



## RESEARCH PAPER

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## Characterization of bio-oil produced obtained fast pyrolysis of groundnuts shell

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### Abstract

This article reports a study of characterization of bio-oil produced from fast pyrolysis of groundnuts shell in a fluidized-bed reactor. The temperature of pyrolysis was varied in the ranges 400-500°C and using a biomass particle size of 250-425 µm. Results showed that the optimum pyrolysis temperatures for groundnuts shell was 475°C, which gave maximum bio-oil yields of 63.48 wt.% on dry biomass basis. The bio-oil products were also tested for their basic properties. Results showed that the water solids and ash contents of the bio-oil were 18.56 wt.%, 0.89 wt.% and 0.03 wt.%, respectively. Moreover, the density, pH value, low heating value (LHV) and viscosity measured to be 1,215 kg/m<sup>3</sup>, 4.20, 31.07 MJ/kg and 24.56 cSt, respectively.

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## Introduction

Biomass resources including wood and wood wastes, agricultural residues, municipal solid waste, animal wastes, waste from food processing, and aquatic plants and algae. It is a renewable resource, whose utilization has received great attention due to environmental considerations and the increasing demands of energy worldwide (Balat *et al.*, 2009; Tsai *et al.*, 2007). Biomass can be converted to energy via thermochemical conversion processes, such as direct combustion, pyrolysis and gasification (Pattiya and Suttibak, 2012). Fast pyrolysis or rapid pyrolysis is mostly applied to biomass so as to change its less energy-dense solid form into a liquid form, which is called "bio-oil". It is thermal decomposition occurring in the absence of oxygen. In fast pyrolysis, biomass decomposes very quickly to generate mostly vapours and aerosols and some charcoal and non-condensable gas. After cooling and condensation, a dark brown homogenous mobile liquid is formed which has a heating value about half that of conventional fuel oil. A high yield of liquid is obtained with most biomass feeds low in ash (Bridgwater, 2012). Many researchers studied the production of bio-oil from various types of biomass by many fast pyrolysis reactor configurations. The yield of bio-oil could be as high as 75 wt.% on dry biomass feed depending on the type of biomass and the reactor unit (Bridgwater, 2012). Bio-oil can be used as an alternative fuel in furnaces and engines in order to produce heat and/or power. In addition, bio-oil can be used as a raw material for chemical production.

Groundnuts or peanut is a species in the legume. It is oil crops same oil palm, soybeans, coconuts, sesame and castor beans. The main sources of groundnuts are in China, India, Nigeria, USA, Myanmar, Indonesia, Sudan, Vietnam and Thailand (FAO, 2013). Typically, groundnuts is grown for food production. Lately, groundnuts is also a feedstock for oil production. Residues from groundnuts is called "shell". The groundnuts shell is mostly burnt in the fields and are not efficiently used for energy. Only small part of the groundnuts shell is used as a compost and animal feed. By applying fast pyrolysis technology to

groundnuts shell for bio-oil production, the advantage that could be gained is not only on the fuel value aspect, but also on the environmental aspect. Abnisa *et al.* (2011) studied production of bio-oil via fast pyrolysis of palm shell in a fix-bed reactor and found that the bio-oil yield was 46.4 wt.% at 500°C and contains high water and oxygen. Therefore, the current paper objectives to close this gap by providing experimental results found when investigating the influence of pyrolysis temperature on the product yields and the properties of bio-oil produced from fast pyrolysis of groundnuts shell in a fluidized-bed reactor.

## Materials and methods

### *Biomass feedstock*

The raw biomass material used in this work is groundnuts shell from plantations in the north-east Thailand. The biomass sample was sundried, ground and sieved to a particle size range of 250-425 µm. Prior to experiments, the samples were dried in an oven at 105°C for 24 hours to reduce the moisture content to below 10 wt.%. The drying of the samples also eased the feeding step and could reduce the water content of the bio-oil product. Biomass sample were tested for their basic properties including proximate and ultimate analyses as well as heating value. The proximate analysis is to determine the moisture, volatile matter, fixed carbon and ash contents according to the ASTM standard methods (E1756-01, E872-82 and E1755-01). The ultimate analysis is to determine the content of carbon (C), hydrogen (H), nitrogen (N), sulfur (S) and oxygen (O). The ultimate analysis is conducted at the Center of Scientific and Technological Equipment (CSTE), Suranaree University of Technology, Nakhon Ratchasima, Thailand, using a Carbon Hydrogen Nitrogen & Sulfur analyzer Model Carbon 628, 628 S. All experiments were carried out in triplicate and the average values were reported. The heating values are calculated based on the ultimate analysis results and equations (1) and (2). The higher heating value (HHV<sub>dry</sub>) of biomass is calculated from a correlation developed by Sheng and Azevedo (2005) as shown by the following equation:

$$HHV_{dry} \left( \frac{MJ}{kg} \right) = -1.3675 + 0.3137C + 0.7009H + 0.0318O^* \quad (1)$$

where C, H are percentages on dry basis of carbon, hydrogen, respectively and  $O^*$  is  $100 - C - H - \text{Ash}$ .

The lower heating value ( $LHV_{dry}$ ) was calculated from  $HHV_{dry}$  and the hydrogen content by the following equation (ECN, 2013):

$$LHV_{dry} \left( \frac{MJ}{kg} \right) = HHV_{dry} - 2.442 \times 8.936 \left( \frac{H}{100} \right) \quad (2)$$

The biomass analysis results are summarized in Table 1.

#### *Thermogravimetric analysis (TGA)*

Biomass samples were subjected to a preliminary thermal decomposition analysis by thermogravimetric analysis (TGA) technique. Thermogravimetric analysis experiments were performed using a Mettler TGA, UMX5/XP5U analyzer. Approximately 21.2221 mg of each sample was heated in nitrogen atmosphere from room temperature to 1,000°C at a heating rate of 10°C/min and a run duration of 97.5 min. These experiments were carried out at the Center of Scientific and Technological Equipment (CSTE), Suranaree University of Technology, Nakhon Ratchasima, Thailand.

#### *Fast pyrolysis unit*

Fast pyrolysis of groundnuts shell was performed in a fluidized-bed reactor unit. The unit was composed of a pre-heater, a biomass hopper, a two-staged feeder, a fluidized-bed reactor, two cyclone separators, a hot filter and a bio-oil product collection unit (see Fig. 1). The reactor was made from a SUS 304 stainless-steel pipe, and its internal diameter and height were 45 and 450 mm, respectively. Silica sand with a particle size range of 250–425  $\mu\text{m}$ , was used as the fluidizing and heat transfer medium. The reactor and the pre-heater were heated by heating wires. Thermal insulation was accomplished with adiabatic material around the reactor and the immediate vicinity to minimize heat loss. The temperature of the

experimental system was adjusted using PID temperature controllers and was monitored using K-type thermocouples. The pyrolysis vapour produced was cleaned up in the cyclone separators and a hot filtration unit prior to condensation. Pyrolysis vapour was condensed into liquid product by a water-cooled heat exchanger and an electrostatic precipitator (ESP), a dry ice/acetone condensers and a cotton wool filter.

#### *Fast pyrolysis experiments*

Table 2 lists the different pyrolysis conditions for each experiment of. There were 5 levels of the setting temperatures including 400°C, 425°C, 450°C, 475°C and 500°C. The biomass particle size range was 250–425  $\mu\text{m}$ . The feed rate of each experiment was around 200–300 g/hr. Nitrogen was used as the fluidizing medium at a flow rate of 6 L/min. Silica sand rested on a distributor plate, with a nominal diameter of 250–425  $\mu\text{m}$ , acted as the fluidizing medium and heat carrier. The total time of each run was approximately 1 hour. To achieve the goal of these variables, a total of 5 experimental runs were performed.

#### *Mass balance calculation*

The main products from fast pyrolysis process are liquid bio-oil, solid char and non-condensable gases. The yields of each product were calculated by weighing all parts of the fast pyrolysis system such as biomass, silica sand, biomass hopper, fluidized-bed reactor, cyclone separators, hot filter and product collection unit, before and after each experiment. The bio-oil yields were the combined weight of the liquid from the product collection unit. The char yields were the combined weight of the solid from the reactor, the cyclones, the hot filter, transfer line and the solids in bio-oil. The gas yields were calculated by difference.

#### *Bio-oil analysis*

The bio-oil was characterized by measuring elemental composition, water, solids and ash contents, density, pH, viscosity and heating value. Each analysis was performed in triplicate.

The elemental composition of bio-oil is to determine the content of carbon, hydrogen, nitrogen, sulfur and oxygen. The elemental composition is conducted at the Center of Scientific and Technological Equipment (CSTE), Suranaree University of Technology, Nakhon Ratchasima, Thailand, using a Carbon Hydrogen Nitrogen & Sulfur analyzer Model Carbon 628, 628 S. The water content of bio-oil was determined by the Bioenergy Laboratory, Suranaree University of Technology, Nakhon Ratchasima, Thailand, using Karl-Fischer (KF) titration technique. The solids content of bio-oil was determined by vacuum filtration technique. The solids in bio-oil were defined as ethanol insoluble. About 3 g of bio-oil was dissolved in ethanol and filtered through a pre-dried and pre-weighed WINTTECH No. 1 qualitative filter paper with mean pore size of 6  $\mu\text{m}$ . The liquid was then washed with excess amount of ethanol until the filtrate was clear to ensure that there was no organic liquid left on the paper. The filter paper with the solids was air-dried for approximately 15 min and further dry in an oven at 105°C for 30 min. Then the paper was cooled in a desiccator and weighed. This method is suggested by Oasmaa and Peacocke (2010). The ash content of bio-oil is determined as the amount of residues when heating bio-oil to 775°C, 24 hr with oxygen supply. Direct heating of bio-oil would result in foaming and splashing due to their high water contents. Thus, the first controlled evaporation of water at 105°C is needed before rapid heating to 775°C (Oasmaa and Peacocke, 2010). The density of bio-oil is measured using a density bottle at room temperature. The pH value of bio-oil was measured with a pH meter (HANNA instruments) at room temperature. The bio-oil kinematic viscosity is measured at 40°C using a Cannon-Fenske Routine Viscometer with a SDM viscosity bath (Art. 370) according to ASTM D 445 and D446 standard methods. The higher heating value on dry basis ( $\text{HHV}_{\text{dry}}$ ) was calculated based on the elemental analysis results using a correlation developed by Channiwala and Parikh (2002) as shown by equation (3). The lower heating value on dry basis ( $\text{LHV}_{\text{dry}}$ ) was calculated from equation (2) (ECN, 2013). For heating values on wet basis ( $\text{HHV}_{\text{wet}}$  and  $\text{LHV}_{\text{wet}}$ ), equations

(4) and (5) were used ECN (2013) by taking into account of the water content in bio-oil ( $\text{H}_2\text{O}$ ).

$$\text{HHV}_{\text{dry}} \left( \frac{\text{MJ}}{\text{kg}} \right) = 0.3491C + 1.1783H + 0.1005S - 0.1034O - 0.0151N - 0.0211A \quad (3)$$

The C, H, S, O, N and A in equation (3) are percentages of carbon, hydrogen, sulfur, oxygen, nitrogen and ash in bio-oil on dry basis.

$$\text{HHV}_{\text{wet}} \left( \frac{\text{MJ}}{\text{kg}} \right) = \text{HHV}_{\text{dry}} \left( 1 - \frac{\text{H}_2\text{O}}{100} \right) \quad (4)$$

$$\text{LHV}_{\text{wet}} \left( \frac{\text{MJ}}{\text{kg}} \right) = \text{LHV}_{\text{dry}} \left( 1 - \frac{\text{H}_2\text{O}}{100} \right) - 2.442 \left( \frac{\text{H}_2\text{O}}{100} \right) \quad (5)$$

#### Char analysis

The solid char obtained from cyclone collectors from the experimental runs that gave highest bio-oil yields for each biomass feedstock. The char samples were tested for their elemental composition, ash content and heating value. The methods used for determining the elemental composition are the same as those used for biomass and bio-oil analysis. The procedure for ash content analysis was the same as those used for biomass. The char heating values were calculated according to the equations (2) and (3).

## Results and discussion

### Thermogravimetric analysis (TGA)

To determine the range of the pyrolysis temperature, thermogravimetric analysis (TGA) was carried out with groundnuts shell samples. Figs. 2 show the resultant TGA and DTG curves. The initial weight loss of both samples occurred at about 25-110°C due to water evaporation. The main weight loss was noticed at around 200-400°C, which is expected to be due to the decomposition of mainly hemicelluloses and cellulose. The final weight loss from 400-1,000°C was mainly due to the decomposition of lignin. The residues after slow pyrolysis at 1,000°C of groundnuts shell was around 42%.

### Influence of pyrolysis temperature on the product distribution

The influence of pyrolysis temperature on the product distribution is shown in Fig. 3. As can be seen from the graph, the groundnuts shell bio-oil yields reached their maxima at 475°C, which was 63.48 wt.% on dry

biomass basis. The char yield reduced with the temperature whereas the gas yield increased with temperature. These results were due to the greater primary decomposition of the biomass at higher temperature and/or secondary thermal decomposition of the char formed before being entrained out of the reaction zone (Pattiya and Suttibak, 2012). When pyrolysis temperature is too

high, a secondary cracking of pyrolysis vapour could take place, thus reducing the bio-oil yield and increasing the gas yield. The result is consistent with the result reported by Abnisa *et al.* (2011) in which the bio-oil yield of 46.4 wt.% on dry basis, was achieved for palm shell fast pyrolysis in a fix-bed reactor at 500°C.

**Table 1.** Characteristics of groundnuts shell.

Analysis	groundnuts shell	Palm shell <sup>a</sup>
<i>Proximate (wt.%, dry basis)</i>		
Volatile matter	70.46	74.59
Fixed carbon*	12.26	21.87
Ash	17.28	2.33
<i>Ultimate (wt.%, dry, ash-free basis)</i>		
Carbon (C)	44.25	57.24
Hydrogen (H)	6.92	6.12
Nitrogen (N)	3.76	0.09
Sulfur (S)	0.28	0.18
Oxygen* (O)	44.79	36.36
H/C molar ratio	1.88	1.28
O/C molar ratio	0.76	0.48
Molecular formula	CH <sub>1.88</sub> O <sub>0.76</sub>	CH <sub>1.28</sub> O <sub>0.48</sub>
<i>Heating value (MJ/kg, dry basis)</i>		
HHV	15.42	21.47
LHV	14.17	20.16

\*Calculated by difference.

<sup>a</sup>Abnisa *et al.* (2011)

**Table 2.** Pyrolysis conditions of groundnuts shell.

Parameters	Run				
	1	2	3	4	5
Pyrolysis temperature (°C)	400	425	450	475	500
Particle size (µm)	250-425				
Feed rate (g/hr)	200-300				
Nitrogen flow rate (l/min)	6				
Heat transfer medium	Silica sand				
Time (hr)	1				

#### Characterization of pyrolysis products

##### Bio-oil analysis

In this work, bio-oil samples were characterized by measuring elemental composition, water content,

solids content, ash content, density, pH value, viscosity and heating value. The bio-oil analysis results are summarized in Table 3.

**Table 3.** Properties of bio-oil obtained from fast pyrolysis of groundnuts shell.

Feedstock	Groundnuts shell	Rice Straw <sup>a</sup>	ASTM burner fuel standard <sup>b</sup>
Pyrolysis Temperature	475	403	-
Elemental composition (wt.%, dry basis)			
Carbon	71.46	64.0	N/A
Hydrogen	7.73	6.4	N/A
Nitrogen	6.57	0.3	N/A
Sulfur	1.35	N/A	N/A
Oxygen <sup>*</sup>	12.86	29.2	N/A
H/C molar ratio	1.30	1.20	N/A
O/C molar ratio	0.13	0.34	N/A
Molecular formula	CH <sub>1.30</sub> O <sub>0.13</sub>	CH <sub>1.20</sub> O <sub>0.34</sub>	N/A
Water content (wt.%)	18.56	31.23	30 max
Solids content (wt.%)	0.89	0.6	2.5 max
Ash content (wt.%)	0.03	0.01	0.25 max
Density (kg/m <sup>3</sup> )	1,215	1,100	1,100-1,300
pH value	4.20	3.2	Report
Kinematic viscosity @ 40°C (cSt.)			
Fresh bio-oil	24.56	8.3	125 max
Heating value by calculation method (MJ/kg)			
HHV (water-free basis)	32.76	26.8	N/A
HHV (as-produced basis)	26.68	18.5	15 min
LHV (water-free basis)	31.07	25.4	N/A
LHV (as-produced basis)	24.85	16.7	N/A
<sup>*</sup> Calculated by difference.			
<sup>a</sup> Pattiya and Suttibak (2012).			
<sup>b</sup> Oasmaa <i>et al.</i> (2009).			

**Table 4.** Properties of chars obtained from fast pyrolysis of groundnuts shell.

Feedstock	Groundnuts shell	Rice straw <sup>a</sup>
Elemental composition (wt.%, dry basis)		
Carbon	61.78	64.16
Hydrogen	3.56	3.50
Nitrogen	2.75	1.37
Sulfur	0.21	N/A
Oxygen <sup>*</sup>	8.93	14.50
Ash content (wt.%)	22.98	16.43
H/C molar ratio	0.69	0.65
O/C molar ratio	0.11	0.17
Molecular formula	CH <sub>0.69</sub> O <sub>0.11</sub>	CH <sub>0.65</sub> O <sub>0.17</sub>
Heating value (MJ/kg, dry basis)		
HHV	24.34	24.66
LHV	23.56	23.89

<sup>\*</sup>Calculated by difference.<sup>a</sup>Pattiya and Suttibak (2012).

Table 3 shows the elemental composition, molecular formula, H/C molar ratio and O/C molar ratio of groundnuts shell bio-oil. The carbon content of bio-oil was 71.46 wt.% on dry basis, which is relatively high compared to that of rice straw bio-oil (Pattiya and Suttibak, 2012). The H/C molar ratio and O/C molar ratio of bio-oil were 1.30 and 0.13, respectively. The water contents of groundnuts shell bio-oil was 18.56 wt.%, which met the ASTM burner fuel standard requirement (Oasmaa *et al.*, 2009). In contrast to the palm shell bio-oil (Abnisa *et al.*, 2011), the water content of groundnuts shell bio-oil was lower. These differences are possibly due to the different moisture content of biomass. The solids content of groundnuts shell bio-oil was 0.89 wt.%, which is in the acceptable range for burner fuel suggested by Oasmaa *et al.* (2009). The ash content of bio-oil was approximately 0.03 wt.%. This result is quite consistent with previously reported results (Pattiya and Suttibak, 2012). The density of bio-oil from pyrolysis groundnuts shell was around 1,215 kg/m<sup>3</sup>. This is a typical value of fast pyrolysis liquid. The pH value of the bio-oil products in this work was 4.2. When comparing the pH value of groundnuts shell bio-oil with palm shell bio-oil (Abnisa *et al.*, 2011) and rice straw bio-oil (Pattiya and Suttibak, 2012), it is apparent that the pH values of the bio-oils are quite similar. The viscosity of groundnuts shell bio-oil was around 24.56 cSt. Comparing the viscosity of groundnuts shell bio-oil with rice straw bio-oil (Pattiya and Suttibak, 2012), the viscosity of groundnuts shell bio-oil are higher than that of rice straw bio-oil because of the lower water content of the groundnuts shell bio-oil. This result is quite consistent with previously reported results (Oasmaa *et al.*, 2009). The lower heating value (LHV) of bio-oil was found to be 31.07 MJ/kg (water-free basis). When comparing the heating value of groundnuts shell bio-oil with rice straw bio-oil (Pattiya and Suttibak, 2012), it is apparent that the heating value of the groundnuts shell bio-oil are higher. These differences are possibly related to the carbon content and oxygen content of bio-oils.

### Char analysis

Table 4 shows the properties of the char obtained from fast pyrolysis of groundnuts shell. The elemental composition showed that the H/C molar ratio was around 0.69 and the O/C molar ratio was 0.11. The ash content of char obtained from fast pyrolysis of groundnuts shell was 22.98 wt.%. The char obtained in the experiment has higher heating value of around 24.37 MJ/kg. When comparing the elemental composition, ash contents and heating values of char derived from groundnuts shell with those from rice straw (Pattiya and Suttibak, 2012), it is evident that they are similar.

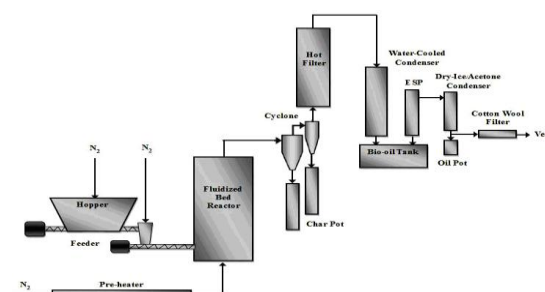


Fig. 1. Schematic diagram of the fast pyrolysis unit.

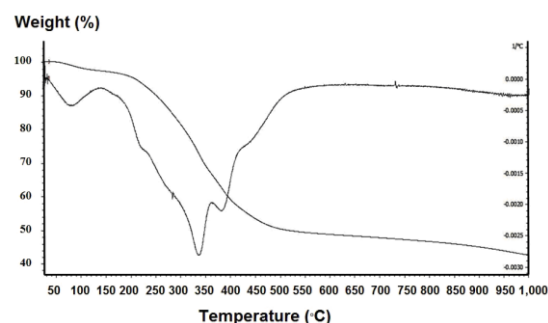


Fig. 2. TGA and DTG curves of groundnuts shell.

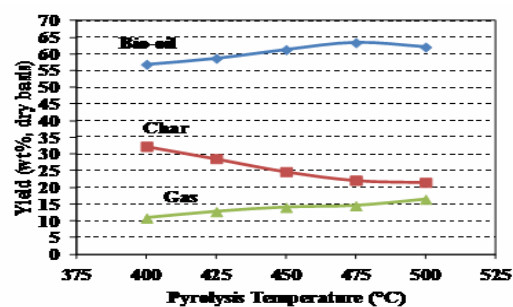


Fig. 3. Influence of temperature on product yields derived from fast pyrolysis of groundnuts shell.



## Conclusions

Fast pyrolysis of groundnuts shell was investigated using a bench-scale fluidized-bed reactor at different temperatures. The aim of this research was to investigate the influence of reaction temperatures and the properties of bio-oil produced. Results showed that the optimum pyrolysis temperature for groundnuts shell was 475°C, which gave maximum bio-oil yield of 63.48 wt.% on dry biomass basis. The bio-oil products were also tested for their basic properties. Results showed that the water solids and ash contents of the bio-oil were 18.56 wt.%, 0.89 wt.% and 0.03 wt.%, respectively. Moreover, the density, pH value, viscosity and low heating value (LHV) measured to be 1,215 kg/m<sup>3</sup>, 4.20, 31.07 MJ/kg and 24.56 cSt, respectively. The bio-oil obtained in experiments can be used as fuel; for example, as fuels in many static applications. It may either be used directly or in mixture with other conventional fuels.

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