



# A Comparative study of microwave-induced pyrolysis of lignocellulosic and algal biomass



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## HIGHLIGHTS

- Microwave pyrolysis of algal and lignocellulosic biomass is studied.
- Activation energy for pyrolysis of samples was calculated by Coats–Redfern method.
- Temperatures as high as 1170 °C were achieved by mixing samples with lignite char.
- Bio-oil from lignocellulosic biomass contained more single-ring aromatics.
- Bio-oil from microalgae had a higher content of nitrogen-containing compounds.

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## ABSTRACT

Microwave (MW) pyrolysis of algal and lignocellulosic biomass samples were studied using a modified domestic oven. The pyrolysis temperature was recorded continuously by inserting a thermocouple into the samples. Temperatures as high as 1170 and 1015 °C were achieved for peanut shell and *Chlorella vulgaris*. The activation energy for MW pyrolysis was calculated by Coats–Redfern method and the values were 221.96 and 214.27 kJ/mol for peanut shell and *C. vulgaris*, respectively. Bio-oil yields reached to 27.7 wt.% and 11.0 wt.% during pyrolysis of *C. vulgaris* and peanut shell, respectively. The bio-oil samples from pyrolysis were analyzed by a gas chromatography–mass spectrometry (GC–MS). Bio-oil from lignocellulosic biomass pyrolysis contained more phenolic compounds while that from microalgae pyrolysis contained more nitrogen-containing species. Fourier transform infrared spectroscopy (FTIR) analysis results showed that concentration of OH, C–H, C=O, O–CH<sub>3</sub>, and C–O functional groups in char samples decreased significantly after pyrolysis.

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## 1. Introduction

The continuously increased energy demand and limited fossil fuel reserves has motivated scientists to look for alternative energy sources. Biomass is one of the most promising renewable energy sources for near future energy, constituting 14% of the global primary energy and being the fourth largest following coal, oil, and natural gas (Yin, 2012). Biomass is a carbon neutral material that can be used in order to reduce the greenhouse gas (GHG) emissions (Wang et al., 2007). Lignocellulosic biomass is mainly composed of hemicellulose, cellulose, and lignin and also contains a variety of minor components (inorganic matters). Cellulose (40–80 wt.% of biomass) is a crystalline, high molecular weight polymer of glucose

which decomposes at 240–350 °C to produce anhydrocellulose and levoglucosan. Hemicellulose (15–30 wt.% of biomass) is an amorphous shorter polymer of various sugars, decomposing at 200–260 °C. Hemicellulose decomposition produces more volatiles, less tar and less char than cellulose. Lignin (10–25 wt.% of biomass) is an aromatic polymer that decomposes at 280–500 °C to produce phenols. Lignin decomposition yields more char than cellulose (Yin, 2012). As an alternative to lignocellulosic biomass, algal biomass features faster growth, higher yields, higher efficiency in CO<sub>2</sub> capture and photosynthesis (Sanchez-Silva et al., 2013; Tahmasebi et al., 2013). Microalgae can be cultivated using marine or fresh water, therefore, it does not compete with agricultural crops for land (Tahmasebi et al., 2013). Microalgae contain three main components, namely carbohydrates, proteins and lipids. The ash content and concentration of alkali and alkali earth metals (AAEM) in microalgae varies between different species and is dependent on the culturing method. The presence of AAEM species in biomass

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can cause slagging and fouling during combustion and gasification. An alternative use of biomass to combustion is the fast pyrolysis of biomass to produce fine chemicals, liquid fuels and synthesis gas.

Pyrolysis can be described as the thermal decomposition of the organic components in an oxygen-free atmosphere to yield char, oil and gaseous products at 400–600 °C (Antonakou et al., 2006). Conventional pyrolysis using fixed- and fluidized-bed reactors involves transfer of energy by superficial heating via convection, conduction and radiation (Yin, 2012). Conventional pyrolysis faces some challenges such as heat transfer resistance, heat losses to surrounding (inefficient heating), non-selective heating, utilization of portion of heat supplied to biomass materials and less controllable process (Salema and Ani, 2011). Application of conventional pyrolysis for large particle sizes is also limited (Bridgwater, 1999). In conventional heating, heat is transferred along temperature gradient from particle surface to particle center. In contrast, microwave heating involves direct conversion of electromagnetic energy into heat at molecular level (Bu et al., 2012).

Microwave energy is a non-ionizing electromagnetic radiation with frequencies ranging from 300 MHz to 300 GHz (wavelength ranging from 1 to 300 mm) (Uslu and Atalay, 2004). Two main mechanisms of dipolar polarization and ionic conduction are involved in MW dielectric heating of materials (Gabriel et al., 1998; Mingos and Baghurst, 1991). MW heating has some unique features: (1) It is quick and efficient and can be turned on and off instantly; (2) Features in-core volumetric and uniform heating (the whole particle heats simultaneously); (3) Penetration of MW energy into the materials is instant; (4) Enhanced mass transport allows faster escape of volatile species out of the bulk material/particle which leads to increase in oil yield (due to less secondary reactions); (5) Selective heating i.e. rapid intense heating for polar substances (Yin, 2012). Better control of the process and more desired products can be obtained due to more uniform temperature distribution in MW heating and the undesired secondary reactions can be minimized (Zhao et al., 2011). Since heat is transformed by MW energy penetration within feedstock internally, the large-sized materials can be processed therefore the size reduction costs can be avoided (Tahmasebi et al., 2011).

Biomass is a poor microwave absorber. It has been reported that some inorganic compounds such as Fe<sub>3</sub>O<sub>4</sub>, CuO, V<sub>2</sub>O<sub>5</sub> as well as carbon undergo remarkable heating under MW and may reach temperatures as high as 700 °C in less than 1 min (Monsef-Mirzai et al., 1992; Yagmur et al., 2008). A method of utilizing MW for biomass pyrolysis is therefore to mix the biomass with an effective MW receptor (Bu et al., 2012; Salema and Ani, 2012a, 2011). The MW pyrolysis of biomass is a versatile process since the operating conditions such as temperature, heating rate and residence time can be optimized to maximize the production of char, oil or gas depending on which product is of interest (Dominguez et al., 2007).

Kinetics analysis is important in order to understand the mechanism of decomposition and the rate of chemical reactions during biomass pyrolysis. Biomass components in lignocellulosic and algal biomass decompose at different temperature ranges and rates. The kinetics analysis of biomass pyrolysis reported in the literature is usually carried out by using light weight samples usually in a thermogravimetric analyzer (TGA). A detailed understanding of MW pyrolysis characteristics of different types of biomass as a function of process parameters and the pyrolysis product analysis will have a significant contribution in investigating the potential application of this technology. Previous investigations were concerned on the pyrolysis conditions required to obtain a maximum bio-oil yield. The objective of this study was to investigate the effects of MW receptor, temperature, and MW power output on the yield and characteristics of products from lignocellulosic and algal biomass. Bio-oil and bio-char product from the MW pyrolysis were analyzed in detail using GC–MS and FTIR. The kinetics parameters for MW

pyrolysis of lignocellulosic and algal biomass were also obtained. The results obtained in this study can also help to understand the influence of the biomass feedstock on pyrolysis characteristics and product composition.

## 2. Experimental

### 2.1. Material

The microalgae *Chlorella vulgaris* (*C. vulgaris*) sample was obtained from Spirulina Bio-Engineering Co. Ltd., China in powder form. The peanut shell sample was from North China. As-received peanut shell samples were crushed and sieved to particle size of <1 mm. The samples were then dried in a vacuum oven at 105 °C for 12 h to remove the moisture and then stored in desiccators. The proximate and ultimate analyses of the samples are given in Table 1. Lignite char and magnetite (Fe<sub>3</sub>O<sub>4</sub>) (Sinopharm Chemical Reagent Co. Ltd., China) were used as MW receptor in pyrolysis experiments. Lignite char was prepared by pyrolysing lignite at 700 °C in a fixed-bed quartz reactor for 30 min.

### 2.2. Microwave-induced pyrolysis experiments

Pyrolysis experiments were carried out using a quartz reactor which was placed in a modified domestic MW oven with maximum power output of 700 W and frequency of 2.45 GHz. The reactor was designed in the way that volatile matter including bio-oil and gaseous products could leave the reactor immediately. Experimental setup and procedure were detailed elsewhere (Wang et al., 2014). Briefly, experiments were carried out by placing biomass samples (ca. 6 g) in the quartz reactor. Microwave receptor (lignite char or Fe<sub>3</sub>O<sub>4</sub>) was evenly blended with biomass at the mass ratio of 1:5, 2:5 and 3:5. A N<sub>2</sub> flow rate of 400 ml/min was passed through the sample bed for 30 min prior to pyrolysis experiment to ensure that air was flushed completely out of the sample bed. MW output power varied in the range of 390 to 700 W. A thin thermocouple was inserted into the biomass sample from the top of the reactor and the temperature of biomass sample was recorded at 10 s intervals. The yields of solid and liquid products were measured by directly weighing each fraction whereas the gas production was calculated by the difference. All the experimental results are an average of at least three experimental runs. The yields of pyrolysis products were calculated from the following equations:

**Table 1**  
Proximate and ultimate analyses of peanut shell and *C. vulgaris* biomass.

Sample	Peanut shell	<i>C. vulgaris</i>
Moisture (wt.%, ar)	8.03	6.26
Volatile matter (wt.%, ar)	58.39	76.13
Fixed carbon (wt.%, ar)	22.29	11.50
Ash (wt.%, ar)	11.31	6.11
C (wt.%, daf)	37.87	47.32
H (wt.%, daf)	5.18	6.9
N (wt.%, daf)	1.57	8.48
S (wt.%, daf)	0.14	0.85
O <sup>a</sup> (wt.%, daf)	55.24	36.45
H/C <sup>b</sup>	1.64	1.75
O/C <sup>b</sup>	1.09	0.58
Biomass LHV (MJ/kg)	17.71	22.05
Bio-oil LHV (MJ/kg)	27.96	30.45

ar: as received; daf: dry ash free.

<sup>a</sup> Calculated by difference.

<sup>b</sup> Molar ratio.

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