



Effects and mechanism of ball milling on torrefaction of pine sawdust



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HIGHLIGHTS

- The structure of pine sawdust was destroyed into irregular granules by ball milling.
- Ball milling reduced the thermal stability of hemicellulose, cellulose, and lignin.
- Torrefied ball-milled samples had higher heating values than hammer-milled samples.
- Biomass pretreated by ball milling and torrefaction can produce good solid fuels.

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ABSTRACT

The effects and mechanism of ball milling on the torrefaction process were studied. Ball- and hammer-milled (screen size 1 mm) pine sawdust samples were torrefied at three temperatures (230, 260, and 290 °C) and two durations (30 and 60 min) to investigate into their torrefaction behavior and physico-chemical properties. The results showed that, under identical torrefaction conditions, torrefied ball-milled pine sawdust had a higher carbon content and fixed carbon, and lower hydrogen and oxygen contents than torrefied hammer-milled pine sawdust. Torrefied ball-milled pine sawdust produced lower mass and energy yields, but higher heating values than torrefied hammer-milled pine sawdust. Ball milling destroyed the crystalline structure of cellulose and thus reduced the thermal stability of hemicellulose, cellulose, and lignin, causing them to degrade at relatively lower temperatures. In conclusion, biomass pretreated with a combination of ball milling and torrefaction has the potential to produce an alternative fuel to coal.

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1. Introduction

With the depletion of fossil fuels and increasing concern over environment, the research on biomass has attracted attention around the world in recent years. Biomass is considered as an important renewable resource that could be converted into fuels and chemical feedstock by various pretreatment methods (Demirbas, 2009). When biomass is to be processed thermochemically, torrefaction and hydrothermal carbonization are main methods to improve its energy value. Generally, torrefaction is more popular for biomass with low moisture contents, while hydrothermal carbonization is more suitable for municipal waste, sewage slug, animal manures, and biomass residue with high moisture contents (Acharya et al., 2015).

Torrefaction is a thermal pretreatment for biomass in which raw biomass is heated to 200–300 °C in an inert atmosphere to improve its performance as a solid fuel (Tran et al., 2013). The main benefits of torrefaction include higher energy density and hydrophobicity, lower atomic O/C and H/C ratios, and improved grindability and reactivity (Chen et al., 2015). Torrefaction can be classified into light (200–235 °C), mild (235–275 °C), and severe (275–300 °C) processes (Chen and Kuo, 2011). Temperature and residence time (duration) are influential factors in the torrefaction process, while biomass particle size also affects the reaction rate. Generally, torrefaction time is controlled within 1 h because the reaction rate becomes very slow at longer durations (Chen and Kuo, 2010; Chew and Doshi, 2011). The degree of thermal degradation of biomass constituents increases at increased temperature and residence time (Li et al., 2015). Prins et al. (2006) postulated that for sufficiently small particles (<2 mm), the impact of intra-particle heat and mass transfer became insignificant. Peng et al. (2012) showed the internal diffusion of vapors generated inside particles

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affects the overall torrefaction reaction rate, despite the temperature gradient inside particles smaller than 1 mm being very small during torrefaction. Overall, the order of significance of parameters that affect the torrefaction process is: temperature > residence time > particle size (Bridgeman et al., 2010).

Thermal stability of components in biomass (mainly hemicellulose, cellulose, and lignin) has great effects on torrefaction process. Hemicellulose is an amorphous polymer that contains various polymerized monosaccharides, such as xylose, glucose, mannose, galactose, arabinose, and glucuronic acid (Mohan et al., 2006). Hemicellulose has low thermal stability because it contains branches that include glucuronic acid and acetic acid, which inhibit the formation of hydrogen bonds (Bach and Skreiberg, 2016). Cellulose is composed of D-glucose linked by β-1, 4 glycosidic bonds (Hendriks and Zeeman, 2009). Cellulose molecules are virtually linear, containing hydroxyl groups that are involved in a number of intra- and intermolecular hydrogen bonds, creating a crystalline fibrous structure (Park et al., 2010). Therefore, cellulose is much more stable than hemicellulose with respect to thermal degradation. Lignin is an amorphous, hyper-branched, mainly aromatic polymer consisting of phenylpropane units (Bach and Skreiberg, 2016). Lignin is also a more recalcitrant substance during thermal degradation compared with hemicellulose and cellulose.

As an example of mechanical pretreatment, ball milling is an environmentally friendly and low-cost method that uses friction, collision, and shear to modify the crystalline structure of biomass materials (Nuruddin et al., 2016). It is an excellent method for destroying crystalline structure of cellulose to form amorphous cellulose (Wang et al., 2013). Previous studies have shown that ball milling is an effective pretreatment for enhancing the enzymatic hydrolysis of biomass (Silva et al., 2012; Ji et al., 2016). Although there have been many torrefaction studies on biomass such as wheat straw, rice straw, oil palm waste, sugarcane bagasse, and corn stover (Chiou et al., 2015), ball milling combined with torrefaction as a pretreatment of biomass has rarely been studied.

Sawdust, as a large amount of woodwaste generated from the wood-processing industry every year in China, could be easily utilized as a solid fuel (Kong et al., 2016). To our best knowledge, no studies have investigated the effects and mechanism of ball milling on the torrefaction process of pine sawdust. In this study, two types of pine sawdust samples were prepared by (a) ball milling and (b) hammer milling, with a screen size of 1 mm. Samples were torrefied at temperatures 230, 260, and 290 °C, and residence times of 30 and 60 min to study the torrefaction behavior and physicochemical properties of ball- and hammer-milled pine sawdust.

2. Materials and methods

2.1. Raw materials

Raw pine sawdust was obtained from the Guan county in the Hebei Province, China, in 2015. The initial moisture content of the raw pine sawdust was 4.55 wt% (wet basis). To remove impurities, the pine sawdust was first sieved and samples retained on a 5-mm screen were used for the experiments in this study. The chemical composition of raw pine sawdust (dry basis) was 14.32% hemicellulose, 47.64% cellulose, 29.57% acid insoluble fibers (mostly lignin). The details of the measurement procedure were reported in our previous work (Gong et al., 2015). For samples preparation, pine sawdust was milled with a hammer mill (Jiading Instrument Ltd., Shanghai, China) with a screen size of 1 mm to prepare hammer-milled pine sawdust (HMP). Ball-milled pine sawdust (BMP) was produced with an ultrafine vibration ball mill CJM-SY-B (Qinghuangdao Taiji Ring Nano Ltd., Hebei, China). In this process, HMP and ZrO₂ balls (6–10 mm diameter)

were mixed at a volume ratio of 1:2 and milled for 8 h, while a cooling system was used to keep the milling process below 30 °C. After HMP and BMP samples were prepared, they were dried in an oven at 105 °C for 24 h, then sealed and stored for torrefaction.

2.2. Particle size measurements

The particle size distribution of samples was measured by a laser diffraction particle analyzer, Mastersizer 3000 (Malvern Instruments Ltd, United Kingdom) with a range of 0.01 μm to 3000 μm. Raw HMP and BMP samples were dispersed in distilled water by ultrasonication before measurement (Liu et al., 2015). All measurements were carried out in triplicate. The particle size distribution of measured samples was showed in Fig. S1. The mean sizes of raw HMP and BMP were 554.08 ± 5.82 μm and 61.21 ± 3.15 μm, respectively. The particle size of pine sawdust decreased significantly after ball milling.

2.3. Torrefaction experiments

The torrefaction process was finished in a fixed-bed tubular reactor (SK-G08123K; Tianjin Zhonghuan Experimental Furnace Co. Ltd., China) that included a tubular unit, gas supply, and an electric heater. The reactor was pre-heated to the desired temperature before torrefaction. Samples were carried by a moving quartz ark and heated with a flow of nitrogen (99.99%, 0.4 L/min) to prevent oxidation. The set torrefaction conditions for HMP and BMP were 230, 260, and 290 °C with residence times of 30 and 60 min. Samples were weighed before and after torrefaction to obtain mass and energy yields. An approximately 10 g sample was torrefied under each condition and this was repeated in triplicate. Subsequently, the torrefied samples were further analyzed to study their physicochemical properties.

2.4. Sample analysis and torrefaction yields

Proximate and ultimate analyses were carried out to characterize raw and torrefied samples. The proximate analysis was performed according to standard analysis methods: ASTM D3174-04 for ash analysis and ASTM D3175-89 for volatile matter (VM). The fixed carbon (FC) was calculated by difference. The carbon, hydrogen, nitrogen, and sulfur contents were determined by an Elementar Vario EL II (Vario Macro, Germany), while the oxygen content was calculated by difference. Proximate and ultimate analyses were repeated twice.

The higher heating values (HHV) of samples were attained from the ultimate analysis by the equation (Friedl et al., 2005) given below:

$$\text{HHV} = 3.55 \cdot \text{C}^2 - 232 \cdot \text{C} - 2230 \cdot \text{H} + 51.2 \cdot \text{C} \cdot \text{H} + 131 \cdot \text{N} + 20600, \text{ kJ/kg.} \quad (1)$$

To study torrefaction yields, the mass and energy yields were calculated on a dry basis and defined by Eqs. (2) and (3), respectively (Bridgeman et al., 2008).

$$\text{Mass yield (YM)} = \frac{\text{Mass of torrefied biomass}}{\text{Mass of raw biomass}} \times 100\% \quad (2)$$

$$\text{Energy yield (YE)} = \frac{\text{HHV of torrefied biomass}}{\text{HHV of raw biomass}} \times \text{YM} \quad (3)$$

2.5. X-ray diffraction analysis

The crystallinity of samples was measured by a D8 ADVANCE X-ray diffractometer (Bruker, Germany) using Cu-Kα at 40 kV

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