Microwave-assisted Pyrolysis of Cotton Stalk with Additives

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The purpose of this study was to investigate the effects of microwave power and additives on the microwave-assisted pyrolysis of cotton stalk. Pyrolysis temperature profiles, product yields, and bio-oil components were analyzed. The results indicated that as the microwave power increased, so did the final temperature and heating rate. When microwave power increased from 800 to 1200 W, the yield of the solid residue decreased and gas yield increased, but the maximum bio-oil yield of 26.7% was achieved under a microwave power of 1000 W. All three of the additives (NaOH, Na_2CO_3, and H_3PO_4) increased the yields of solid residue and gas, while the yield of the bio-oil decreased. Gas chromatography-mass spectrometry (GC-MS) analysis indicated that the main components of bio-oil from microwave-assisted pyrolysis of pure sample were acetol, furfural, 2-methoxy-phenol, and 4-methyl-2-methoxy-phenol. Sodium hydroxide and sodium carbonate (NaOH and Na_2CO_3) greatly favor the formation of acetol. Phosphoric acid (H_3PO_4) remarkably promoted the formation of furfural and 4-methyl-2-methoxy-phenol, while H_3PO_4 inhibited the formation of acetol.

INTRODUCTION

Because of the fast depletion of fossil fuels and the increasing environmental concerns resulting from their use, developing renewable energy to substitute for fossil fuels is imperative (Mohan *et al*. 2006). Biomass is the only renewable resource that can be converted into liquid, gas, and solid fuels (Bridgwater and Peacocke 2000). Biomass, as an abundant and potentially CO_2-neutral renewable energy, has gained great interest from all over the world (Müller-Hagedorn *et al*. 2003). Biomass conversion routes can be divided into two categories: biochemical conversion technologies, which include digestion and fermentation, and thermochemical conversion technologies, which include combustion, gasification, and pyrolysis (Chen *et al*. 2008).

Thermochemical conversion processes are thought to have great promise as a means for biomass utilization. In the thermochemical conversion processes, pyrolysis has received increased interest. Pyrolysis can convert biomass in the absence of oxygen to solid (char), liquid (bio-oil), and gaseous products whose yields and components depend on feedstock characteristics (Font and Williams 1995; Raveendran *et al*. 1995; Williams and Besler 1996; Chen *et al*. 2003; Kawamoto *et al*. 2008) and pyrolysis conditions (including heating rate, pyrolysis temperature, and additives). Among pyrolysis products, bio-oil has the advantages of easy storage and transport. Moreover, bio-oil can be used as fuels and chemical feedstock (Bridgwater and Peacocke 2000).

Pyrolysis includes conventional pyrolysis and microwave-assisted pyrolysis. Compared with conventional pyrolysis, microwave-assisted pyrolysis has shown several advantages, such as fast and selective heating, easy control, and energy efficiency (Macquarrie *et al*. 2012). Microwave-assisted pyrolysis can obtain greater proportions of syngas (H_2+CO) and lower proportions of environmentally harmful polycyclic aromatic hydrocarbons (PAHs) than conventional pyrolysis (Domínguez *et al*. 2006). However, the scaling-up of microwave-assisted pyrolysis is the main challenge in comparison with conventional pyrolysis (Luque *et al*. 2012). Studies of microwave-assisted pyrolysis of biomass of some agricultural and forestry residues, such as wood block, pine sawdust, fir sawdust pellet, corn stover, wheat straw, and rice straw, have been recently reported (Miura *et al*. 2004; Huang *et al*. 2008, 2010, 2013, 2015; Chen *et al*. 2008; Wang *et al*. 2009; Lei *et al*. 2009; Wan *et al*. 2009; Ren *et al*. 2012; Bu *et al*. 2013; Zhao *et al*. 2013, 2014; Shang *et al*. 2015).

Results have shown that some additives (activated carbon, Na_2CO_3, K_2CO_3, and $MgCl_2$) in the microwave field can improve the yield of hydrogen-rich gas or the content of a target component in bio-oil. However, there are few studies on microwave-assisted pyrolysis of cotton stalk. In this study, microwave-assisted pyrolysis of cotton stalk was carried out under various microwave powers and with additives (NaOH, Na_2CO_3, and H_3PO_4). The pyrolysis temperature profiles, product yields, and the components of the bio-oil product were analyzed.

EXPERIMENTAL

Materials

The biomass used in this study was cotton stalk, collected from the suburban areas of Huainan, Anhui province, China. The result of the proximate analysis of the cotton stalk is listed in Table 1, where moisture is weight loss percentage on air dry basis at 75 °C, ash is the residue percentage on dry basis after complete combustion at 800 °C, volatile is weight loss percentage on dry basis after devolatilization at 700 °C for 10 min under scarce oxygen atmosphere, and fixed carbon is calculated by difference.

Table 1. Proximate Analysis[a] (wt.%)

Moisture	Volatile	Ash	Fixed carbon
7.65	69.03	6.94	16.38

a: Air-dry basis

NaOH, Na_2CO_3, and H_3PO_4 were purchased from the Sinopharm Chemical Reagent Co. Ltd., China. The purity of the three additives is AR (analytical reagent). The concentration of H_3PO_4 was 85%.

Preparation of Samples

The cotton stalk was naturally dried in air and was then ground and sieved to less than 1 mm in size. Finally, it was dried in the drying oven at 105 °C until constant weight and then was stored in a desiccator for use. An additive (6 g) was put into a beaker containing 400 mL of distilled water, then 60 g of dry cotton stalk sawdust was added to the beaker, followed by vigorous stirring until mixing uniformity at room temperature was

reached. The mixture in the beaker was then transferred to a tray and was subjected to drying at 105 °C until a constant weight was reached. Finally, the mixture was stored in a desiccator for experiments.

Experimental Apparatus

A schematic diagram of the experimental apparatus for microwave-assisted pyrolysis of cotton stalk is shown in Fig. 1. The experimental apparatus included a microwave oven (NJL4-2, Nanjing Jiequan Microwave Oven Equipment Co. Ltd., China) carrying a thermocouple and a beaker-shaped quartz reactor (13 cm in length, 12 cm outer diameter), condenser system, liquid collecting system, gas collecting bag, and carrier gas (N_2) bottle.
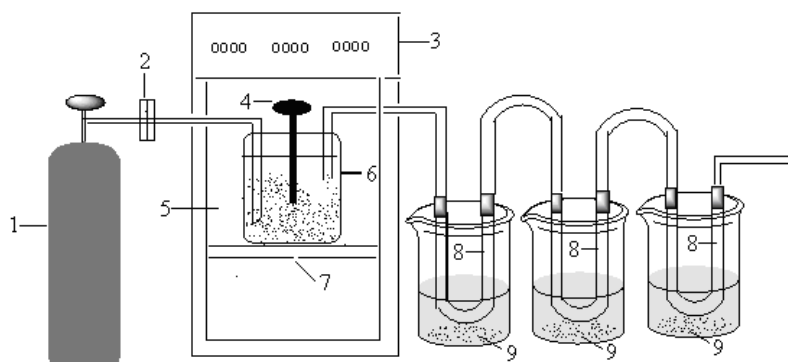

Fig. 1. Schematic diagram of microwave-assisted pyrolysis system: (1) N_2 bottle; (2) flow meter; (3) control system of microwave pyrolysis oven; (4) thermocouple; (5) microwave cavity; (6) quartz reaction reactor; (7) holder; (8) U-shaped tube; (9) ice water condenser

Microwave-assisted Pyrolysis Procedure

The pyrolysis of sample was carried out in the microwave oven with a maximum power of 2000 W at a frequency of 2450 MHz. The prepared sample (15 g of pure sample or 16.5 g of mixed sample) was placed into the quartz reactor, and the reactor was then placed inside the microwave cavity. To maintain an inert atmosphere, the carrier gas (N_2), with a flow rate of 3 L/min, was purged into the reactor system for 15 min before the experiment. Then, the microwave oven was turned on and set to the designated power. The pyrolysis temperature, measured by a thermocouple, was recorded at periodic intervals (2 min). The pyrolysis time was set for 20 min after the temperature was stabilized. After the designated reaction time passed, the microwave was turned off. The condensable volatiles (bio-oil products) were collected using three U-shaped tubes immersed in ice water. The non-condensable volatiles (gas products) were discharged into the atmosphere. The solid residue was collected in the reactor.

The yields of the solid and liquid fraction were calculated from the weight of each, while the yield of the gas fraction was calculated by the difference based on the mass balance. All of the experiments were repeated three times to ensure accuracy of the experimental results. The final result is the average value of these three experimental results.

Product Yield Calculation

The computing formulas for product yields are as follows,

$$\eta_s = \frac{m_s - m_{sa}}{m_o} \times 100\% \tag{1}$$

$$\eta_l = \frac{m_l - m_{la}}{m_o} \times 100\% \tag{2}$$

$$\eta_g = 100\% - \eta_s - \eta_l \tag{3}$$

where η_s is the yield of solid residue (%), η_l is the yield of bio-oil (%), η_g is the yield of gas (%), m_s is the weight of solid residue (g), m_l is the weight of bio-oil (g), m_0 is the weight of the sample (g), m_{sa} is the weight of the solid additive (g), and m_{la} is the weight of the liquid additive (g).

Analysis of Bio-oil Components

The chemical components of the bio-oil products were determined using a gas chromatograph-mass spectrometer (GC-MS; Shimadzu QP5050A, Japan) with a capillary column (DB-17, 30 m × 0.25 mm i.d. × 0.25 mm) after dehydration using absorbent cotton and anhydrous sodium sulfate. Acetone was used as the solvent to dilute the dehydrated liquid product to a concentration appropriate for analysis. The solvent cut time was set to 2 min. Helium was employed as the carrier gas with a flow rate of 1.0 mL/min. The detector temperature was set to 230 °C. The injection temperature was set to 300 °C. The column oven was initially maintained at 60 °C for 4 min, then increased to 140 °C with a heating rate of 10 °C/min. After that, the temperature was increased to 148 °C with a heating rate of 1 °C/min. Finally, the temperature was increased to 280 °C with a heating rate of 33 °C /min and kept for 5 min. The compounds of bio-oil were identified by comparing their mass spectra with the National Institute of Standards and Technology (NIST) Mass Spectral library.

RESULTS AND DISCUSSION

Effect of Microwave Power on Pyrolysis Temperature Profile without Additives

The pyrolysis temperature profiles of pure samples under various microwave powers are given in Fig. 2. The pyrolysis temperature profiles can be divided into two stages. The first stage is the section where the temperature rose rapidly. In this stage, the sample was dried and pyrolyzed quickly. The water inside the cotton stalk and the coke produced from the pyrolysis process are good microwave absorbers. Therefore, the temperature increased rapidly. In the meantime, some volatiles were evolved. The second stage was the section where the temperature was maintained approximately at a constant level. This was possibly because of the balance between the heat lost and the heat generated by microwave. It can be seen that both the final temperature and the heating rate increased as the microwave power increased. This occurred because the microwave energy absorption of the sample increased as the microwave power increased, so the pyrolysis temperature became higher and the pyrolysis time became shorter. These results are similar to those reported in the literature (Huang *et al*. 2010; Du *et al*. 2011; Li *et al*. 2013). The

variations are primarily a result of differences in the sample components and the characteristics of the microwave pyrolysis system.
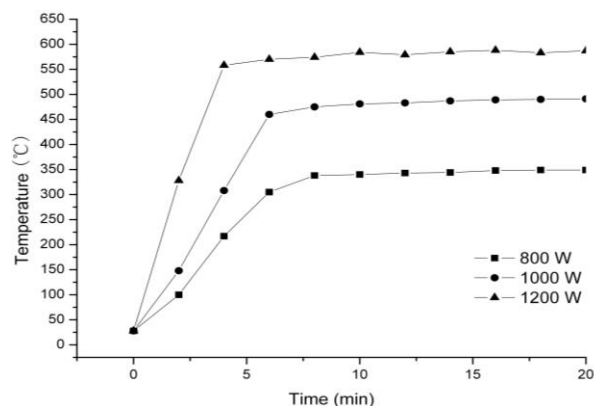

Fig. 2. Temperature profiles under various microwave powers

Effect of Microwave Power on Product Yield without Additives

The product yields of pure samples under various microwave powers are shown in Fig. 3. When the microwave power was increased, the yield of the solid residue reduced gradually and the gas yield increased gradually, while the yield of the bio-oil increased initially and then decreased. The maximum bio-oil yield of 26.7% was obtained at 1000 W.
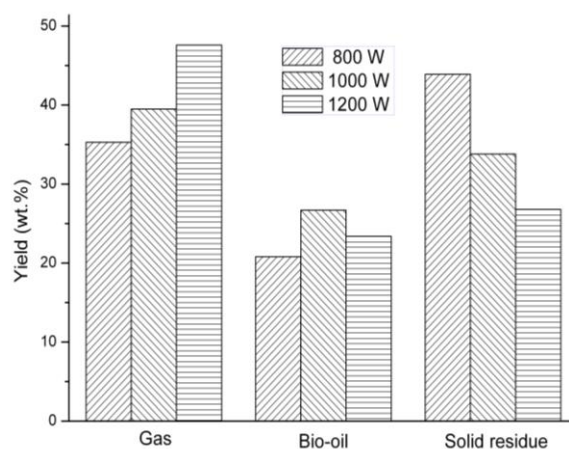

Fig. 3. Product yields under various microwave powers

The fundamental reason for these behaviors was that the absorbed microwave energy was not sufficient to pyrolyze the sample completely below 1000 W, and it was decomposed completely at the microwave power where the maximum oil yield was obtained. Above 1000 W, the decrease in the bio-oil yield and increase in the gas yield was primarily caused by the secondary cracking of condensable volatiles into incondensable gases at high temperatures. Lower power with lower heating rate was apt to produce a higher yield of the solid residue, while higher power with higher pyrolysis temperatures always led to gasification reactions, which therefore decreased the yield of the bio-oil and increased the yield of the gas. These results are similar to those reported in the literature (Huang *et al*. 2010; Du *et al*. 2011; Hu *et al*. 2012; Li *et al*. 2013). In this study, 1000 W

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was the optimal power for obtaining the highest bio-oil yield, of 26.7%; therefore, in the following experiments, 1000 W was selected as the pyrolysis power.

Effect of Additive on Product Yield

The product yields with various additives are given in Fig. 4. The three additives (NaOH, Na_2CO_3, and H_3PO_4) increased the yields of the solid residue and gas while decreasing the yield of the bio-oil. The solid yields' increase was in agreement with a previous study (Wang *et al*. 2006a). This increase may have occurred because the three additives could promote the formation of coke. Meanwhile, the bio-oil vapor secondary cracking at high temperatures would lead to the production of more gas. Therefore, as the bio-oil yield reduced, the gas yield increased. However, because of the complexity and sensitivity of microwave-assisted pyrolysis, it was still difficult to precisely determine the catalytic effect of the additive on the product yield.

Biomass microwave-assisted pyrolysis produced low yields of bio-oil in previous reports. One report found that 22.6 wt.% bio-oil yield was obtained from microwave-assisted pyrolysis of rice straw at a temperature of 407 °C with a power input of 300 W (Huang *et al*. 2008). Another report stated that 31.5 wt.% bio-oil yield was obtained from wood block microwave-assisted pyrolysis with a power consumption of 1.11 kWh/kg and reaction time of 11 min (Miura *et al*. 2004). Ren *et al*. (2012) reported that 57.8 wt.% bio-oil yield, based on dry biomass, was obtained from microwave-assisted pyrolysis of fir sawdust pellets with a reaction temperature of 471 °C and a reaction time of 15 min. Zhao *et al*. (2013) found that approximately 33 wt.% bio-oil yield was obtained from microwave-assisted pyrolysis of wheat straw at a reaction temperature of 500 °C with a power input of 400 W. Addition of three additives (Na_2CO_3, K_2CO_3, and CaO) made the bio-oil yield decrease. Bu *et al*. (2013) noted that different activated carbon additives decreased the bio-oil yield from microwave-assisted pyrolysis of fir sawdust pellets; the bio-oil yield decreased to about 30% from 45.2%. In the present research, the yield of the bio-oil was 26.7 wt.%, obtained from microwave-assisted pyrolysis of cotton stalk with a power input of 1000 W and a reaction time of 20 min. All three additives (NaOH, Na_2CO_3, and H_3PO_4) decreased the bio-oil yield. But conventional fast pyrolysis in a fluidized bed had a bio-oil yield of 70% to 80% (Mohan *et al*. 2006). Compared with the fluidized bed fast pyrolysis, microwave-assisted pyrolysis produces a much lower bio-oil yield.
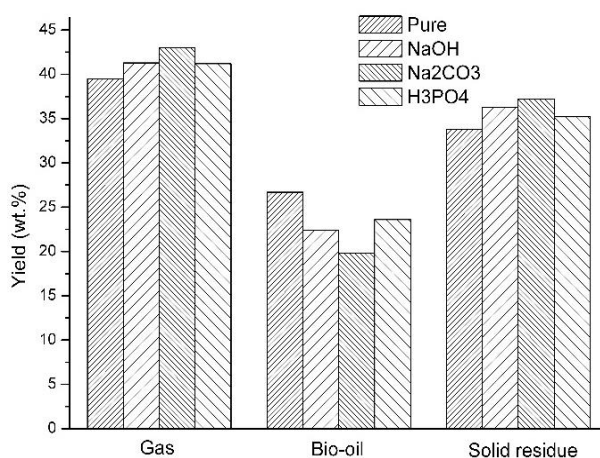

Fig. 4. Product yields with various additives under 1000 W microwave power

Effect of Additive on Components of Bio-Oil Products

Total ion chromatograms (TIC) from GC-MS analysis of the bio-oil products from microwave-assisted pyrolysis of pure sample and samples treated with the three additives are shown in Fig. 5. Although the composition of the bio-oil from microwave-assisted pyrolysis of untreated sample was complex, the main components included acetol, furfural, 2-methoxy-phenol, and 4-methyl-2-methoxy-phenol. The most abundant organic component in the bio-oil was acetol. A trace amount (0.12%) of levoglucosan was found in the bio-oil. However, Miura *et al.* (2004) obtained a considerable amount of levoglucosan from pyrolysis of wood blocks by microwave heating, whereas a considerable amount of acetol was obtained instead of levoglucosan in the pyrolysis of untreated cotton stalk by microwave heating. The reason for this was that the levoglucosan can be decomposed to gas and volatiles at a high temperature and long reaction residence time (Ren *et al.* 2012). Moreover, the type and concentration of the dominant components were governed by the additive types. Table 2 shows the relative peaks percent of the main components in the bio-oil with different additives obtained by GC-MS analysis.

Both NaOH and Na_2CO_3 greatly favored the formation reaction of acetol. The relative concentration of acetol increased to about 50% from 35.24%.

H_3PO_4 remarkably promoted the formation of furfural and 4-methyl-2-methoxy-phenol. Meanwhile, H_3PO_4 inhibited the formation of acetol. The total relative peaks of these two components, *i.e.*, furfural and 4-methyl-2-methoxy-phenol, were up to 70%. However, the relative concentrations of furfural and 4-methyl-2-methoxy-phenol in bio-oil from microwave-assisted pyrolysis of pure sample was less than 10%.
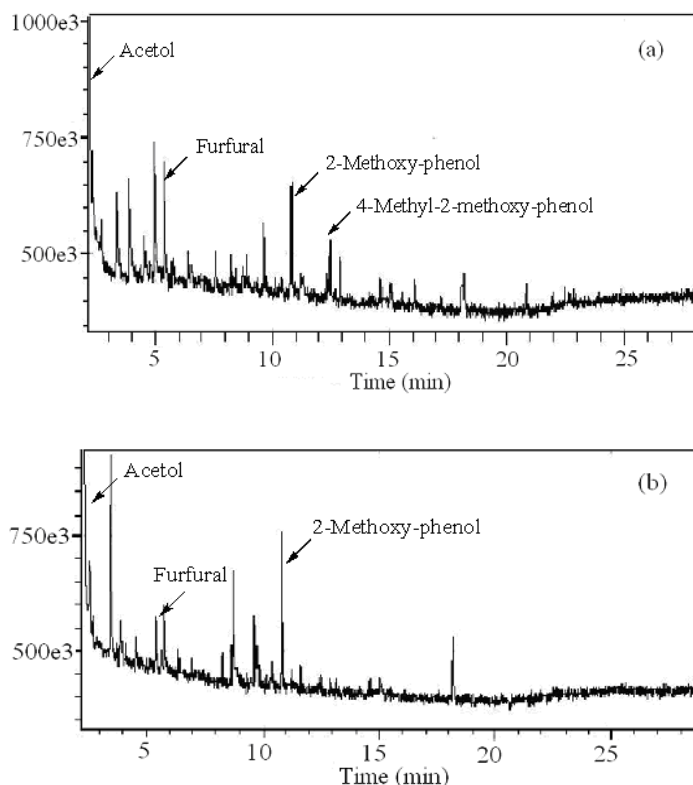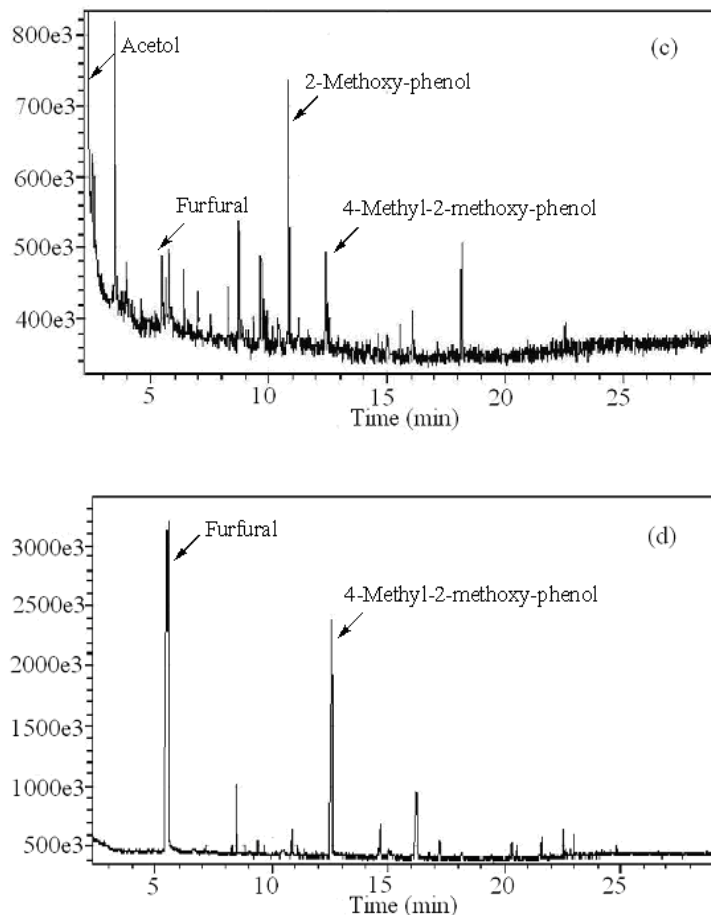
Fig. 5. TIC for bio-oil products from microwave-assisted pyrolysis: (a) pure; (b) 10% NaOH; (c) 10% Na$_2$CO$_3$; (d) 10% H$_3$PO$_4$

Table 2. Relative Peaks (Percent) of the Main Components in Bio-Oil with Various Additives

Compounds	Relative peaks (%)			
	Pure	NaOH	Na$_2$CO$_3$	H$_3$PO$_4$
Acetol	35.24	53.55	49.28	0
Furfural	7.95	2.27	2.86	43.15
2-Methoxy-phenol	4.96	4.11	4.25	1.49
4-Methyl-2-methoxy-phenol	2.47	0	5.12	26.78

Biomass is primarily composed of three components: cellulose, hemicelluloses, and lignin. The reaction mechanism and characteristics of the bio-oil with respect to the three components have been widely investigated in previous research. Cellulose can be mainly decomposed to levoglucosan, acetaldehyde, and acetol; hemicelluloses can be mainly decomposed to acetic acid and furfural; and lignin can be mainly decomposed to phenols (Liao *et al*. 2003; Wang *et al*. 2003, 2006b; Tan *et al*. 2005; Shen and Gu 2009; Shen *et al*. 2010a,b; Lu *et al*. 2011).

There have been some studies on the reaction mechanism of biomass microwave-assisted pyrolysis. A possible reaction pathway for acetol formation from levoglucosan secondary cracking was proposed by Chen *et al*. (2008). Ren *et al*. (2012) proposed the reaction pathway of Douglas fir pellet's three components: cellulose, hemicelluloses, and lignin pyrolysis. However, the reaction mechanism of biomass microwave-assisted pyrolysis still needs further study.

CONCLUSIONS

1. Higher microwave power led to a higher final temperature and heating rate. When increasing the microwave power from 800 to 1200 W, the yield of the solid residue decreased and the gas yield increased; the microwave power of 1000 W was found to be optimal for achieving the maximum bio-oil yield of 26.7%.

2. All three additives (NaOH, Na_2CO_3, and H_3PO_4) increased the yields of solid residue and gas, but decreased the yield of bio-oil.

3. Gas chromatography-mass spectrometry analysis indicated that the main components of bio-oil from microwave-assisted pyrolysis of pure sample were acetol, furfural, 2-methoxy-phenol, and 4-methyl-2-methoxy-phenol. Both NaOH and Na_2CO_3 greatly favored the formation of acetol. H_3PO_4 remarkably promoted the formation of furfural and 4-methyl-2-methoxy-phenol while inhibiting the formation of acetol.

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