



Influence of sewage sludge-based activated carbon and temperature on the liquefaction of sewage sludge: Yield and composition of bio-oil, immobilization and risk assessment of heavy metals



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HIGHLIGHTS

- SSAC increased the yield and energy density of bio-oil at 350 °C.
- Liquefaction with SSAC redistributed the species of HMs in SR.
- RAC assessment indicates that HMs in SR-350 pose lower risk than SR-400.
- 350 °C with SSAC-550 favored the oil yield and risk decrease of HMs.

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ABSTRACT

The influence of sewage sludge-based activated carbons (SSAC) on sewage sludge liquefaction has been carried out at 350 and 400 °C. SSAC increased the yield and energy density of bio-oil at 350 °C. The metallic compounds were the catalytic factor of SSAC obtained at 550 °C (SSAC-550), while carbon was the catalytic factor of SSAC obtained at 650 °C. Liquefaction with SSAC redistributed the species of heavy metals in solid residue (SR). With the addition of SSAC, the risk of Cu, Zn and Pb decreased at 350 °C, while at 400 °C the risk of Cd, Cu, and Zn were decreased. Ecological risk index indicated that 400 °C was preferable for the toxicity decrement of SR, while risk assessment code indicated that SR obtained at 350 °C contained lower risk. Considering the bio-oil yield, liquefaction at 350 °C with SSAC-550 was preferable.

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1. Introduction

Sewage sludge (SS) from wastewater treatment plant, which is accompanied with unpleasant odors, pathogens, and heavy metals (HMs), has been deemed as a common pollution source (Huang et al., 2011; Li et al., 2012). Besides, accompanied with the high water content and large production, conventional treatment processes (e.g. agricultural application, landfilling) of SS are becoming increasing complex. Furthermore, the rigorous requirement of environmental protection has limited the application of conventional treatment processes and gives rise to the treatment of SS an urgent issue to tackle. On the other hand, due to the high contents of organic matter, nitrogen and phosphorus, SS has been viewed as a promising alternative source of energy. Hydrothermal reaction in supercritical water

is broadly defined as chemical and physical transformations in high temperature (200–700 °C) and high pressure (5–40 MPa). This thermochemical means has energetic advantages since when water is heated at high pressures a phase change to steam is avoid which avoids large enthalpic energy penalties. Several biomass hydrothermal conversion processes are in development now. Liquefaction processes are generally lower temperature (200–400 °C) reactions which produce liquid products, often called “bio-oil” or “bio-crude” (Peterson et al., 2008). Liquefaction of SS in sub-/supercritical water to generate bio-oil has received much attention (Gasafi et al., 2008; Vostrikov et al., 2008). By liquefying SS in sub-/supercritical water, both the disposal of SS and production of bioenergy can be achieved at the same time. Moreover, the extremely high water content (>80% by wet mass) of SS (Li et al., 2012), which has been identified as a main obstacle for conventional thermochemical treatment processes, turns out to be an advantage for the direct liquefaction of SS (Zhang et al., 2010).

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As the application of liquefaction increases in scope, effects of various operating parameters on the increase of energy output from liquefaction of SS have been systematically studied along with its countermeasures (Starl et al., 2006; Yanik et al., 2008). Due to the low-cost, abundance, availability and non-toxicity of activated carbon (AC), it has been used as catalyst directly or supporter for noble metals in supercritical water reaction (Matsumura et al., 1997; Nunoura et al., 2002; Xu et al., 1996). While these studies mainly focused on the gasification or oxidation of organic compounds, only few published literatures focused on the application of AC in supercritical water liquefaction of SS, not to mention the application of sewage sludge-based activated carbon (SSAC). SSAC, or biochar, is the solid byproduct from pyrolysis of SS. It is rich in carbon and HMs and can be used to prepare AC directly or after further activation. Considering the catalytic effect of AC in supercritical water reaction and the properties of SSAC, the addition of SSAC to accelerate the liquefaction of SS is a possibility. Moreover, as the by-product of pyrolysis of SS, the production of SSAC itself is economical.

In addition to studying energy output from SS conversion, the goal of cleaner production has sparked more and more interest in studies on the characteristics of the by-products from liquefaction, especially HMs in the solid residue (SR) (Xu and Lancaster, 2008). Huang et al. (2011) quantitatively evaluated the potential ecological risk and pollution degrees of HMs in SRs from the liquefaction of SS at 320 °C with acetone as solvent. Li et al. (2012) investigated the characteristics of HMs in SRs from sub-(375 °C) and supercritical water (400 °C) gasification without additives. HMs had been pointed out to accumulate in SR after liquefaction (Shi et al., 2013). Apart from these scattered findings on the bioavailability and toxicity of HMs in SR, the essential knowledge on the distribution and assessment of HMs in SRs from SS liquefaction with additives is unfortunately lacking.

In light of the existing work, the objectives of this study are to study the effect of SSAC on the yield and composition of bio-oil from SS liquefaction in sub-/supercritical water at the temperature of 350 °C and 400 °C as well as the immobilization of HMs in SR. In order to evaluate the risk of HMs in SR, the geo-accumulation index (I_{geo}) and overall total potential ecological risk index (RI) of HMs based on distribution of the chemical species' distribution are applied, as well as the risk assessment code (RAC).

2. Methods

2.1. Materials preparation

The feedstock used in this work was a dewatered SS supplied by the municipal wastewater treatment plant in Changsha, China. In order to control the ration of SS and water with accuracy and consistency, SS was dried completely in an oven kept at 105 °C for at least 24 h, and subsequently sieved into fine particles (<150 μm). SSAC used in this work was obtained by pyrolyzing SS in a horizontal furnace at 550 °C and 650 °C with retention time of 60 min, 90 min, and 120 min, respectively. Six kinds of SSACs were applied in the liquefaction experiments in order to obtain a full scale investigation. The resulting SSAC was called as X–Y, where X stands for the pyrolysis temperature, Y represents the retention time. Therefore, SSACs are called as 550–60, 550–90, 550–120, 650–60, 650–90, and 650–120.

Prior to the experiment, elemental analysis (2400 Series II CHNS Analyzer, PerkinElmer, USA), and distribution of HMs (Cd, Cu, Zn, and Pb) in SS (characterized by BCR extraction procedures) were conducted. Both the content of C, H, S, N, and O and contents of HMs of the four fractions F1, F2, F3, and F4 which will be introduced in detail in Section 2.4.1 are shown in Table 1. Cd and Zn

Table 1
Contents of main elements and distribution of heavy metals (HMs) in SS.

Elements ^a	Contents (wt%)	HMs	F1 (mg/kg) ^c	F2 (mg/kg) ^c	F3 (mg/kg) ^c	F4 (mg/kg) ^c
C	28.94	Cd	6.49	15.11	5.58	0.28
H	4.48	Cu	15.02	40.58	97.53	6.08
N	4.23	Pb	15.23	13.87	28.15	254.81
S	0.65	Zn	107.67	904.33	40.47	69.45
O ^b	13.92					

^a Volatile basis.

^b Determined by the mass difference.

^c Dry basis.

were mainly in F2, which accounted for 55.03 wt% and 80.61 wt%, respectively. Cu was mainly in F3 (63.26 wt%), and Pb was highly stabilized in F4 (81.66 wt%). The bioavailable fraction (F1+F2) of Cd, Cu, Zn and Pb were 78.66 wt%, 34.92 wt%, 90.21 wt%, and 9.32 wt%, respectively. This indicates that both Cd and Zn were highly toxic to environment, Cu was in potential toxicity level, and Pb had low directly toxicity.

2.2. Liquefaction procedure

Liquefaction experiments were conducted in a high-pressure autoclave reactor (316 stainless steel) with a volume of 500 mL. The reactor is exactly the same as the reactor used in other research work (Li et al., 2012; Xu et al., 2012), which can be operated at a temperature of 450 °C and pressure of 35 MPa.

153 mL of deionized water and 17 g of SS powder were loaded into the reactor after homogenous mixing. For the experiment with catalyst, 3.4 g of SSAC was loaded. A 4.0 kW electric furnace was used to heat the reactor to a preset temperature (350 °C or 400 °C) at a proximately heating rate of 3.33 °C/min. Once the desired temperature was reached, it was maintained for the reaction time of 30 min. After the heating was stopped, the reactor was cooled to room temperature with the cooling coil.

The solid and liquid products were removed completely from the reactor and separated using a vacuum filtration apparatus with a 0.45 μm membrane filter. The SR on the filter was dried at 90 °C for 24 h and collected for characterization. Oil in the liquid was extracted with dichloromethane, which was latter evaporated to obtain the oil products. The SRs obtained are called as M–N, where M stands for the liquefaction temperature; N represents the type of SSAC. For example, SRs obtained at 350 °C with SSAC-550 are called as 350-550-60, 350-550-90, 350-550-120. In particular, the liquefaction of SS without SSAC are named as 350-B and 400-B, where B represents for blank.

2.3. Analysis of bio-oils

Oil analysis was carried out with gas chromatograph (GC) equipped with a mass selective (MS) detector (GC–MS, QP2010, SHIMADZU, column: Rtx-5MS, 30 m × 0.25 mm × 0.25 μm). Ethanol (GR) was used as the solvent and helium (5 mL/min) was the carrier gas. The interface was kept at 250 °C, and the ion source was kept at 230 °C. The oven temperature program was as follows: 40 °C (hold for 2 min), increase to 190 °C (12 °C/min, hold for 1 min), up to 290 °C (4 °C/min, hold for 20 min), up to 320 °C (20 °C/min, hold for 5 min). The MS system was operated in the full scan mode with a mass range from m/z 50 to 700. Chromatograms of the compounds in the oil were compared with the standard compounds in the NIST mass spectral data library, which generated all the information available for the compounds.

Higher calorific values (HHV) of the bio-oils were monitored with a bomb calorimeter (SADCM5000, SUNDY). The calorific value

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