



Study on demetalization of sewage sludge by sequential extraction before liquefaction for the production of cleaner bio-oil and bio-char



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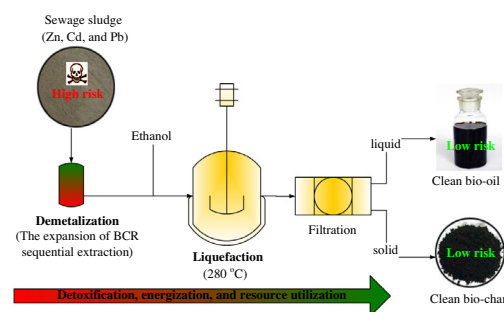
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HIGHLIGHTS

- Demetalization of SS by a modified BCR sequential extraction before liquefaction.
- Demetalization had little effect on oil quantity, but had positive effect on quality.
- Bio-oil and bio-char produced after demetalization pose low environmental risk.
- Removal of F1 and F2 fractions of heavy metals before liquefaction is recommended.
- The expansion of BCR can be widely used to study the heavy metal in other processes.

GRAPHICAL ABSTRACT



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ABSTRACT

Demetalization of sewage sludge (SS) by sequential extraction before liquefaction was implemented to produce cleaner bio-char and bio-oil. Demetalization steps 1 and 2 did not cause much organic matter loss on SS, and thus the bio-oil and bio-char yields and the compositions of bio-oils were also not affected significantly. However, the demetalization procedures resulted in the production of cleaner bio-chars and bio-oils. The total concentrations and the acid soluble/exchangeable fraction (F1 fraction, the most toxic heavy metal fraction) of heavy metals (Cu, Cr, Pb, Zn, and Cd) in these products were significantly reduced and the environmental risks of these products were also relieved considerably compared with those produced from raw SS, respectively. Additionally, these bio-oils had less heavy fractions. Demetalization processes with removal of F1 and F2 fractions of heavy metals would benefit the production of cleaner bio-char and bio-oil by liquefaction of heavy metal abundant biomass like SS.

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1. Introduction

Sewage sludge (SS), the main byproduct from biological wastewater treatment, is one of the promising biomass that is inevitably produced in huge quantities (Wang et al., 2015a). China

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generated more than 30 million metric tonnes of sewage sludge (at a moisture content of 80%) in 2012, and this is expected to rise to 34 million metric tonnes in 2015 (Feng et al., 2015). SS mainly consists of non-toxic organic matter which has high energy and therefore can be utilized as a biomass resource for energy recovery (Manara and Zabaniotou, 2012; Tyagi and Lo, 2013). Energy production from sewage sludge via liquefaction seems of great potential as its main product (bio-oil) can be used as bio-fuel (Huang and Yuan, 2015; Huang et al., 2013; Leng et al., 2015a; Li et al., 2010; Manara and Zabaniotou, 2012; Tyagi and Lo, 2013) and its byproduct can also be used for environmental application (e.g. as adsorbent (Leng et al., 2015b,c)).

Except the non-toxic organic matter, SS also contains toxic organic compounds, macronutrients, micronutrients, organic micropollutants, microorganisms, parasitic eggs, and heavy metals (Chen et al., 2008; Yuan et al., 2011; Zhao et al., 2014). These properties may hinder its utilization or application. For example, the high contents of O, N, and S may probably result in their high contents in the corresponding bio-oils, which will lead to the formation of more NO_x and SO₂ when combusted (Manara and Zabaniotou, 2012; Park et al., 2010). Transferring O, N, and S containing chemicals into gas or solid phase by adding catalysts or regulating process parameters may be possible ways to mitigate this problem (Manara and Zabaniotou, 2012). In addition to that, heavy metal is another most concerned issue that has been studied. Heavy metals such as Cu, Zn, Cr, Cd, Ni, Hg, and Pb are common in SS (Alvarez et al., 2002, 2015), but Cu, Zn, Cr, Cd, and Pb are the most studied ones. The immobilization and transformation behavior of these heavy metals during liquefaction of SS has been studied by several researchers (Chen et al., 2014; Huang et al., 2011; Leng et al., 2014; Shi et al., 2014, 2013; Yuan et al., 2011; Zhai et al., 2014). Heavy metals in SS can be largely concentrated in the bio-char during liquefaction, with the mobile and easily available heavy metal fractions (acid soluble/exchangeable and reducible fractions) being transformed into the relatively stable form (oxidizable and residual fractions) (Huang et al., 2011; Leng et al., 2014; Yuan et al., 2011; Zhai et al., 2014). Thus, the pollution levels of heavy metals in bio-char were markedly mitigated after liquefaction (Chen et al., 2014; Hu et al., 2015; Huang et al., 2011; Leng et al., 2014; Shi et al., 2013). However, attention should also be paid to the fact that part of the organic matter bound to heavy metals in bio-char can be degraded under oxidizing conditions which may lead to the release of heavy metals (Yuan et al., 2011). In addition, heavy metals transferred to bio-oil was observable with large part being the mobile and easily available fraction, and showed potential environmental risk (Yuan et al., 2015). Therefore, the production of clean bio-oil and bio-char has become one of the most important issues in the liquefaction of SS.

There may be three ways to produce cleaner bio-char and bio-oil from heavy metals rich biomass like SS by liquefaction: (i) removal or immobilization of heavy metals in SS before liquefaction (by pretreatment method such as water-washing, extraction); (ii) removal or immobilization of heavy metals in bio-char and bio-oil after liquefaction (e.g. by thermochemical treatment with gaseous hydrochloric acid (Vogel and Adam, 2011) and water-washing (Wang et al., 2015b)); and (iii) immobilization of heavy metals during liquefaction of SS (e.g. by hydrothermal upgrading (Yang, 2010; Yang et al., 2010) and co-liquefaction (Chen et al., 2014; Shi et al., 2013)). This article focuses on investigations on demetalization of heavy metals in SS by an extraction process before liquefaction in order to produce cleaner bio-char and bio-oil; and also focuses on the study of the heavy metal immobilization and transformation behavior during liquefaction of treated SS.

2. Methods

2.1. Materials

Dewatered SS sample was obtained from a municipal wastewater treatment plant in Changsha, Hunan Province. The SS was dried in an oven at 105 °C for 24 h ground and screened into fractions of particle diameter 30–120 meshes.

2.2. Demetalization procedure (extraction process)

In the 1990s, the Community Bureau of Reference (BCR) developed an evaluation program of the sequential extraction schemes for determining metals in soils and sediments, which is recognized as the best available method for gaining information on heavy metals (Pan et al., 2009). In this study, the analytical method BCR was modified and used as a demetalization process (the expansion of BCR) to pretreat SS.

Demetalization step one: the extraction of acid soluble/exchangeable fraction (F1, exchangeable metal and carbonate-associated fractions). 5.0 g SS were introduced into a 500 mL Erlenmeyer flask containing 200 mL of acetic acid (0.1 mol/L) and then shaken for 16 h at room temperature (9 parallel samples). The solution and solid phases were separated by filtration and then the solid residues of 3 samples were dried at 105 °C for 24 h and preserved for liquefaction (the dried solid obtained in step one was titled SS1). The solid residues of the other 6 samples were not dried and were preserved for subsequent extraction.

Demetalization step two: the extraction of reducible fraction (F2, fraction associated with Fe and Mn oxides). Each solid residue from demetalization step one was introduced into a 500 mL Erlenmeyer flask containing 200 mL volume of 0.1 mol/L hydroxylammonium chloride (adjusted to pH 2 with nitric acid) for 16 h. The solution and solid phases were separated by filtration and then the solid residues of 3 samples were dried at 105 °C for 24 h and preserved for liquefaction (the dried solid obtained in step two was titled SS2). The solid residues of the other 3 samples were not dried and were preserved for subsequent extraction.

Demetalization step three: oxidizable fraction (F3, fraction bound to organic matter). Each solid residue from demetalization step two was introduced into a 500 mL Erlenmeyer flask and was dispersed in 50 mL volume of hydrogen peroxide (30%) and digested at room temperature for 1 h with occasional shaking. Other 50 mL of hydrogen peroxide was introduced and digested at 80 °C (water bath) for 1 h. Then the contents were evaporated to a small volume (5–10 mL). About 250 mL of ammonium acetate (1.0 mol/L, adjusted to pH 2 with nitric acid) was added to the cool and moist residue, and then shaken and centrifuged. The solution and solid phases were separated by filtration and the solid residues were dried at 105 °C for 24 h and preserved for liquefaction (the dried solid obtained in step three was titled SS3).

The solid mass recovery (SR) was calculated based on raw SS.

$$SR = M_{\text{solid residue}}/M_{\text{SS}} \times 100\% \quad (1)$$

where $M_{\text{solid residue}}$ is the mass of the dried solid obtained in each step of the demetalization process; M_{SS} is the mass of the raw SS.

2.3. Liquefaction process

Liquefaction of SS, SS1, SS2, and SS3 was conducted in a 500 mL airtight autoclave (GSHA-0.5, China) at 280 °C. In each test, 8.0 g sample was mixed with 100 mL of ethanol and heated to 280 °C which was maintained for 20 min, then cooled down to the room temperature. After each test, the solid/liquid products were rinsed from the autoclave by washing with ethyl acetate and filtered

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