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Production and separation of rice husk pyrolysis bio-oils from a fractional distillation column connected fluidized bed reactor

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ABSTRACT

Rice husks are pyrolyzed in a fluidized bed pyrolyzer and the produced vaporized bio-oils are immediately separated by fractional distillation. The effects of the pyrolysis temperature, rice husk feeding rate and superficial gas velocity on the distributions of chars, bio-oils and syngases in the product are studied. When the pyrolysis temperature is 500 °C, the rice husk feeding rate is 20 g/min and the superficial gas velocity is 0.340 m/s, the mass fraction of bio-oil in the product shows the highest value of 30.4%. The decomposable volatiles in the chars are the lowest for the chars obtained at the lower rice husk feeding rate of 10 g/min and higher superficial gas velocity of 0.425 m/s. GC–MS analyses indicate that the major chemicals in the bio-oils are acetic acid, furfural, phenol and phenol derivatives. The fractional distillation separates the bio-oil into a light fraction (C4–C7) and a heavy fraction (C7–C10). Relatively high content of acetic acid in the light fraction of the bio-oil is responsible to the lower pH value of the light fraction of the bio-oil. Phenolic compounds are rich in the heavy fraction. Vaporized bio-oils directly from the fluidized bed pyrolyzer are successfully graded by fractional distillation.

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1. Introduction

Renewable energy is becoming increasingly important as the fossil fuel reserves gradually deplete and the increasing concerns about the global warming caused by the utilization of the fossil fuels. The carbon neutral biomass is considered as one of the most promising renewable energies [1–3]. Among the various biomass conversion technologies, pyrolysis offers a convenient way to thermally decompose biomass macromolecules into lower molecular weight chemicals as solid chars, liquid bio-oils and non-condensable syngases [4]. While slow pyrolysis produces a higher mass fraction of solid chars, fast pyrolysis produces a higher mass fraction of liquid bio-oils [5]. The physical and chemical compositions of the biomass and the rate of condensation also affect the mass fraction distribution of the chars, bio-oils and syngases in the product [6].

Although chars, bio-oils and syngases have different applications, bio-oils are comparably attractive. Bio-oils, having higher specific energy density than the raw biomasses, are readily for transportation and may be combusted in some conventional boilers without further treatment [7]. Different biomasses were tested to maximize the production of bio-oils [8–10]. Hence, extensive works have been done to maximize the bio-oil mass fraction in the products and to find new applications of

the bio-oils. However, bio-oil has some drawbacks. They show relatively low heating values due to the higher oxygen and water contents. The low pH values of the bio-oils may be corrosive. Bio-oils also show low stability and are not totally compatible with current petroleum fuels [11]. Although bio-oils have been upgraded as diesel fuels and value-added chemicals have been extracted therein, effective and economic biomass pyrolysis bio-oil production methods and bio-oil treatment technologies are still required for the wider applications of the bio-oils [12–13].

Bio-oils are complex containing nearly 400 known compounds with a wide range of boiling points [14]. To have a wider application of the bio-oils, separation operations were applied to bio-oils. The applied bio-oil separation technologies include solvent extraction, column chromatography, and distillation [15]. Although supercritical CO₂ extraction has been adopted for bio-oil extractions [16], large amounts of solvents are typically involved in the solvent extraction operations, which are usually not favored for industrialization. Column chromatography is economic but its low throughput performance may only be applicable for higher value product separations [17]. Distillation separates components based on the differences in their boiling points or the various mean free paths of the component molecules. Bio-oils were separated into a light fraction, a middle fraction and a heavy fraction by molecular distillation and a maximum distillate yield of 85% has been reported. However, coking or polymerization may occur during molecular distillation [18–19]. Fractional distillation of pyrolysis bio-oils has been investigated under atmospheric and reduced pressure/vacuum

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conditions. A 61 wt% distillation yield was reported from reduced pressure distillation and the distilled bio-oil has lower oxygen content and a higher heating value. While oxygenated and aromatic compounds were found in the light and middle fractions, phenolic compounds were rich in the heavy fraction [20–21]. Although the conventional fractional distillation is effective in bio-oil separation, the operation is energy intensive. Reactive distillation combining the bio-oil production and separation is relative energy saving, but may experience catalyst deactivation [11]. Under atmospheric pressure, bio-oil boiling begins below 100 °C and the bio-oil distillation stops at 280 °C, leaving some oils as residues [14]. Because biomass pyrolysis is carried out well above 280 °C (say 500 °C) and direct separation of the vaporized bio-oils leaving the pyrolyzer could save the energy utilized later for bio-oil boiling in distillation. However, few studies have been done to directly apply distillation to the vaporized bio-oils immediately produced from the pyrolyzer.

Biomass should be supplied locally to reduce the cost and carbon emission during transportation. Nevertheless, the constituents of biomass affect the chemical compositions of the corresponding pyrolysis bio-oils and the variety of biomass feedstocks/types make it difficult to predict the compositions of the bio-oils [6]. Extensive works are required to enrich the pyrolysis bio-oil databases. Rice farming area of Taiwan is about 2.7 billion m² and the handling of the large amount of the rice husks is a big challenge in Taiwan. In this work, rice husks are pyrolyzed in a fluidized bed followed by a fractional distillation column. The effects of the biomass feed rate, the superficial gas velocity and the pyrolysis temperature on the quality and quantity of the bio-oils are investigated to enrich the pyrolysis bio-oil database. The vaporized bio-oils produced from the fluidized bed pyrolyzer are immediately separated by fractional distillation and the graded bio-oils are characterized.

2. Experimental method

Rice husks are from Changhua county, Taiwan. Element analysis of rice husks shows that the mass fractions of N, C, S, H, O are 0.33%, 40.26%, 0.058%, 5.36%, 31.02%, respectively (Vario EL, Heraeus). The sulfur mass fraction is as low as 0.058%, indicating that rice husks are clean energy resources. The rice husk thermogravimetry proximate analysis (Q50, TA instruments) indicates that the water mass fraction is 6.0%, the

volatile matter mass fraction is 71.1%, the fixed carbon mass fraction is 9.9% and the ash mass fraction is 13.0%. The heating value of the rice husk is 3946 kcal/kg on dry bases (IKA C200). Rice husks were sieved into the size range of 1.68 mm to 3.36 mm and dried in a 110 °C oven for at least 12 h before each experimental run.

Fig. 1 shows the schematic diagram of the fluidized bed pyrolysis-fractional distillation system. The cylindrical pyrolyzer is made of stainless steel. The reactor is 50 mm in internal diameter and 500 mm in length. The rice husks are delivered to the reactor by a side screw feeder located 350 mm below the top of the reactor. The gas distributor was a stainless steel mesh (#40) located at the bottom of the reactor. 200 g of bed material was loaded above the gas distributor. The bed materials are sodium glass beads within the size range of 0.42 mm to 0.84 mm having a density of 2370 kg/m³.

In a typical experiment, excess rice husks were loaded into the tank (1) and they were fed to the preheated fluidized bed reactor (2) by the screw feeder (3). The feeding rate of biomass was controlled by the rotational speed of the motor connected to the screw. Nitrogen flows into the top of the tank (1) with a rate of 10 l/min to prevent the entering of the gaseous products to the tank (1). The carrier gas was nitrogen. The exact pyrolysis temperature was determined by the thermocouple probing inside the pyrolyzer. The superficial gas velocity was controlled by a gas mass flow rate controller. Rice husks were pyrolyzed in the fluidized bed and solid chars, vaporized bio-oils and gaseous syngases were produced in the reactor.

While most solid chars were collected by the cyclone (4), some chars stayed in the reactor after the experiment. In the yield calculations, the solid chars include those in the cyclone and those in the reactor after the experiment. The vaporized bio-oils and gaseous syngases were introduced to a 4-stage fractional distillation column (5) packed with 140 g of glass beads of 5 mm in diameter at each stage. The distillation column was 47.5 mm in diameter and 430 mm in length. The temperature distributions inside the distillation column were determined by measurements. The experimental setup was built inside a chemical hood. The temperature inside the hood was approximately constant during the course of the experiment. The bio-oils were condensed at *ca.* 120 °C (in column), *ca.* 80 °C (in column), *ca.* 20 °C (water condenser for top stream) and 0 °C (ice condenser for top stream). Bio-oils collected in the column are heavy fractions and collected in the water trap and

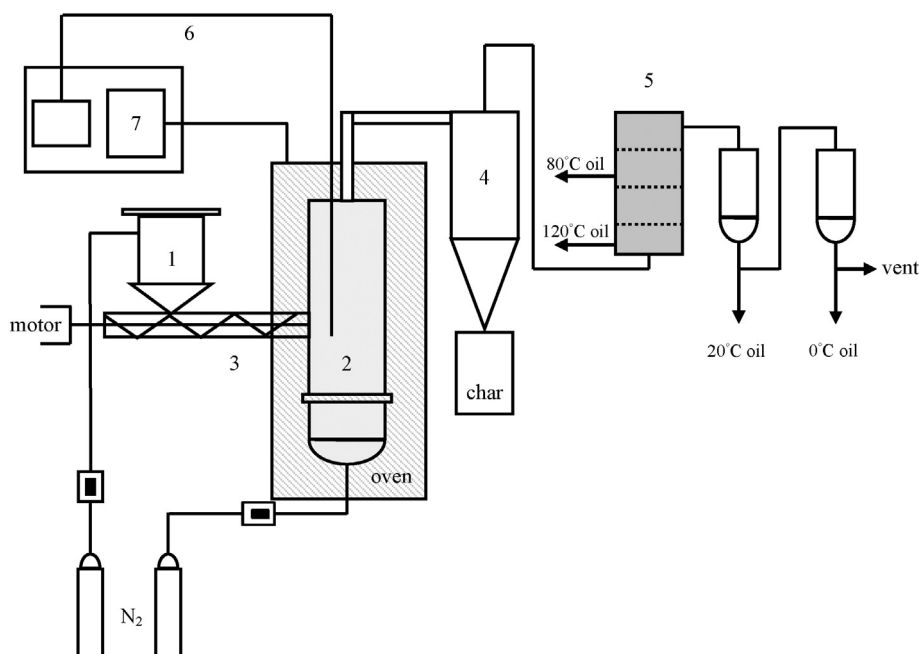


Fig. 1. Schematic diagram of the rice husk pyrolysis-fractional distillation setup: (1) feed storage tank, (2) fluidized bed pyrolyzer, (3) screw feeder, (4) cyclone separator, (5) fractional distillation column, (6) thermocouple, (7) temperature controller.

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