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Orthogonal test design to optimize products and to characterize heavy oil via biomass hydrothermal treatment

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ABSTRACT

The key parameters for biomass hydrothermal treatment were optimized for product distribution in this study on the basis of a L16 (45) orthogonal experiment design. Results showed that biomass species, particle size, and hydrothermal temperature significantly affected heavy oil yield. By contrast, the effect of biomass concentration was negligible. The maximum heavy oil yield was 28.00 wt.% at the optimal condition (biomass species, pine sawdust; 250 °C; 80–150 mesh; 15 min; 10 g/110 g). In addition, cotton straw yielded the most liquid in the agricultural straws, although ash content was low. The influences of temperature, residence time, catalysts, and the size of cotton straw particles on product distribution were investigated as well. The results of analysis with GC–MS (gas chromatography–mass spectroscopy) indicated that the liquid product contained organic components, namely, acids, esters, aldehydes, ketones, and phenols. Among these components, acids, esters, phenols, and their derivatives were dominant. The addition of catalysts increased oil yield and also affected the oil components. Specifically, acids and ketones were reduced and the pH value of the oil increased. As a result, its quality improved to a certain extent. This research provides a reference for biomass hydrothermal treatment.
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1. Introduction

Unlike solar, wind, and other renewable resources, biomass is the sole material that contains carbon and can be used in the large-scale production of liquid fuel. The liquid fuel derived from biomass is expected to replace fossil fuels, such as natural gas, gasoline, and diesel, as the main transportation fuel in the future. Thus, this fuel has broad application prospects [1]. Biomass resources are scattered, with low energy density and high collection and transportation costs. Thus, the processing system of distributed biomass must be developed according to local conditions by converting biomass resources into the bio-oil of intermediate products to improve energy density and to reduce transport cost. The bio-oil of intermediate products is then collected from bio-refinery factories to improve and modify quality for the preparation of high-grade liquid biofuels [2,3]. Supercritical fluid technology has been widely applied in material preparation, chemical reaction, and many other fields given its excellent heat and mass transfer capability and controllability. This technology has also been used successfully in biofuel production [4–6].

The technology for biomass hydrothermal treatment processes biomass by considering the properties of subcritical/supercritical water. This topic is popular in current research on biomass resource degradation and transformation. All conditions that can affect the properties of water during hydrothermal reaction are regarded as influential factors in this reaction. These conditions mainly include reaction temperature, pressure, residence time, and catalyst [7].

Reaction temperature, pressure, and time are important parameters in hydrothermal treatment. Therefore, their settings directly affect the treatment goals and effect. In particular, the settings of temperature and pressure influence the properties and role of water in hydrothermal treatment firsthand. This effect is the focus of recent research [8–10]. The temperature and pressure requirements for hard-degradation biomass are generally high. By contrast, those for cellulose, hemicelluloses, and lignin are low [11,12]. When temperature and pressure increase, the hydrothermal reaction becomes increasingly exhaustive as well. The material

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reacts quickly, and the reaction process is usually completed within a few seconds. This procedure produces CO_2 and water. Nonetheless, the pressure of the hydrothermal reaction is generally determined by the temperature setting during actual treatment research.

Residence time is another important factor that affects the influence of hydrothermal treatment. Terminating the reaction at the appropriate time can not only generate a good treatment effect but can also save energy; thus, this factor is also a hot topic in the research on hydrothermal treatment [13,14]. Residence time is determined based on the raw materials, temperature, and pressure applied during the treatment, as well as the desired product. In particular, the temperature and pressure of the treatment are negatively related to this factor. In other words, residence time is shortened accordingly if temperature and pressure increase when the same kinds of materials are treated and when the desired results are similar.

The use of catalysts is another influential factor in hydrothermal treatment. The addition of suitable catalysts can not only accelerate the reaction rate but can also change the reaction route. Furthermore, new desired products are generated, thereby improving treatment effect and efficiency. The catalysts studied in relation to hydrothermal reaction technology can be categorized into oxidant and chemical catalysts. The former is investigated by adding peroxides such as hydrogen. Research confirms that the incorporation of oxidant catalysts can improve treatment efficiency and can change the reaction route. At present, studies on this aspect mainly focus on the added amount and residence time of oxidants. The scope of research on chemical catalysts mainly includes dilute acid [15,16], dilute alkaline solution [17], and neutral or basic salt and metal [18,19].

Gao et al. discussed the influence of reaction temperature on the distribution of the gas products of heavy oil, solid residue, and light oil from hydrothermal treatments conducted on cellulose. As per analysis results, heavy oil yield is high when cellulose temperature is within the range of 250 °C–350 °C and residence time ranges from 5 min to 30 min [20]. Therefore, the liquid product prepared within this range is suitable for the characteristics and high-value application of products derived from the yield. Yin et al. studied the pyrolysis behavior of cow manure in subcritical water and determined that its oil yield is maximized at 48.38 wt.% when hydrolyzed at 310 °C given a residence time of 15 min and an atmosphere of CO. The main ingredients are toluene, ethylbenzene, and xylene, as in gasoline and diesel. The average heat value of the oil is 35.53 MJ/kg [21]. Cheng et al. investigated the hydrothermal liquefaction of switch grass with an 11 ml hydrothermal reactor in subcritical water. They determined that the conversion rate of biomass can exceed 90 wt.% when the temperature is between 250 °C and 350 °C, pressure is 20 MPa, and residence time ranges from 1 s to 300 s. This finding indicated that the hydrothermal reaction is successful in the conversion of biomass to liquid fuel oil under the rapid reaction condition [22]. Huang et al. studied the hydrothermal characteristics of straw, microalgae, and sludge at 350 °C and a residence time of 20 min. Despite the low content of organic matter in sludge, the yield of bio-oil reached 39.5 ± 1.16 wt.%. Moreover, the calorific value was 36.14 MJ/kg, which was significantly higher than those of straw (21.1% ± 0.93%) and microalgae (34.5% ± 1.31%). GC–MS (gas chromatography–mass spectroscopy) results showed that the bio-oil of straw mainly contained phenols, whereas those of sludge and microalgae primarily consisted of esters [23]. Akalin et al. conducted a hydrothermal treatment on cherry stones at reaction temperatures of 200 °C, 250 °C, and 300 °C and residence times of 0, 15, and 30 min. Oil yield is high at 28.00 wt.% under temperatures of 250 °C and 300 °C, as well as a residence time of 0 min. Solid residue yield gradually decreases with temperature and residence time. Furthermore, the heat values of light and heavy oils are 23.86 and 28.35 MJ/kg, respectively. The light oil mainly contained furfural, phenol, and vanillin. In heavy oil, the concentration of linoleic acid is maximal at 250 °C and 300 °C [24]. To improve oil yield and quality, catalysts can be added to the hydrothermal reaction by restraining the condensation reaction from oil to coke. Song et al. conducted a liquefaction experiment that incorporates catalysts and determined that oil yield increases from 33.4 wt. % to 47.2 wt. % when 1 wt. % of Na_2CO_3 is added [25].

The hydrothermal liquefaction of biomass converts the active group in the biomass to liquid organic material to maximize its use. Thus, the reaction route of the liquefaction of the main biomass component, the method of generating the active group, and the structure and distribution of liquefied products must be clarified. This clarification is the theoretical basis for controlling the liquefaction path. In the current study, the hydrothermal method is first applied in an orthogonal experiment. The optimal process conditions are determined for different target products. Among the agricultural straws, cotton straw displays the highest liquid yield. However, ash content is low. Thus, the quality of bio-oil is relatively high. Cotton straw is used as the raw material in this study. Furthermore, the production distribution during the hydrothermal reaction of biomass is investigated according to different influential factors, such as the reaction temperature, residence time, catalyst, and particle size of biomass samples. It is also examined by separating and analyzing products effectively. The formation process and chemical composition of the oil phase products are analyzed as well.

2. Material and method

2.1. Samples

Rice straw, cotton straw, pine sawdust, and water hyacinth were utilized as the raw materials in the experiment. The samples were collected from the city of Wuhan, which is located south of China. The samples were dried at 55 °C for 8 h after crushing and screening. They were then sealed in the drying tower. The solvents (acetone) and bases [NaOH, Na_2CO_3, KOH, K_2CO_3, and $Ca(OH)_2$] were purchased from Shenshi Chemicals, China and were used as received.

2.2. Experiment procedure and analysis

The biomass was dried at 105 °C for 12 h. It was hydrothermally treated in a high-temperature (maximum working temperature of 600 °C) and high-pressure (maximum pressure of 40 MPa) CWYF-type batch reactor manufactured by Haian Scientific Research Devices Co., Ltd. (Jiangsu, China). This reactor was equipped with a 500 cm^3 vessel made of 316 L stainless steel, was 150 mm high, had an internal diameter of 65 mm, and possessed 2 mm-thick walls. It also contained with a 1000 rev/min magnetic mixer, a manometer, and an internal cooler in the form of a U-loop. A thermo-coupler was also placed in the vessel within the reactor. The temperature was controlled at 5 °C, and the pressure was measured at an accuracy of 2%.

In the typical catalytic hydrothermal experiment, the reactor was loaded with 8 g (dry basis) of cotton straw and 110 ml of 1 M alkaline solutions [NaOH, Na_2CO_3, KOH, K_2CO_3, and $Ca(OH)_2$]. In the thermal run, the reactor was loaded with a certain amount of biomass and deionized water once the raw materials swelled completely. After the air was displaced, the autoclave was heated to the desired reaction temperature and set to the specified residence time. Then, it was cooled to room temperature with an electric fan and the internal cooling U-loop. In a flow-through process, the

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