



## Full length article

# Adsorption-pyrolysis technology for recovering heavy metals in solution using contaminated biomass phytoremediation



Ziyu Han, Zhaohui Guo\*, Yong Zhang, Xiyuan Xiao, Zhi Xu, Yang Sun

Institute of Environmental Engineering, School of Metallurgy and Environment, Central South University, Changsha, 410083, PR China

## ARTICLE INFO

## Keyword:

Phytoremediating biomass  
Adsorption  
Pyrolysis  
Cadmium  
Copper  
Bio-ore

## ABSTRACT

Feasibility of adsorption-pyrolysis technology to recover metals from solution using contaminated biomass phytoremediation was discussed. The technology can meet three objectives: the disposal of metals-contaminated biomass from phytoremediation, the adsorption of metals from wastewater and the reuse of metals-saturated biomass. In this study, the recovery of Cd and Cu in metals-containing solution from contaminated biomass of phytoremediation was achieved through adsorption and pyrolysis technology. The results showed that pH value of the metals-containing solution and the pyrolysis temperature for metals concentration are the key factors of the novel technology. When the pH of the metals-containing solution was 6, the content of Cd and Cu in the saturated biomass after approximately 30 min adsorption was 2.7 wt% and 1.25 wt%. Next, the saturated biomass is pyrolyzed at 450 °C, and the final content of Cd and Cu in the solid phase product was 7.13 wt% and 6.58 wt%, respectively. The concentration of metals from the adsorption-pyrolysis process for bio-ore was primarily due to the redox reaction of Cd and Cu salts and the volatilizing properties of elements. The results suggested that the adsorption-pyrolysis technology is feasible for recovery of Cd and Cu in solution.

## 1. Introduction

Contamination of soil and water results from many industrial activities, such as the mining and smelting of ores, metals processing, energy and fuel production, fossil fuel combustion, and urban waste incineration (Jarup, 2003). Meanwhile, with the gradual decrease in the global fossil and ore resources, attention must turn to the development and utilization of regenerative resources (Kopetz, 2013). Therefore, the recovery of valuable and toxic metal ions from wastes become an essential necessity.

Phytoremediation of metal-contaminated soils offer a low-cost method for soil remediation, and some extracted metals may be recycled due to the remarkable enrichment capacity of some plants (Chaney et al., 1997). Therefore, phytoremediation is one of the main resources of metal-contaminated biomass. Meanwhile, bio-absorbent is another resource of metal contaminated biomass. Biomass is a common absorbent material for heavy metals in contaminated water (Sena et al., 2008). Areco et al. (2013) indicated that *fatua* biomass can be used as an efficient adsorbent for heavy metals; the functional groups in the weed cell wall have an adsorbent capacity for Cd(II) and Cu(II) ions of 0.180 and 0.063 mmol/g. Torres et al. (2006) and Dasilva et al. (2006) found that the Cu(II) ions adsorption capacity of cellulose modified with ethylenediamine was from 1.42 to 1.63 mmol/g, respectively.

Gurgel and Gil (2009) modified the biomass and adjusted the adsorption capacity for Cd(II) and Cu(II) ions to reach 0.77 and 1.09 mmol/g. The biomass has excellent capacity for heavy metal removal from water (Areco et al., 2013; Torres et al., 2006; Dasilva et al., 2006; Chai et al., 2010), and it should be a novel technology for the disposal of metal-containing solution using phytoremediation.

Pyrolysis takes full advantages of biomass resources (Ferella et al., 2016), but very little information of implementing pyrolysis is available for valorization heavy metal-contaminated biomass. Pyrolysis is an oxygen-free heating method to degrade biomass into biochar, tar, bio-oil and other products (Amutio et al., 2012; Koppolu et al. (2003) used biomass impregnated with acetates and citrates of Ni, Zn, Cu, Co, and Cr, and more than 98% of heavy metals were retained in the solid phase after pyrolysis in a lab-scale and pilot-scale test. Liu et al. (2012) used *Typha angustifolia* biomass to recover Pb from aqueous solution, and 98.8% of the Pb was retained in the solid phase after pyrolysis for adsorbed biomass. Lievens et al. (2008a,b) found that Cd, Cu, Pb and Zn in biomass could remain in the solid phase during the pyrolysis process, while Cd was easier to turn into the gas and liquid phase. Stals et al. (2010) found that temperature was the principal factor for heavy metal distribution in the thermal decomposition of contaminated biomass. These researchers also observed that the most suitable temperature for high quality bio-oil is 723 K, at which temperature the content of heavy

\* Corresponding author.

E-mail addresses: [zhguo@csu.edu.cn](mailto:zhguo@csu.edu.cn), [zhghuo@163.com](mailto:zhghuo@163.com) (Z. Guo).

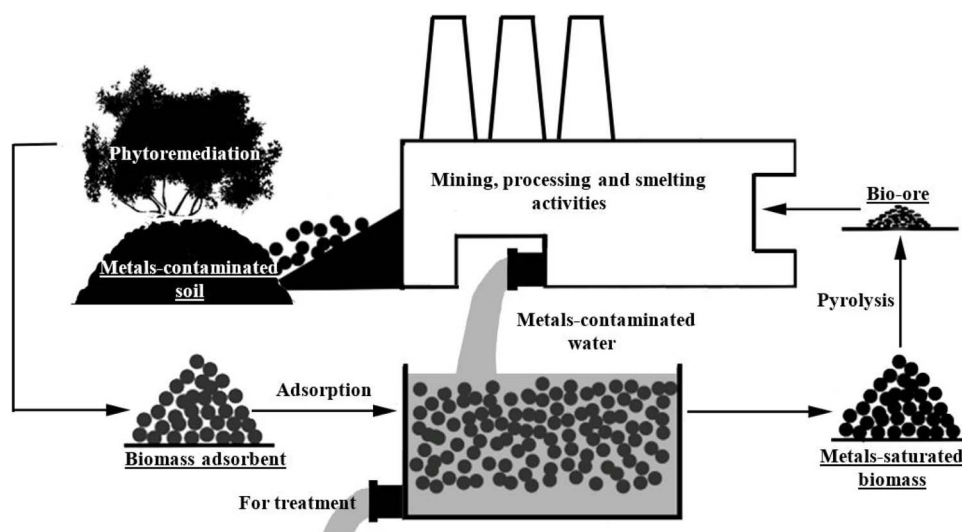


Fig. 1. Schematic of recovery heavy metals in metals-containing wastewater using metals contaminated biomass from phytoremediation.

metals in the oil is very low. However, heavy metals in biomass can stay in solid phase products during the pyrolysis process, which can be used for recovery of heavy metals. Therefore, the pyrolysis technology can serve three objectives: the disposal of metals-contaminated biomass from phytoremediation, the adsorption of metals from wastewater and the reuse of metals-saturated biomass as bio-ore.

In this study, a local plant in the Hunan province of China, *Broussonetia papyrifera* was selected to investigate heavy metals recovery from contaminated soil and water (Fig. 1). As a local species, the *Broussonetia papyrifera* was more tenacious vitality and larger biomass, meanwhile, it had potential tolerance on the heavy metals (Tong et al., 2015).

Soil and water contamination contain the same heavy metals in a mining or smelting industrial space. In the vicinity of a nonferrous metals industrial district, the soil and water is mainly polluted by heavy metals, such as copper (Cu), cadmium (Cd), lead (Pb), and zinc (Zn). Some of them are high-value such as Cu while some of them are high-toxicity such as Cd. Moreover, under thermal treatment condition, the stability of these metals may be closely related to the melting point, moreover, among these metals, metals with highest and lowest melting point is Cu (1083 °C) and Cd (321 °C), therefore, Cu and Cd are selected. In this study, the aims are to 1) study the adsorptive properties of *Broussonetia papyrifera* biomass for Cd and Cu in wastewater and effect of solution pH on metals adsorption, 2) analyse the proportion of Cd and Cu in solid phase after pyrolysis at 450 and 950 °C, and 3) elucidate the stabilization mechanism of heavy metals in the solid phase in the adsorption-pyrolysis process.

## 2. Materials and methods

### 2.1. Preparation of biomass

*Broussonetia papyrifera* biomass was collected from a phytoremediation site in Hunan province of China. The biomass was cut into small parts and washed carefully with deionized water and oven dried at 60 °C for 72 h. The dried biomass was ground by a high-speed rotary cutterbar, and the particles of approximately 1 mm were stored in a drying vessel for experiments. The chemical composition, proximate analysis and content of Cd and Cu are summarized in Table 1.

### 2.2. Adsorption experiment

The adsorption experiments were carried out in 250 mL conical flasks with *Broussonetia papyrifera* biomass at a room temperature of approximately 20 °C and shaken by oscillation with a frequency of

Table 1  
Basic properties of tested *Broussonetia papyrifera* biomass.

Content of alkaline earth metals (mg/kg)				Proximate analysis (wt%)				Content of heavy metals (mg/kg)	
Na	K	Ca	Mg	Moisture	Ash	Volatiles	Fixed carbon	Cd	Cu
63	6402	3205	514	4.08	7.33	71.64	15.50	1.6	6.0

180 rpm. Acetate Cd and Cu solution was used to stimulate contaminated water with concentrations of Cd and Cu at 1.43 and 1.29 mmol/L, respectively. The initial solution pH was adjusted to 2.0, 4.0 and 6.0 by adding 1.0 mol/L HCL and NaOH. For the adsorption experiment, 3 g of *Broussonetia papyrifera* biomass mixed with 150 mL acetate Cd and Cu solutions in 250 mL conical flasks at various time intervals between 10 and 180 min. After the sorption, the suspension liquid was separated by filtration, and the heavy metals concentration in filtrate was determined by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES, IRIS intrepid II XSP, USA). After being filtrated and dried, the saturated biomass was used as a raw material for the pyrolysis experiment.

### 2.3. Pyrolysis experiment

First, *Broussonetia papyrifera* biomass was employed on a thermal gravimetric analyser (TGA, SDT-Q600, USA). A 20 mg biomass was placed in the burning room and pyrolyzed with temperature increasing from room temperature (25 °C) to 950 °C at a heating rate of 10 °C/min. A high purity nitrogen stream was continuously passed into the burning room at a flow rate of 40 mL/min in order to control the level of oxygen. Based on the appropriate pyrolysis temperature for *Broussonetia papyrifera* biomass, a lab-scale pyrolysis experiment in tube furnace was designed (Fig. 2). The length of the quartz tube in the tube furnace was 1 m, and the inner diameter was 130 mm. The sample of 1.5 g adsorbent *Broussonetia papyrifera* biomass was placed in a porcelain boat and pulled in the centre of the heating zone, and the quartz tube was heated by an electric ring at a rate of 10 °C/min to temperatures of 450 and 950 °C for 2 h. The reactor was continuously purged with nitrogen at 0.5 L/min to sweep the related gases from the quartz tube and the products of liquid was collected by cooling bottles with absorption liquid. Biomass and pyrolysis products were digested by nitric acid and perchloric acid according to standards of the Annual Book of Standards (1994), while the solutions were determined by ICP-AES. The solid products that had relatively higher Cd and Cu content were detected by

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