



# Beneficial synergistic effect on bio-oil production from co-liquefaction of sewage sludge and lignocellulosic biomass



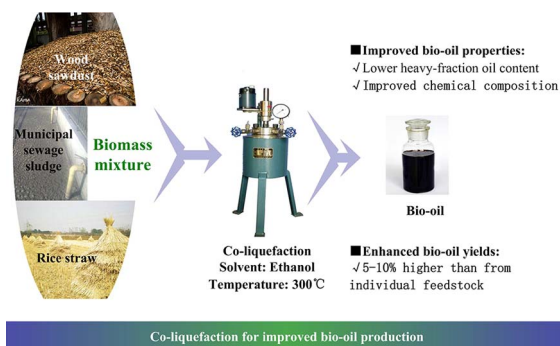
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## GRAPHICAL ABSTRACT



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## ABSTRACT

Co-liquefaction of municipal sewage sludge (MSS) and lignocellulosic biomass such as rice straw or wood sawdust at different mixing ratios and the characterization of the obtained bio-oil and bio-char were investigated. Synergistic effects were found during co-processing of MSS with biomass for production of bio-oil with higher yield and better fuel properties than those from individual feedstock. The co-liquefaction of MSS/ rice straw (4/4, wt) increased the bio-oil yield from 22.74% (bio-oil yield from liquefaction of MSS individually) or 23.67% (rice straw) to 32.45%. Comparable increase on bio-oil yield was also observed for MSS/wood sawdust mixtures (2/6, wt). The bio-oils produced from MSS/biomass mixtures were mainly composed of esters and phenols with lower boiling points (degradation temperatures) than those from individual feedstock (identified with higher heavy bio-oil fractions). These synergistic effects were probably resulted from the interactions between the intermittent products of MSS and those of biomass during processing.

## 1. Introduction

Municipal sewage sludge (MSS), the main solid waste (a byproduct) produced in biological wastewater treatment processes, has drawn

increasing attentions in recent years for finding suitable utilization and/or disposal methods. Conventional MSS treatment technologies such as landfilling, incineration or ocean disposal is becoming less applicable since the banning of ocean disposal and the increasingly stringent

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landfilling and incineration criteria. The utilization of MSS as a renewable raw material for sustainable energy recovery is becoming more and more attractive because of its advantages of disposing the waste, and meanwhile, generating valuable resources such as bio-oil, biodiesel, solid briquette, or biogas, etc. (He et al., 2014; Manara and Zabaniotou, 2012; Tyagi and Lo, 2013).

Thermochemical liquefaction is one of the promising technologies for producing bio-oil (Huang and Yuan, 2015; Manara and Zabaniotou, 2012), which is a process under mild temperature (250–400 °C) and high pressure (5–20 MPa) with the participation of solvent such as water and/or other organic solvents in the presence or absence of catalyst (Huang and Yuan, 2015; Mochida et al., 2014). Liquefaction of MSS has been studied by several researchers concentrating on optimizing the liquefaction parameters for high bio-oil yield. Higher bio-oil yield was obtained when MSS was liquefied with ethanol as the solvent at higher temperature and solvent filling ratio (Li et al., 2010). Huang et al. received higher bio-oil yield with methanol or ethanol than with acetone as the solvent (Huang et al., 2013a). Catalysts such as KOH, FeS and HCO<sub>2</sub>H (Zhang et al., 2011), and MSS-based activated carbon (Zhai et al., 2014) can also be effective on enhancing bio-oil production. On the other hand, co-liquefaction of MSS with other biomass could probably be another promising method to enhance bio-oil production note that co-liquefaction of biomass mixtures can increase bio-oil yield and improve bio-oil qualities (Dandamudi et al., 2017; Gai et al., 2015; Jasiūnas et al., 2017; Jin et al., 2013; Yang et al., 2017; Zhang et al., 2011). For example, co-liquefaction of microalgae and rice husk resulted in increased bio-oil yield and decreased acidity and nitrogen content of bio-oil (Gai et al., 2015). The severe reaction conditions was alleviated and deoxygenation of bio-oil was promoted after co-liquefaction of micro- and macro-algae (Jin et al., 2013). In addition, viscosity and relative molecular mass were reduced when spent coffee grounds and lignocellulosic feedstocks were co-liquefied (Yang et al., 2017).

Co-liquefaction of MSS with *Camellia oleifera* cake was beneficial for obtaining high bio-oil yield; but only very limited information about the bio-oil from co-liquefaction was reported since these studies focused on heavy metal transformation behavior research (Chen et al., 2014; Zhai et al., 2015). In another study, phenol and alanine were used as model compounds to study the interactions between lignin (main component of lignocellulosic biomass) and proteins (the main component of MSS) during the co-treatment in supercritical water (Su et al., 2015). Co-processing of lignin and proteins promoted the production of gas (H<sub>2</sub>) and oil phase (Su et al., 2015). In the present study, co-liquefaction of MSS and typical lignocellulosic biomass (agriculture waste rice straw or wood processing waste wood sawdust) was implemented and detailed information about the yield and properties of the liquefaction products was reported.

## 2. Materials and methods

### 2.1. Materials

Dewatered MSS sample was obtained from a local wastewater treatment plant in Changsha, Hunan Province, Southern China. Wood (Chinese fir) sawdust and rice straw were collected from a local furniture factory and the local farmland, respectively. These raw materials were dried in an oven at 105 °C until constant weight achieved. Then, they were pulverized and screened into fractions with particle diameter less than 0.45 mm.

### 2.2. Liquefaction process

Liquefaction was conducted in a 500-mL airtight autoclave (GSHA-0.5, China) at 300 °C. In each test, 8.0 g dried feedstock was mixed with 100 mL of ethanol and heated to 300 °C, which was maintained at this temperature for 20 min; then the autoclave was cooled down to the

room temperature. The solid and liquid products were separated and collected by the method reported in Ref. (Leng et al., 2016, 2015). In brief, solid biochar was obtained after filtering the reactant matrix; bio-oil was then collected from evaporating the filtered liquid portion under reduced pressure at 50 °C and 90 °C to remove the solvent (including unreacted ethanol and washing solvent ethyl acetate) and water, respectively. Products from liquefaction of MSS/rice straw mixture of ratios 8:0, 6:2, 5:3, 4:4, 3:5, 2:6, and 0:8 were labeled M8, M6R2, M5R3, M4R4, M3R5, M2R6, and R8, respectively. Similarly, M8, M6W2, M5W3, M4W4, M3W5, M2W6, and W8 were used for products produced from MSS and wood sawdust mixtures.

The experimental bio-oil/bio-char yields were calculated from dividing the weight of bio-oil/bio-char obtained above by the weight of raw sample (8 g). The theoretical bio-oil/bio-char yields of the MSS/rice straw (or wood sawdust) mixtures were calculated from the mixture ratios and the experimental yields from the individual liquefaction of MSS and rice straw (or sawdust) based on the assumption that no synergies existed during co-liquefaction.

### 2.3. Bio-oil characterization

#### 2.3.1. Elemental analysis

The elemental compositions of raw materials and some specific bio-oils obtained from liquefaction were analyzed by an elemental analyzer vario MACRO cube (Elementar, German).

#### 2.3.2. GC-MS analysis

The chemical composition of the bio-oil was analyzed by the method reported in Ref. (Leng et al., 2015), using gas chromatography-mass spectroscopy (GCMS-QP2010 Plus spectrometer; column: Rtx-5MS, 5% biphenyl + 95% dimethyl polysiloxane, 30 m × 0.25 mm × 0.25 μm; Shimadzu, Japan) with ethanol as the solvent, helium (5 mL min<sup>-1</sup>) as the carrier gas. GC program: 40 °C (hold 2 min) → 190 °C (8 °C min<sup>-1</sup>, hold 2 min) → 290 °C (6 °C min<sup>-1</sup>, hold 5 min). National Institute of Standards and Technology (NIST) mass spectral library was used to identify the compounds detected.

#### 2.3.3. Thermogravimetric analysis

Thermogravimetric analyses (TGA) of raw feedstocks and the corresponding bio-oils and bio-chars were carried out on thermo-balance DTG-60 (Shimadzu, Japan) with nitrogen (purity of 99.99%, flow rate of 100 mL min<sup>-1</sup>) as the purge gas, heating from room temperature to 800 °C at 20 °C min<sup>-1</sup>.

## 3. Results and discussions

### 3.1. Characterization of raw materials

As seen in Table 1, while MSS had very high ash yield (35.62%) compared with rice straw (11.56%) and wood sawdust (1.61%), its carbon and hydrogen contents were significantly lower, which means its lower energy content than the lignocellulosic biomasses. However, the oxygen content of the three raw materials was all as high as 40%, which requires deoxy-liquefaction to reduce the oxygen content for desirable biofuel production.

The TG and DTG curves of the three biomasses seen in Fig. 1 also show that the MSS had the largest weight loss while the sawdust was almost decomposed after temperature being raised to 800 °C. Rice straw had the largest mass lose rate with a peak approximately 0.037%/min at about 370 °C. Its main decomposition phase ranged from 200 to 500 °C while that of sawdust had a more narrowed range of 250–400 °C. However, MSS had two decomposition phases which were 200–500 °C and 650–800 °C with the peak mass lose rate of about 0.008%/min found at about 750 °C. The TG/DTG curves suggest that rice straw and wood sawdust were much easier to decompose (pyrolyze) than MSS. Since pyrolysis is the first stage of liquefaction and the liquefaction is

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