



Research paper

Hydrothermal liquefaction of spent coffee grounds in water medium for bio-oil production



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ABSTRACT

Spent coffee grounds (SCG) were liquefied in hot-compressed water to produce crude bio-oil via hydrothermal liquefaction (HTL) in a 100 cm³ stainless-steel autoclave reactor in N₂ atmosphere. We investigated the effects of operating parameters such as retention times (5 min, 10 min, 15 min, 20 min and 25 min), reaction temperatures (200 °C, 225 °C, 250 °C, 275 °C and 300 °C), and water/feedstock mass ratios (5:1, 10:1, 15:1 and 20:1) and initial pressure of process gas (2.0 MPa and 0.5 MPa) on the yield and properties of the resulting crude bio-oil. The highest yield of the crude bio-oil (47.3% mass fraction) was obtained at conditions of 275 °C, 10 min retention time and water/feedstock mass ratio of 20:1 with an initial pressure of 2.0 MPa. The elemental analysis of the produced crude bio-oil revealed that the oil product had a higher heating value (HHV) of 31.0 MJ kg⁻¹, much higher than that of the raw material (20.2 MJ kg⁻¹). GC–MS and FT-IR measurements showed that the main volatile compounds in the crude bio-oil were long chain aliphatic acids and esters.

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1. Introduction

The inherent conflict between the increase of global energy demand and the depletion of fossil fuel reserves, along with the environmental concerns caused by the extensive use of fossil fuels, has necessarily called for sustainable energy alternatives [1]. Using biomass for liquid biofuels production has sparked the interest worldwide due to its abundance, renewability and carbon neutrality [2]. However, today, the major viable commercial options for producing liquid biofuels use edible crops (vegetable oils for biodiesel and corn/sugarcane for bioethanol), which has negative impacts on food and feed supplies [2,3]. Research efforts are, therefore, re-orientated to use the biological waste such as agricultural and forest residues, animal manure, food processing waste and municipal waste [4–10]. Utilizing waste streams for biofuels production also has additionally environmental benefits, including reducing the local pollution, decreasing greenhouse gas emissions, and lessening the burden on landfills.

Coffee is one of the most popular beverages and widely consumed in America, Japan and Europe [11,12]. According to the coffee trade record from the International Coffee Organization [13], the global coffee production has increased by 6% since 2010, and the total coffee production in 2014 was 142 million bags, equalling 9.6 million tonnes. The amount of spent coffee grounds (SCG), the solid residues obtained from coffee brewing, therefore increases accordingly. Despite such biomass residues are generated in large quantities each year, they are mostly underutilized, being disposed in landfills or small amounts used for composting [14]. However, the complex organic substances (e.g., caffeine, tannins and polyphenols) left in the coffee residue were found to have adverse impacts on land and the environment [11]. Some industries attempted to process SCG into coffee logs or pellets for heating and electricity generation, while its combustion generated particulate matter and hazardous gases, especially high nitrogen oxides in emissions, compared to wood pellets or briquettes. This dramatically limits the direct use of SCG as solid fuels [15,16]. New interests are recently developed to use SCG for liquid biofuels production. Kondamudi et al. [12] extracted coffee oil from SCG for biodiesel production. However, the average oil content in SCG is fairly low, only 10–15% mass fraction, and the necessary solvent extraction makes this option economically unfavorable. Mussatto et al. [17]

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evaluated the feasibility of producing bioethanol from SCG through fermentation using three different yeasts, while the ethanol production yield was only 26.0% mass fraction. Other researchers [18–20] used a fast pyrolysis process to convert SCG into the bio-oil. The results were very promising, with an oil yield as high as 54.9% mass fraction. Unfortunately, SCG has a fairly high moisture content (50–60% mass fraction) [12], and thus a pre-drying process is required before SCG is fed to a pyrolysis system. This leads to a large energy consumption and decreases the overall economic viability. Moreover, the quality of the pyrolysis bio-oil is not satisfactory. A relatively high oxygen (35.2–59.5% mass fraction) resulted in lower HHVs in a range of 17 MJ kg⁻¹ to 23 MJ kg⁻¹, depending on the composition of feedstock and pyrolysis conditions. Therefore, it is essential to explore other pathways to utilize this waste stream effectively, efficiently and economically.

Hydrothermal liquefaction (HTL) is an emerging technology that is able to convert wet biomass (especially waste materials) to the oil (referred to as bio-oil or biocrude), aqueous, gaseous and solid products at high pressures (5–22 MPa) and moderate temperatures (250–380 °C) in sub-/supercritical water medium [21,22]. HTL is advantageous over other thermochemical conversion technologies such as fast pyrolysis and gasification. It is carried out at lower temperatures; more importantly, it accepts wet biomass, and thus obviates an energy-intensive pre-drying process. Also, the bio-oil obtained from the liquefaction process is hydrophobic, typically has a lower oxygen and water content, giving much higher HHV (25–35 MJ kg⁻¹) compared to those of the fast pyrolysis bio-oil (14–20 MJ kg⁻¹). The HTL crude bio-oil can be mixed with other fuels and used for combustion or be further upgraded to high quality hydrocarbon liquid fuels such as gasoline and diesel [22–26].

Many researchers have been focusing on the bio-oil production via the HTL process from a wide variety of biomass, including woody biomass, mainly forest residues [27–30], agricultural waste such as barley straw, corn stalk, corncob and rice husk [31–34], animal manure [35–37], municipal solid waste [38–40] and most recently microalgae [41–48]. Through these studies, it was found that the yield and quality of the bio-oil resulting from the HTL process were highly associated with the operating parameters such as the reaction temperature, retention time, ratio of feedstock and solvent, etc., as reviewed by Akhtar and Amin [49] as well as the experimental results reported in the literature [27–48]. The commonly used temperature range is from 250 °C to 400 °C; and usually around 300 °C, the maximum bio-oil yield was obtained. At a low temperature, an incomplete decomposition of biomass gives a relatively high solid residues yield and a low bio-oil yield. With an increase in the liquefaction temperature, the yield of bio-oil increases while the solid residue decreases. However, at the temperature higher than 300 °C, for most feedstocks, a decreased bio-oil yield and increased gas fraction yield are observed due to a secondary decomposition of the bio-oil and Bourdard gas reactions. However, the solid residue yield may also increase, being caused by the re-polymerization of the bio-oil components and/or recombination of high-concentration free radicals to form bio-char. Retention time is another key factor influencing the bio-oil production and the overall biomass conversion. Generally, a short retention time (10–30 min) is preferred, giving a relatively high bio-oil yield. The ratio of feedstock and solvent also plays an important role in the biomass liquefaction process. It was observed that a low ratio of feedstock and solvent led to a high bio-oil yield, however it was not economically favorable. In contrast, some researchers also found that a higher biomass concentration might promote the dehydration/polymerisation of the intermediates products, which resulted in an increase of the bio-oil yield. Despite enormous studies conducted and significant progress made, the

underlying mechanism of the effects of operating parameters on HTL process was so far not fully understood. The optimization of reaction conditions and knowledge of the interaction of operating parameters have to be obtained by experiments.

In addition to operating parameters, the nature of biomass, i.e., different components of cellulose, lignin, protein and fat, significantly impacts the yield and chemical composition of the HTL products. To date, most of studies examined the lignocellulose-based feedstock (e.g., forest residues, agricultural wastes), which contains a relatively high content of lignin while low contents of lipid and protein [27–30,50]. The HHVs of the HTL bio-oils obtained from the lignocellulose biomass (without using any catalysts and organic solvents) are in the range of 30–36 MJ kg⁻¹, and the oil yields are relatively low (20%–36% mass fraction). These studies indicated that a high content of lignin in feedstock was not desirable for the bio-oil production. Recently, it was observed that high lipid and protein contents in feedstock benefited the yield and the quality of the bio-oil [41–48,51,52].

A thorough literature review encouraged us to explore the feasibility of converting the spent coffee grounds (SCG) to bio-oil via HTL. SCG has higher contents of lipid (\approx 15%) and protein (\approx 17.4%), while a lower content of lignin (\approx 23.9%), compared to the lignocellulose biomass [11,12,53]. It might be a promising feedstock for the bio-oil production using the HTL technology. More attractively, the size of SCG is in the range of 200–300 μ m [19], which makes it suitable for being directly fed to the HTL reactor without a prior energy-intensive grinding process.

In this study, SCG was liquefied in hot-compressed water in N₂ atmosphere. Effects of the retention time (varied from 5 min to 30 min), reaction temperature (varied from 200 °C to 300 °C), water/feedstock mass ratio (varied from 5:1 to 20:1) and initial pressure (0.5 MPa and 2.0 MPa) on the yield of product fractions were investigated, with an expectation of maximizing the oil yield. The properties of the resulting bio-oil (e.g., elemental composition, higher heating value, and chemical composition) were characterized. To the best of our knowledge, this is the first to examine SCG as a feedstock for the bio-oil production via a hydrothermal liquefaction process.

2. Experimental

2.1. Materials

The wet SCG for this study were collected after coffee beans (*Coffea arabica*) brewing from Tim Hortons, London, Canada, and were first air-dried at room temperature over several days, and then were oven dried at 105 °C. The dried SCG were kept in sealed bags and stored in refrigerator at 4 °C. For each experiment, the required amount of raw material was dried further in an oven at 105 °C overnight. The proximate and ultimate analyses of dried SCG are respectively listed below: 82.3% volatiles, 1.4% ash, 4.0% moisture and 50.4% C, 7.2% H, 2.1% N, and 40.3% O (calculated by difference). The solvents used were distilled water and reagent grade acetone, purchased from Caledon laboratories Ltd., which was used as received.

2.2. Hydrothermal liquefaction procedure

The experiments were carried out in a 100 cm³ stainless-steel autoclave (Parr Instrument, 5513) equipped with a stirrer. In a typical run, 2.5–10 g dried SCG were weighed and loaded into the reactor, followed by the addition of certain amount of distilled water with desired water/feedstock mass ratios 5:1, 10:1, 15:1 and 20:1, respectively. The occupation of reactor volume was maintained between 50 cm³ to 60 cm³. The reactor with the suspension

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