temperatures result in a decrease of available phosphate [19]. The type of activation gas also affects biochar's characteristics [20,21]. Typically, CO₂ gas partly inhibits the decomposition of calcium carbonate (CaCO₃ → CaO + CO₂) due to the saturation of CO₂ concentration. In addition, the carbon matrix also can react with CO₂ gas at thermodynamically favorable temperatures (~700 °C) [22,23]. This reaction transforms organic matter combined phosphorus into inorganic phosphorus, thereby increasing the amount of available phosphorus [24]. Undoubtedly, adjustment of the activation temperature and gas type is important in order to optimize the metal immobilization capacity of biochar.

As reported in a previous study, AR material retains plentiful carboxylate and phosphate, which are initially added as fermentation substances [25]. Accordingly, AR-derived biochar, when activated under an appropriate activation temperature and gas type, could be an ideal metal restorer for resisting soil and solution pollution. However, few studies have focused on recycling AR and transforming it into high performance biochar.

In this study, LR was sustainably recycled to biochar in order to immobilize lead (Pb) in both solution and soil. To accurately determine Pb immobilization mechanisms, the study analyzed the effect of activation temperature on the carbon and phosphorus-containing groups of LR-derived biochar in both N₂ and CO₂ gas. In order to more accurately understand the transformation of phosphorus species during LR pyrolysis, three main techniques, including chemical extraction, P K-edge XANES, and P 2p XPS were used to study the phosphorus species changes [26]. By combining this with an analysis of post-adsorbed biochar, we aim to clarify biochar application mechanisms for the remediation of polluted solutions and soils.

2. Materials and methods

2.1. Synthesis of LR-derived biochar

LR was collected from a pharmaceutical factory in Henan Province, China. After being dried at 60 °C for 24 h, the raw material was crushed and filtered through a 40-mesh sieve for further analysis.

Twenty grams of LR were fed into a quartz tubular reactor and pyrolyzed at different temperatures (300, 400, 500, 600, 700, and

800 °C) for 2 h at a heating rate of 8 °C/min under CO₂ or N₂ gas (flow rate of 200 mL/min) to synthesize LR-derived biochar. The prepared biochar samples are hereafter denoted as LC-X and LN-X, where X represents the activation temperature in °C, LC represents the sample activated in CO₂ gas, and LN represents the sample activated in N₂ gas.

2.2. Characterization of LR-derived biochar

The ash content of samples was determined by heating samples to 600 °C for 2 h in air. A CHN element analyzer (Vario EL III, Germany) was used to measure the contents of carbon (C), nitrogen (N), and hydrogen (H). Samples' specific surface area was analyzed using the BET test (Quantachrome, Quantasorb SI). The concentrations of magnesium (Mg), calcium (Ca), aluminum (Al), and total phosphorus in the samples were determined by ICP following the removal of carbon organic matter and acid digestion (HCl-HNO₃). For the analysis of water soluble phosphorus, dissolved organic carbon (DOC), and pH, 0.5 g of sample was suspended in 30 mL ultrapure water for 16 h at 25 °C. The supernatant was filtered and analyzed by ICP, a total organic carbon (TOC) analyzer, and a combination electrode.

To discern the proportion of phosphorus species, 0.5 g of the solid sample was sequentially extracted in 30 mL solutions of 0.5 M NaHCO₃ (pH adjusted to 8.5), 0.1 M NaOH, and 1 M HCl [24]. After shaking at 25 °C for 16 h during each extraction step, the filtrate was separated by centrifugation and filtered to analyze the phosphorus concentration by ICP. Two replicate experiments were conducted. The phosphorus species extracted in the three solutions correspond to labile phosphorus (NaHCO₃-P), phosphorus adsorbed on iron and aluminum compounds (NaOH-P), and phosphorus associated with or trapped in the minerals (HCl-P). Water-soluble phosphorus is considered part of NaHCO₃-P. Phosphorus in the residue (Residue-P) is the difference in total phosphorus and the sum of the concentrations of NaHCO₃-P, NaOH-P, and HCl-P. The relative abundance of phosphorus in different phosphorus species in a sample can be calculated using Eq. (1).

$$\text{relative abundance(\%)} = \frac{P_x}{P_{\text{total}}} \times 100\% \quad (1)$$

where P_x is the content of a certain phosphorus species and P_{total} is the phosphorus content.

To elucidate the activation mechanism of samples, pyrolytic CO, H₂, CH₄, and CO₂ gases were discontinuously collected using an aluminum foil gas collecting bag and analyzed using a gas chromatography (GC) system equipped with a thermal conductivity detector. The analytical methods for X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), and X-ray photoelectron spectroscopy (XPS) are detailed in the previous study [27–29].

X-ray absorption near edge structure (XANES) spectroscopy was used to determine the phosphorus inorganic species using fluorescence modes in a helium (He) chamber at room temperature at 2120–2200 eV. P K-edge (2145.5 eV) measurements were performed at beamline 4B7A at the Beijing Synchrotron Radiation Facility (BSRF). Data processing was performed using the software package ATHENA [30]. The contribution of different phosphorus species to total phosphorus fitting was calculated by linear combination fitting.

2.3. Immobilization of solution Pb by LR-derived biochar

Adsorption isotherms of Pb²⁺ for the samples were performed by mixing 0.03 g of sample with 60 mL of Pb²⁺ solution at different concentrations ranging from 2.5 to 250 mg/L. A 10-mM MES buffer was used to control solution pH at approximately 5. After shaking at 30 °C and 150 rpm for 24 h, the solution was separated by a 0.22-μm filter membrane and the concentration of Pb²⁺ was determined by inductively coupled plasma (ICP, Optima 8000). All adsorption experiments were performed in duplicate. The post-adsorption samples were

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