Bioresource Technology 238 (2017) 182–187

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Treatment of urban sludge by hydrothermal carbonization

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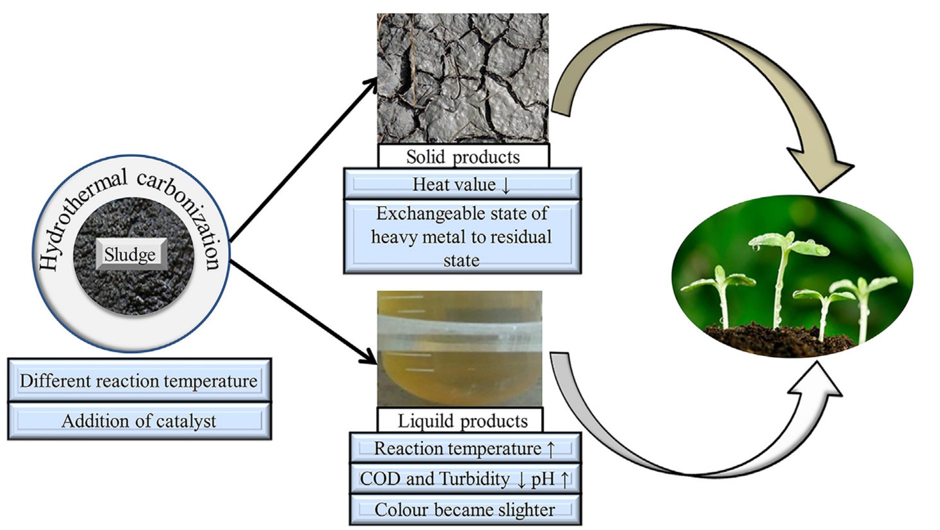
HIGHLIGHTS

- Urban sludge was harmless treated by hydrothermal carbonization (HTC).
- Solid productivity of urban sludge decreased from 92.04% to 52.65% by HTC.
- After HTC, the metal contents under exchangeable states reached discharge standard.
- The turbidity and COD of hydrothermal liquid decreased from 450° to 175° and 13 to 6.8 g/L.
- Solid productivity from pyrolysis is higher than HTC and metal exceeded limiting values.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 2 January 2017
Received in revised form 25 March 2017
Accepted 27 March 2017
Available online 30 March 2017

Keywords:
Hydrothermal carbonization
Urban sludge
Heavy metals
COD

ABSTRACT

Urban sludge was treated by Hydrothermal carbonization (HTC). The effect of hydrothermal carbonization temperature, mixing with or without catalysts on solid products yield, heavy metal contents, turbidity and COD value was evaluated. The result showed solid products yield decreased from 92.04% to 52.65% when the temperature increased from 180 to 300 °C. And the Cu, Zn, and Pb contents under exchangeable states decreased and reached discharge standard. Addition of $FeCl_3$ or $Al(OH)_3$ resulted in a significant increase in the exchangeable states of Zn, Pb, Cr, and Cd and decrease in their residual states. The turbidity and COD value of hydrothermal liquid decreased from 450° to 175°, and 13 to 6.8 g/L, with increasing hydrothermal temperature. Comparison with HTC, solid productivity from low-temperature pyrolysis is higher. The exchangeable states of Cu, Zn, and Cr exceeded the limiting values. Our results show HTC can facilitate transforming urban sludge into no-pollution and energy-rich products.

1. Introduction

It was reported that the amount of accumulated urban sludge in the country would be beyond 82,000 t/year dry weight from 2016 (Praspaliauskas and Pedišius, 2017; Kelessidis and Stasinakis,

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http://dx.doi.org/10.1016/j.biortech.2017.03.174
0960-8524/

2012; Li et al., 2010; Grøn, 2007). The urban sludge includes not only organic compounds and but also heavy metals (HMs), organic pollutant and pathogen (Zhai et al., 2016). Currently, most urban sludge is not properly treated, which is environmentally hazardous and increasing health risks (Shi and Kone, 2010). The effective utilization of sewage sludge are the emergency tasks for the fast growing populations and developing industry.

Thermal utilization of sewage sludge is considered as a promising method to deal with the sludge, especially, the hydrothermal carbonization (HTC). HTC is a promising thermochemical process to treat wet solid waste due to the relatively low temperature thermochemical treatment process (180–260 °C) and the acceptance for wide range of moisture content (Funke and Ziegler, 2010; Wikberg et al., 2015). The hydrothermal carbonization of sludge could reduce the cohesive force of sludge colloidal structure, damage microbial cell structures, improve the dehydration properties, and realize sludge reduction. Recent, the studies for the harmless treatment of sludge by HTC into the renewable resource or energy are hot debate all over the world (Zhang and Liu, 2016; Mariusz et al., 2016). Lin et al. (2016) have studied the resource utilization of municipal solid wastes for energy purposes by Hydrothermal carbonization (HTC). Koottatep et al. (2016) investigated HTC of process sludge collected from septic tanks into hydrochar for practical energy recovery. And study the influence of different temperatures and reaction times on the products in a 1L high-pressure reactor. Mau et al. studies the hydrothermal carbonization of poultry litter under a range of process parameters (Mau et al., 2016). Yao chose the activated sludge mixed with food waste as the resource to produce glucose and free amino nitrogen, equivalent to soluble chemical oxygen demand (SCOD) by anaerobic digestion (Yin et al., 2016). Zhai et al. (2016) investigated the effect of feedwater pH (pH = 2–12) on fate and risk of heavy metals (HMs) in hydrochars which was produced from the HTC of sewage sludge.

However, treating the urban sludge by HTC and pyrolysis, aiming to decrease the heavy metals, COD and the turbidity of hydrothermal liquid, has not been investigated. This study investigated harmless utilization of urban sludge by HTC and pyrolysis and focused on the study of chemical propriety of the solid and liquid products. Especially, the content of heavy metal in solid products, and COD and turbidity of hydrothermal liquid in order to get a no environmentally hazardous method for treatment urban sludge and get energy resource from sludge.

2. Methods

2.1. Materials

The urban sludge with moisture content of 80%, was obtained from Guangzhou Liede Sewage Treatment Plant, Guangdong Province, China. It was pretreatment by A2/O fabrication processing (hypoxia/anaerobic/aerobiotic). The urban sludge was preserved in 4 °C at the refrigerator before HTC.

2.2. Hydrothermal carbonization of sludge water

HTC were conducted in a high-pressure autoclave reactor (316 stainless steel) with a volume of 500 mL. The 60 g test sludge (moisture content 80%) and 120 mL deionized water were stirred into a slurry with a glass rod and then poured into the batch reactor. The sludge was stirred for 15 min with 160 rpm, and N_2 was replenished for 5 min. A 2.0 KW electric furnaces was used to heat the reactor and five different final temperatures was set: 180 °C, 210 °C, 240 °C, 270 °C, and 300 °C. And corresponding pressure was monitored by gas pressure meter during HTC experiments. Then the reactor was cooled to room temperature naturally. The required temperature was maintained for 1 h. The total heating time was recorded, and the reaction temperature and pressure were recorded every 15 min. After completion of the reaction, a syringe was used to absorb the liquid in the batch reactor. Quantitative filter paper was used for suction filtration, and solid and liquid products were collected. Without rinsing, solid products were dried at 80 °C for 24 h. Afterward, they were labeled and placed under sealed preservation. Liquid products were collected by using centrifugal tube and placed in a refrigerator at 4 °C for preservation.

2.3. Method for analysis of heavy metal content

Due to the similar physical-chemistry property of sludge and soil, the modified version of the BCR (European Communities Bureau of Reference) three-step sequential extraction procedure, was chosen to analyze the content of heavy metals in urban sludge (Mossop and Davidson, 2003; Zhai et al., 2014a). This paper mainly focused on exchangeable (F1) and residual (F5) states of heavy metals in solid products after hydrothermal carbonization of sludge.

Briefly, 0.1 g (dry basis) of samples (raw materials, 180 °C, 210 °C, 240 °C, 270 °C, 300 °C, 180 °C + $FeCl_3$, 180 °C + $Al(OH)_3$), which passed an 80 mesh sieve, were weighed and placed in eight 50 mL centrifuge tubes.

A drop of sulfuric and nitric acid solution, with a mass ratio of 2:1, was dissolved in 500 mL of deionized water (pH = 3.2). The sulfuric (10 mL) and nitric acid extraction solution was weighed and placed in a polyethylene bottle, which was placed in an oscillator for 16 h under indoor temperature of 22 ± 5 °C. The supernatant was carefully removed, the constant volume was regulated to 50 mL, and the solution was collected in a centrifuge tube. Atomic absorption spectroscopy analysis was conducted to detect Zn, Cu, Pb, Cr, and Cd contents in the leached liquid, that is, the exchangeable state of heavy metals in the solid products.

A total of 10 mL of H_2O_2 (8.8 mol/L) was poured in the centrifuge tube and allowed to stand for 5 min. The tube was covered with cap, which was not screwed down, shaken once every 15 min, and digested at room temperature for 1 h. The tube was then placed in a water bath at 85 ± 2 °C to digest for 1 h and shaken once every 15 min. The cap was then opened, and the solution was heated until the volume was reduced to below 3 mL. The tube was added with 10 mL of H_2O_2 (8.8 mol/L), covered, and shaken once every 15 min. The cap was removed, and the tube was heated continuously until the volume of the solution decreased to about 1 mL. Finally, the solution was placed in an oscillator at 22 ± 5 °C for 16 h. After complete digestion, the supernatant was discarded, oscillated, and rinsed for 15 min. The residue was added with 20 mL of deionized water. The supernatant was then discarded.

All residues in the centrifuge tube were transferred into a microwave digestion tank. The tube was added with 6 mL of high-concentration HNO_3 and 1.5 mL of H_2O_2 (8.8 mol/L) and allowed to stand for 10 min. After depressurization, the digestion tank was removed. A volumetric flask was used to measure a constant volume of 50 mL, and the liquid was collected in a 50 mL centrifuge tube. Atomic absorption spectroscopy analysis was conducted to detect Zn, Cu, Pb, Cr, and Cd contents in solid residues, that is, residue state (F5) of heavy metals in solid products.

3. Results and discussion

3.1. Productivity analysis of solid products

Solid products presented a granular shape in aqueous solution and natural sedimentation; solid and liquid were separated in two layers.