



An approach for upgrading biomass and pyrolysis product quality using a combination of aqueous phase bio-oil washing and torrefaction pretreatment



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HIGHLIGHTS

- A novel approach was performed to upgrade the quality of biomass pyrolysis products.
- Aqueous phase bio-oil washing pretreatment removed metals from the sample.
- Torrefaction reduced the oxygen but increased the metal content of cotton stalk.
- 3D FTIR results were consistent with the pyrolysis stages of the TG/DTG curve.
- Combined pretreatment reduced water and acid and increased phenol content of bio-oil.

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ABSTRACT

Bio-oil undergoes phase separation because of poor stability. Practical application of aqueous phase bio-oil is challenging. In this study, a novel approach that combines aqueous phase bio-oil washing and torrefaction pretreatment was used to upgrade the biomass and pyrolysis product quality. The effects of individual and combined pretreatments on cotton stalk pyrolysis were studied using TG-FTIR and a fixed bed reactor. The results showed that the aqueous phase bio-oil washing pretreatment removed metals and resolved the two pyrolysis peaks in the DTG curve. Importantly, it increased the bio-oil yield and improved the pyrolysis product quality. For example, the water and acid content of bio-oil decreased significantly along with an increase in phenol formation, and the heating value of non-condensable gases improved, and these were more pronounced when combined with torrefaction pretreatment. Therefore, the combined pretreatment is a promising method, which would contribute to the development of polygeneration pyrolysis technology.

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1. Introduction

Fast pyrolysis of biomass for bio-oil production is one of the most promising technologies used for the utilization of biomass resources. Although bio-oil can be directly used for industrial furnace combustion, it is still a low-grade liquid fuel because of its inferior quality compared to those of traditional fossil fuels (Lehto et al., 2014; No, 2014). The low quality of the bio-oil is due to its poor stability, phase separation propensity, high water

and acid content, low heating value, and complicated composition. In fact, low quality of biomass pyrolysis products directly results from low quality of biomass raw materials (Chen et al., 2017). Therefore, an effective approach is required to enhance the quality of biomass raw materials, which can thereby improve the quality of the pyrolysis products.

It should be noted that bio-oil, and in particular, the aqueous phase bio-oil produced after bio-oil phase separation, contains a high amount of water and organic acids (Chen et al., 2016a). Since phase separation of bio-oil is almost inevitable, it may be worthwhile to use the aqueous and oil phase bio-oil fractions separately. Aqueous phase bio-oil has high water and acid contents and small amounts of ketones and phenols, which hinder its use as a fuel or

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as a source of raw chemical materials (Chen et al., 2014c). Previous studies have shown that organic acids can effectively remove metallic species from biomass, and the phenolic substances in bio-oils have also been helpful in removing metallic species (Karnow et al., 2014; Oudenhoven et al., 2013; Pecha et al., 2015; Zhang and Xiong, 2016). In this case, washing the raw biomass with aqueous phase bio-oil was considered as a promising pretreatment method to improve the quality of the biomass and consequently the quality of the biomass pyrolysis products.

Additionally, torrefaction pretreatment at 200–300 °C in an inert gas atmosphere has also been an effective method for biomass pretreatment (Chen et al., 2015b). Torrefaction pretreatment significantly removes oxygen from biomass, increases the heating value, and improves the grinding properties of biomass (Arias et al., 2008; Chen et al., 2015c,d; Gil et al., 2015). More importantly, it efficiently reduces the acid and water content while increasing the heating value of the bio-oil (Zhang et al., 2015; Zheng et al., 2012, 2013). However, torrefaction pretreatment has certain limitations such as a reduced yield of bio-oil because of an increase in ash and metal content in the torrefied biomass and catalytic cracking of some high-value compounds to small molecule compounds (Chen et al., 2017; Zheng et al., 2015b). The combination of aqueous phase bio-oil washing and torrefaction pretreatments can harness the advantages of each and improve the quality of both the biomass and the pyrolysis products.

Thermogravimetry-Fourier transform infrared spectroscopy (TG-FTIR) has been widely used to study the characteristics of biomass pyrolysis. It can be used to detect weight loss during biomass pyrolysis, and most importantly, it can be used to detect the emission characteristics of the volatile components from pyrolysis in real time (Chen et al., 2014b). In addition, a fixed bed reactor has also been widely used to collect the solid, liquid, and gaseous products of biomass pyrolysis for separate analysis. However, to our knowledge, investigations using TG-FTIR and a fixed bed reactor to assess the effects of aqueous phase bio-oil washing and torrefaction pretreatments on pyrolysis have not been reported.

In this study, an approach that combines aqueous phase bio-oil washing and torrefaction pretreatment was used to upgrade cotton stalk. The effects of the aqueous phase bio-oil washing pretreatment, the torrefaction pretreatment, and the combined pretreatment on the quality of the pyrolysis products of cotton stalk were compared. The goal of this work was to provide data highlighting the application of aqueous phase bio-oil in the polygeneration pyrolysis of biomass.

2. Materials and methods

2.1. Materials

Cotton stalks were used as the experimental material. It was screened into particles with a size 40–60 mesh and dried at 105 °C for 6 h.

2.2. Aqueous phase bio-oil washing

During storage, phase separation of the bio-oil occurred. The originally homogeneous bio-oil gradually divided into two fractions: the aqueous phase and the oil phase. In this study, the aqueous phase bio-oil used to wash the cotton stalks was obtained from the pyrolysis of cotton stalks at 500 °C. The details of the pyrolysis process were as reported in the literature (Chen et al., 2014a). The water content of the aqueous phase bio-oil was 73.18 wt.%. Certain organic compounds with relatively high peak areas were detected using gas chromatography/mass spectrometry (GC/MS). These included acetic acid, hydroxyacetone, 1-hydroxy-2-butanone,

2-cyclopentenone, 1,2-cyclopentanedione, furfural, 2-furan-methanol, 2,3-dihydro-benzofuran, phenol, 2-methyl-phenol, 2-methoxy-phenol, 1,2-benzenediol, 2,6-dimethoxyphenol, and guaiacol. The peak area% of the acids, ketones, furans, and phenols were recorded as 32.28%, 16.39%, 9.46%, and 21.73%, respectively.

For the washing pretreatment, a cotton stalk sample (10 g) was added to a beaker containing 200 mL of aqueous phase bio-oil and stirred at 60 °C for 2 h. After washing, the cotton stalk sample was filtered, rinsed with deionized water until reaching a neutral pH, and dried at 105 °C for 12 h.

2.3. Torrefaction

Torrefaction experiments of cotton stalk samples were performed at 260 °C for 30 min using a tube furnace (OTL1200, Nanjing University Instrument Plant, China). Nitrogen was used as the carrier gas with a flow rate of 300 mL/min. Details of the torrefaction process were as reported in our previous study (Chen et al., 2017).

2.4. Sample labels

The dried cotton stalk was denoted as CS. The stalk pretreated with aqueous phase bio-oil washing was denoted as W-CS, and the one pretreated with torrefaction was denoted as T-CS. The stalk pretreated sequentially with aqueous phase bio-oil washing and torrefaction was denoted as TW-CS.

2.5. TG-FTIR experiments

TG-FTIR analysis of CS, W-CS, T-CS, and TW-CS were performed using a thermogravimetric analyzer (TGA Q500, TA Instrument, USA) connected to a Fourier-transform infrared spectrometer (Nicolet 6700, Thermo Scientific, USA). The amount of cotton stalk used for each experiment was 20 mg. The temperature was increased from 50 °C to 650 °C at a rate of 10 °C/min. The nitrogen flow rate was 70 mL/min. The IR spectra were recorded at 4000–400 cm^{-1} with a resolution of 1 cm^{-1} , and 3D FTIR spectrograms were obtained after the experiments.

2.6. Pyrolysis

Pyrolysis of each CS, W-CS, T-CS, and TW-CS was performed using a fixed bed pyrolysis reactor with 5 g of sample at 500 °C for 15 min with a nitrogen flow rate of 300 mL/min for each experiment. The detailed experimental process was described in a previous study (Chen et al., 2015a). After the experiment, solid (biochar), liquid (bio-oil), and gaseous (non-condensable gases) products were collected. The mass yields of biochar and bio-oil were obtained by mass measurements and that of the non-condensable gases was obtained from the difference of masses.

2.7. Product characterization

The non-condensable gases were detected using a gas chromatography analyzer (GC-TCD 7890, Shanghai Tianmei, China), and the high heating value (HHV) was calculated approximately by summing each gas concentration with its corresponding HHV. The water content of the bio-oil was determined via Karl-Fischer titration, and the HHV was measured using an adiabatic oxygen bomb calorimeter (XRY-1A, Changji Geological Instruments, China). The pH of the bio-oil was measured using a digital pH meter (PHS-3C, Shanghai leici, China), and the organic components were determined by gas chromatography coupled to a mass spectrometer (GC/MS 7890A/5975C, Agilent Company, USA). Proximate analysis of the solid samples was performed according to the Chi-

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