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Temperature rise and weight loss characteristics of wheat straw under microwave heating

Xiqiang Zhao, Wenlong Wang, Hongzhen Liu, Yanpeng Mao, Chunyuan Ma, Zhanlong Song*

National Engineering Laboratory of Coal-fired Pollution Reduction, Shandong Provincial Key Lab of Energy Carbon Reduction & Resource Utilization, Shandong University, No. 17923 Jingshi Road, Jinan 250061, PR China

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ABSTRACT

The temperature rise and weight loss characteristics of wheat straw and their mixture under microwave irradiation have been investigated in the laboratory with a fixed bed microwave reactor. The influences of both the microwave power and the additives on the temperature rise and weight loss characteristics of wheat straw are the focuses of this work, which aims to explore a new theoretical foundation for advanced pyrolysis methods. The temperature rise curves show that pure wheat straw had a weak capability to absorb microwave energy, while the mixture of wheat straw and pyrolytic residue had a very strong absorption capability. The ability to absorb microwave energy of the mixture increased with increasing amount of pyrolytic residue. These results are in good agreement with the results of the weight loss curves. Thus, it is feasible to pyrolyze wheat straw in microwave fields by addition of small amounts of pyrolytic residue.

1. Introduction

Pyrolysis is one of the most promising thermo-chemical conversion routes for recovering energy from biomass. For obtaining more valuable products and convenient for operation, present researches on pyrolysis are focusing on improving pyrolysis conditions and achieving directional transformation to the targeted-products by adding catalysts and/or adopting new heating methods such as plasma heating, microwave heating and so forth [1,2].

Microwave heating has merits of "internal heating" and the large-sized sample can be heated to reduce the pretreatment processes of raw material for improving the economics of pyrolysis. Consequently, research activities on microwave pyrolysis have been carried out by more and more researchers, including pyrolysis of coffee hulls [3], wood [4,5], biomass [6], waste tea [7], corn stover [8], wheat straw [9], rice straw [10,11] and oil palm biomass [12,13]. The researches on conventional pyrolysis of larger-sized materials have shown that the temperature rise and weight loss characteristics of materials can reflect relevant information on the pyrolysis process that are important and helpful for further studies on pyrolysis and its heat and mass transfer processes [14,15]. Therefore, it is necessary to investigate the temperature rise and weight loss characteristics of microwave pyrolysis of biomass. However, so far, studies on temperature rise and weight loss characteristics of microwave pyrolysis are few except Liu [16] and Li [17].

Thus, in this study, we investigated the temperature rise and weight loss characteristics of wheat straw under microwave pyrolysis in a special microwave pyrolysis reactor, focusing on the influences of microwave power and additives as well as microwave thermo-gravity. This reactor is especially suitable for measuring the temperature and weight loss of samples.

The change trends of the sample's temperature and weight with heating time under specific conditions (microwave power, mass of sample, and adding different microwave absorbers or catalysts) were investigated, and in particular the effects of special heat and mass transfer methods during the microwave heating process were explored.

In our research, the weight of the sample was about 15 g, and the heat and mass transfer had a significant influence on the entire pyrolysis process. The results of conventional pyrolysis of larger-sized samples were used for comparison. The findings of this study not only reveal the temperature rise and weight loss

* Corresponding author. Tel.: +86 531 88399372; fax: +86 531 88395877.
E-mail address: zlsong@sdu.edu.cn (S. Zhanlong).

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characteristics of biomass during the microwave heating process but also provide necessary reference information for subsequent analysis of pyrolysis products and mechanisms.

2. Materials and methods

2.1. Materials

The biomass used in this study was wheat straw, collected from Liangshan County, Shandong Province in Eastern China. The wheat straw was naturally air dried under sunlight for 30 days to get rid of the surface water. Then it was shredded, grinded and sieved to 0–200 mesh (0–0.090 mm) in preparation for the sample. The proximate and ultimate analysis of the wheat straw sample are carried out, and the content volatile matters is high, 69.3 wt.%. The element contents of C, H, N and S are 41.41 wt.%, 5.86 wt.%, 1.44 wt.% and 0.17 wt.%.

2.2. Experimental apparatus

Microwave reactor employed in the present study is made by Sanle Microwave Technology CO., LTD, and has an ability to alter the power intensity in the range of 0–3000 W at a frequency of 2.45 GHz. The system is equipped with a 2-figure balance (Shanghai, Yousheng BS1000G), a thermocouple (Type K, 0–1000 °C) placed inside a thermo well for temperature measurement, and a computer running proprietary acquisition and control software. The container that holds the sample is made of quartz. The accurate measurement of the temperature of the sample is crucial in thermal studies. Metal objects, including wires, cannot normally be placed in a microwave field without either severely distorting it or causing electrical discharges. However, in confirmation of the findings of Menéndez [18] and Liu [19], it is possible to use thermocouples in a microwave oven provided they are thin, have a grounded metal sheath, and are held exactly at 90° to the electric field component of the microwave energy. In the present study, 1 mm-diameter, stainless steel sheathed type K (chrome-alumel) thermocouples are used, covering the temperature range of 0–1000 °C. More details of this MWR can be found in our previous studies [20].

2.3. Experimental procedure

To investigate the heating behavior by microwave irradiation, a sample dried at 110 °C for 2 h was placed in the quartz crucible, which was positioned at the center of the microwave chamber. The tip of the thermocouple was placed at the center of the sample for every test. After the quartz crucible was correctly positioned and the quartz tube was tightly sealed, a constant flow (3 L/min) of carrier gas (N_2) was purged into the reactor. When the purging was sufficient to produce an anoxic state, the microwave generator was turned on and switched to the designated power.

As the reaction proceeded, the reaction temperature was recorded at periodic intervals. After a desired residence time, the microwave generator was stopped and temperature was recorded. The carrier gas kept purging until the solid residue (coke) was self-cooled to below 100 °C.

The weight loss experimental steps are: the sample is weighed to the nearest ± 0.001 g, and placed together with the quartz crucible on the PTFE tray inside the furnace, and the door is tightly closed and sealed; N_2 is continuous introduced for 15 min and the flow is 3 L/min, maintaining the inert protective atmosphere in furnace; start the microwave, and the weight data are automatically collected and recorded by the data acquisition system; when the heating time preset is reached, the microwave is closed, and the N_2 is continuously fed until the crucible and the sample are cooled to the room temperature; remove the crucible, and weigh the remaining solids immediately.

3. Results and discussion

3.1. Weight loss characteristics of wheat straw

This section focuses on the weight loss characteristics of wheat straw during microwave heating. The mass of pure wheat straw powder used in experiments including different microwave power and additives is 15 g.

3.1.1. Effect of microwave power

The weight loss curves of the sample under different microwave powers (P) are shown in Fig. 1a. With increasing microwave power, the duration of the weight loss process was significantly shortened, and the total weight loss significantly increased. Depending on the weight losses of the sample under different microwave powers, the microwave power range can be divided into three sections: (1) $P \leqslant 250$ W, where there is only one severe weight loss process with a small total weight loss; (2) $250 < P < 500$ W, where there are two severe weight loss processes, but with a long interval between them, ranging from 2 to 14 min; and (3) $P \geq 500$ W, where two severe weight loss processes occur, but the two processes overlap. The first weight loss process does not end when the second weight loss process has begun.

When microwave power was below 250 W, the total weight loss of the sample did not exceed 40 wt.%, and only one violent release of gases occurred in the entire heating process. This is because straw has a high transmittance of microwaves, and the sample can only absorb a small amount of microwave energy. As a result, only drying and pyrolysis of partial sample occur during the heating process [10]. Furthermore, microwave heating has the characteristic of "selective heating". Heat loss is large near the wall of the container, resulting in low temperature and some of the sample not being pyrolyzed, and the weight loss is primarily attributable to pyrolysis of internal straw.

Microwave power 300 W is a special turning point. After the first weight loss, the weight was maintained for a long time and almost unchanged; while after circa 18 min, suddenly, a dramatic second weight loss occurred, which was about 20–25 wt.% of the total weight of the initial sample. The total weight loss of the two processes accounted for 70 wt.% or more of the total weight of the initial sample. After the experiment, most of the sample was pyrolyzed completely, and only a very small amount of the sample (no more than 0.5 g) close to the inner wall surface of the container was not completely pyrolyzed. These phenomena further validate the speculation that the internal sample is first pyrolyzed, and the external sample is pyrolyzed later.

The interval between the two weight loss processes at microwave power 350 W and 450 W was significantly shortened to 3–4 min, while the interval at microwave power 300 W was 14 min. Furthermore, the two weight loss processes overlapped as one process at microwave power 500 W, and it was difficult to distinguish between them. The total weight loss increased from 30 wt.% at 200 W to about 75 wt.% at 500 W.

The weight loss curve of the sample at microwave power 350 W can be divided into four stages:

1. Drying stage. The adsorbed water of the sample is lost, and the weight loss was 7–8 wt.%. With increasing microwave power, the duration of this stage was significantly shortened.
2. The first weight loss stage. The duration of this stage and the weight loss of the sample were significantly different at different microwave powers. At $P \leqslant 250$ W, with increasing microwave