



Study on co-pyrolysis synergistic mechanism of seaweed and rice husk by investigation of the characteristics of char/coke

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ABSTRACT

This study investigates the characteristics of pyrolysis char/coke and elemental morphological changes on surface of char/coke produced, during co-pyrolysis of seaweed (EN) and rice husk (HU) at various temperatures through XPS, FTIR and SEM analysis methods. Analysis of pyrolysis products including by-products will help to partly explore co-pyrolysis synergistic mechanisms of the seaweed and rice husk. Thus, fast pyrolysis of EN and HU was conducted in a fixed bed reactor. Coke/char from the individual pyrolysis of EN and HU respectively at different temperatures, and those from co-pyrolysis of EN and HU (1:1) at 550 °C, were analyzed. Results revealed that the co-pyrolysis process has synergistic effects. Moreover, the release of nitrogenous substances was found to be inhibited by addition of HU at low temperature (<190 °C) range. It was also observed that co-pyrolysis of EN with HU does not only facilitates pyrolysis of aliphatic C–O functional groups at the middle temperature range, but also contributes to the cleavage of ether bonds of some water-soluble polysaccharides at high temperature (320–550 °C) level, hence generating aromatic compounds. Besides, the analysis of pore structures, pore size distributions and fractal dimensions of pyrolysis char/coke also show that the co-pyrolysis process has a synergistic effect.

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1. Introduction

With the advantage of abundant reserves, renewability and low pollution effect, biomass has now been widely utilized to tackle energy shortage and environmental issues. Seaweed, as a type of biomass, stands out with the quality of fast growth, high production rate per unit area, high photosynthetic efficiency and doesn't compete with food crops nor occupy arable land. Thus, it is recommended for bio-fuel production. Seaweed is a type of complex polymer; therefore, its thermal cracking is a complex physical and chemical process. At present, many scholars at home and abroad have studied the mechanisms of algae pyrolysis, based mostly on

the product yields and compositions. Wang et al. [1] studied the composition of seaweed pyrolysis bio-oil. Bae et al. [2] conducted pyrolysis experiments to extract oil from 3 types of seaweeds. Du et al. [3] and Anastasakis et al. [4] studied pyrolysis characteristics of the main components (carbohydrates, lipids and proteins) of seaweed by Py-GC/MS. Therefore, macroalgae pyrolysis and bio-oil production is one of the hotspots in the field of seaweed energy utilization. The current research on biomass pyrolysis and oil production is focused on the development of advanced pyrolysis technology (e.g., co-pyrolysis, catalytic pyrolysis). Co-pyrolysis is the common pyrolysis of two or more fuels, which takes advantage of the pyrolysis synergistic coupling effect of fuels, to improve oil production or quality. Because of its excellent output/input ratio, co-pyrolysis is considered to be very promising for industrial utilization [5,6]. Meng et al. [7] studied the co-pyrolysis characteristics and synergistic effects of Indus wood and coal. Abnisa and Daud [5],

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conducted in-depth analysis of the impact of various factors on the product during co-pyrolysis of biomass and plastic or rubber. Samanya et al. [8] obtained bio-oil of high calorific value, low nitrogen content by co-pyrolysis of sludge and sawdust. Kositkana-wuth et al. [9] pointed out that co-pyrolysis of algal biomass and polystyrene can improve the quality of bio-oil. These results indicate that co-pyrolysis of selected samples can produce synergistic effects, enhance pyrolysis effect and improve oil quality. Seaweed and terrestrial biomass, each has demonstrated advantages and disadvantages in pyrolysis, and as a result, some scholars have conducted researches on co-pyrolysis of seaweeds and terrestrial biomass [10,11]. However, most scholars mainly focus attention on the yields and compositions of the bio-oils, the char and gas from the pyrolysis and co-pyrolysis processes [12–14]; while a few have investigated the pyrolysis mechanisms by analyzing the changes in the pyrolysis by-products (like the char/coke).

In fact, biomass pyrolysis will produce 15–30% solid residue (typically, a mixture of coke and char). Coke is the solid carbon produced through the secondary pyrolysis of volatile matters, whereas char is the solid residue obtained by emitting volatile matters [15]. On one hand, during biomass pyrolysis, when the organic matter of the composition converts into gas volatiles, the elements on the surface of char/coke changes during the release of gas volatiles. On the other hand, since the biomass pyrolysis mostly occurs in the pores of the biomass particles, the reaction leads to changes of the structures of the pores in char particles. And these changes in pore structure will in turn affect the progress of the reaction; therefore, like the bio-oil properties analysis, it is also very important to study the surface characteristics and pore-structure of biomass coke during the pyrolysis process to partly reveal the reaction mechanisms of the pyrolysis, which will in turn improve the process. However, few studies have focused on studying the pyrolysis, most especially, the co-pyrolysis from the perspective of the analysis of the changes in the pyrolysis by-products and the comparison of the by-products (most especially, char/coke) from various biomass and co-feeding mixtures. Since the algal biomass has high ash content and abundant porosity, it is more feasible to study the mechanisms of the pyrolysis by studying its char/coke characteristics. In this study, the fast pyrolysis experiments of seaweed and rice husk is investigated. Biomass char/coke samples produced via the pyrolysis were characterized with respect to their overall contents, surface area, pore size distribution changes, bonding groups and their effects on the pyrolysis performance. X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) analysis methods were employed in the biomass char/coke analysis and investigation of the elemental morphological changes on the surface of the chars/cokes produced at various pyrolysis temperatures during the pyrolysis process of EN, HU and their mixture. Through the analysis of EN pyrolysis char/coke, its pyrolysis mechanisms could be partly revealed. Furthermore, by studying the characteristics of the co-pyrolysis char/coke and comparing the results with the pyrolysis characteristic of the EN single sample, the synergistic mechanisms of seaweed and rice husk co-pyrolysis could also be explored and partly revealed. The results of this piece of research work are, no doubt, of scientific relevance, and practically valuable for improving biomass treatment via fast co-pyrolysis as an effective thermochemical and upgrading process. Thus, the result of this study will be of great importance in proper understanding of the synergistic co-pyrolysis mechanisms of biomass and the characteristic effects of biomass char/coke on the pyrolysis performance. This result will also serve as a vital piece of information required for efficient pyrolysis process design, improvement of treatment processes and pyrolysis systems for enhanced biofuels production. Therefore, co-pyrolysis process

technique can play a pivotal role in development of the biomass energy industry. However, proper understanding of the synergistic reaction mechanisms involved in the process is very vital in order to improve the overall process design, planning, and operation; as the mechanisms between the biomass and the co-feeding materials are complex and need in-depth investigation [16,17].

2. Materials and methods

2.1. Experimental materials and analysis

The raw materials were *Enteromorpha clathrate* (EN) collected from Fujian Province China and the agricultural waste, rice husk (HU). They were dried and stored in a cool and dry environment. The dried materials were ground by a pulverizer with a particle size <0.18 mm. The results of the proximate and ultimate analyses are summarized in Table 1. The protein content of the EN is 27.05%, the fat mass fraction is 1.18% and the carbohydrate mass fraction is 36.84%. Rice husk is low in the content of fat and protein, and rich in cellulose, lignin and silica.

2.2. Experimental equipment and methods

Fast pyrolysis experiments of EN, HU and their mixture were performed in a fixed bed reactor, as shown in Fig. 1. The fixed bed reactor (internal diameter: 70 mm, height: 100 mm) was heated with electric resistance wire, programmed temperature controlling was adopted. One thermocouple was used to monitor the internal temperature in the reactor. The experiment was carried out in the nitrogen atmosphere at 200 ml min⁻¹ flow rate to ensure an inert atmosphere and effective residence time of pyrolysis volatile. Before every pyrolysis test, N₂ would be preheated at 250 °C for faster heating in the pyrolysis reactor.

From the early studies on TG and DTG curves of EN and HU pyrolysis, Wang et al. [18], found that the mass loss during the pyrolysis process began at about 186 °C, and a significant heat loss rate peak appeared at 323 °C (mainly the weight loss peak of water-soluble polysaccharides). The shoulder peak is mainly caused by protein pyrolysis; after 546 °C, the weight loss change diminished. The mass loss of the rice husk in the pyrolysis process started at about 210 °C, and after 580 °C, the change of the weight decreased. Thus, it can be concluded that different components (water-soluble polysaccharides, proteins) determine different pyrolysis curves. Thereafter, the pyrolysis characteristics of the samples were studied by analyzing the characteristics of the by-product (char/coke) at 190 °C, 320 °C and 550 °C, respectively. Meanwhile, the synergistic mechanism of the mixed pyrolysis at 550 °C when equal amount of rice husk was added was revealed indirectly. About 10 g of sample was introduced in the reactor as soon as the temperature of pyrolysis reactor reached the set temperatures (190 °C, 320 °C, 550 °C). Besides, the asbestosed wire gauze was used to filter the char at the exit of the volatiles and non-condensable gases. During the experiment, three ice-bath condensers were applied to convert the condensable volatiles to liquid form. The whole experiment lasted for about 30 min, and after the reaction, the power source of the electric heating device is automatically turned off to stop the heating action. Then the char was cooled to room temperature in pure N₂ atmosphere, and collected for the subsequent analysis. EN sample was marked as Y0, the chars/cokes of EN at 190 °C, 320 °C and 550 °C were marked as Y1, Y2, and Y3 respectively, the chars/cokes of EN-HU mixture (1:1) at 190 °C, 320 °C and 550 °C were also marked as Y4, Y5 and Y6 respectively, while the chars/cokes of HU at 190 °C, and 550 °C were marked as Y7 and Y8 respectively.

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