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Scaled-up reactor for microwave induced pyrolysis of oil palm shell

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ABSTRACT

We designed and implemented bench-scale equipment for the microwave pyrolysis of biomass, and tested its effects on oil palm shell (OPS). The design uses the interference of two MW sources to increase the electric field intensity, based on model predictions by finite elements simulations. The MW pyrolysis experiments were done varying the mixture ratio of OPS and activated carbon (75:25, 50:50, or 25:75 by weight), the MW power (450 or 800 W), and the use of MW source magnetrons (left, right, or both). The pyrolysis temperature depended on the varied experimental parameters. The highest temperature and the highest yields of liquid bio-oil and gas were obtained with the 75:25 mixture treated with 800 W total power using both magnetrons. The end-products were analyzed with bomb calorimeter, GC–MS, and the solid residue with SEM. The results demonstrate that the bench-scale equipment is suited for experimental work on the pyrolysis of biomass (OPS).

1. Introduction

The biggest issues of the 21th century include the world's population growth and the increasing energy consumption. Some countries are less vulnerable to negative effects, but all countries can be impacted by the human energy use that also grows with the standard of living. However, the energy production dominantly relies on fossil fuels, and by the end of the 21th century the global energy use might be similar over each 10–20 year period as the total use from the dawn of civilization up to now by all of mankind [1]. We have to consider the energy reserves and alternative fuels to satisfy such energy demands. Biomass is a natural renewable alternative that could reduce the impacts of these issues, potentially by replacing some use of fossil fuels in such manner that also limits environmental impacts.

Thailand is an agriculturally strong country, with favorable climate for rapid growth of crops, in a sense a kitchen of the world that also exports food products. The raw materials come from aquaculture and agriculture, the latter products including rice, corn, sugar cane, coconut, and oil palm. Oil palm plantations have flourished in southern Thailand, and the Agricultural Research and Development Agency (Public Organization; ARDA) has reported at the end of year 2010 its palm oil production as 9,229,573 tons. Thus, southern Thailand has much palm oil related industry producing palm oil traditionally by crushing, refining, and fractionation processes, but also in alternative ways. The crushing of fruit bunches provides crude palm oil and crude palm kernel oil, while oil palm fiber and oil palm shell (OPS) are residuals from this process. These are examples of abundant streams of biomass that are largely waste or at least low-cost, and that are renewable and reliable sources of fairly homogeneous biomass in Thailand.

The thermo-chemical process named pyrolysis takes place when oxygen is absent or at least lacking for full combustion, and can generate useful end-products including bio-char, bio-oil, and syngas. During pyrolysis the large hydrocarbon molecules decompose into smaller ones. This process can upgrade biomass to end-products that are solids, liquids and gases with higher energy densities per unit mass (high heating values). The solid bio-char can directly be used as a solid fuel, and alternatively it can be used as adsorbent activated carbon (AC) in purification applications; moreover, it can be used for soil improvement. As for the bio-oil and the gases, they can be used as fuels or refined to higher value fuels and chemicals.

Microwave (MW) heating is based on the dielectric heating of a material when subjected to microwaves. Thus, all materials do not have the appropriate dielectric properties to be good MW absorbers [2–7]. In fact, biomass tends to have poor dielectric properties, so that carbonaceous materials such as char, activated carbon, graphite, etc. are commonly mixed with biomass to make it a better MW absorber. The advantages of MW heating consist of

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volumetric heating, allowing large-sized feedstock, product quality benefits from uniformity of heating, enhanced chemical activity, and time and energy savings due to high thermal efficiency [8,9]. There are several prior studies on the experimental MW pyrolysis of various materials, including Chen et al. [10] and Huang et al. [11] that pyrolysed pine wood sawdust and rice straw, respectively, both in nitrogen atmosphere, while Dominguez et al. [12] used MW to pyrolyse coffee hulls in helium atmosphere. Oil palm biomass [13,14] was pyrolysed with MW heating so that nitrogen flushing of the reactor got particular attention. A good example of MW experiments and techniques is Abubakar et al. [15], in which the inert nitrogen gas was introduced at the top of the reactor, while vapor was condensed by a condensing unit at the bottom of the reactor. This arrangement helps avoid bio-oil deposition in the experimental equipment.

However, household MW units have been used in these prior studies, although with a quartz reactor inside the cavity. This approach has some important limitations. First, it is very difficult to find a quartz reactor of such shape and size that it would well match the household MW oven. The quartz reactor then restricts that sample or batch size. Further, the maximum MW power tends to be around 800–1000 W. Finally, the electromagnetic wave pattern inside an MW cavity is typically a superposition of several standing waves or modes, as acknowledged in the terms “multi-mode MW cavity” or “multi-mode resonant cavity”. A single standing wave would give a highly inhomogeneous temperature distribution in the heated sample, while a multi-mode cavity can be designed to provide more uniform heating. In several recent studies the uniformity of temperature distribution has been pursued by using simulations during the design phase of experimental equipment [16,17].

To address these limitations of prior studies, we designed a MW cavity with waveguides from two microwave sources (magnetrons) using finite elements software to model and predict the electromagnetic fields. The expectation was that this would increase the achievable bio-mass temperatures, while adding magnetrons is the lowest cost alternative to increase microwave power. The two magnetrons used in this study were commercial grade. Another way to increase the bio-mass temperature is to increase the output power of the magnetron. The design is simpler than with two magnetrons, but the cost is higher and the options to pursue uniformity of heating remain limited: we decided in favor of two magnetrons. A one-liter commercial quartz beaker was targeted as the reactor, to be accommodated by the design. After construction of the equipment, 400 g samples of various OPS and AC mixtures were treated in the reactor. The temperature profiles during heating were monitored by type-K thermocouples, and product yields as well as bio-char and bio-oil quality were characterized. The quality parameters included chemical composition and heating value, and a SEM assessment was done for the biochar.

2. Materials and methods

2.1. Materials

In this study, the biomass used was oil palm shell (OPS) received from the Thaksin Palm Oil Mill located in Punpin, Suratthani province, Thailand. OPS samples were sent to the Scientific Equipment Center, Prince of Songkla University Hatyai Campus, for proximate analysis (Thermogravimetric Analyzer, TGA7, Perkin Elmer, USA) and for ultimate analysis (CHNS-O Analyzer, CE Instrument Flash EA 1112 Series, Thermo Quest, Italy), with results shown in Tables 1 and 2. Next, the OPS was ground roughly to 0.5 cm size and then dried in an oven at 140 °C for 30 min. Its moisture content after drying was around 8.5 wt%. Because biomass with low moisture content is a poor microwave absorber, commercial grade water treatment activated carbon (AC, based on coconut shell) was mixed in with the OPS [18–20]. The AC has high dielectric loss factor of about 0.35–0.83 [3] also reported as 0.22–2.95 [21]. Other advantages of AC, causing it to be selected for our experiments, include that it is a common material, inexpensive, easy to find, and easily available in various textures, sizes, and forms.

Table 1
Proximate analysis of oil palm shell (OPS).

Sample	Weight (%)			
	Moisture	Volatile Matter	Fix Carbon	Ash
Palm shell	11.393	65.760	19.730	3.117

Table 2
Ultimate analysis of the oil palm shell (OPS) material used.

Sample	Parameter	Instrument/method	Unit	Result ± SD
Palm shell	Nitrogen (N)	CHNS/O Analyzer	%	0.32 ± 0.01
	Carbon (C)	CHNS/O Analyzer	%	45.65 ± 0.26
	Hydrogen (H)	CHNS/O Analyzer	%	5.49 ± 0.06
	Sulphur (S)	CHNS/O Analyzer	%	Not Detected
	Oxygen (O)	CHNS/O Analyzer	%	36.59 ± 0.36

2.2. Experimental set-up

Mostly microwave induced pyrolysis has been studied with domestic microwave ovens, modified for the experiments. However, finding a quartz reactor that matches the dimensions of a domestic microwave oven is difficult, and the biomass sample sizes are severely limited. Thus, in the current study the microwave cavity was specifically designed to address these problems. It was designed to accommodate a quartz reactor with a cylindrical shape of diameter 10.7 cm, height 14.0 cm, and capacity 1.0 l. For a multimode standing wave the rectangular shape had inner dimensions 20 cm, 22 cm, and 32 cm height. The MW cavity was designed to have two microwave sources (magnetrons), and the interference of standing waves inside the cavity was used to improve MW power delivery. Thus, the two rectangular waveguides operated in TE_{01} mode, and each had the dimensions 8 cm, 10 cm, and 4 cm height. They were placed at opposite sides of the cavity with some asymmetry [22–24], as illustrated in the schematic diagram of the MW pyrolysis system in Fig. 1. These magnetrons taken from domestic microwave ovens operated at 2450 MHz frequency and were rated for 800 W each. They were connected to an MW power supply providing a pulsed electric signal, generated by a high voltage transformer to double voltage, a high voltage capacitor, and a high voltage diode that cuts the signal. The microwave power could be selected from 180, 300, 450, and 800 W, and the timer had 1–90 min range. The temperature of the mixture was measured by a type-K OMEGA thermocouple with a junction type ungrounded probe. It offers electrical isolation of 1.5M1/2 at 500 Vdc in all diameters, so it is suitable for a harsh microwave environment. Moreover, the thermocouple was grounded to avoid arcing when the microwave was operated. This thermocouple was connected to a temperature read-out and a data logger, providing 0.5 °C accuracy.

The governing equation for the electric field (E) is shown in Eq. (1), and this was numerically solved with the Finite Element Method (FEM) software COMSOL MultiPhysics^{TM}, using the features in RF Modules/Electromagnetic Waves/Harmonic Propagation. The simulation results for the selected design are shown in