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Influence of reaction temperature on yields of bio-oil from fast pyrolysis of sugarcane residues

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Abstract

This paper reports a study on production of pyrolysis oil from sugarcane residues, namely sugarcane bagasse (SB), sugarcane leaves (SL) and sugarcane tops (ST). This research investigated the effects of reaction temperature on pyrolysis products of these residues. Pyrolysis oil samples were characterized. Their elemental composition, density, pH value, viscosity and heating values as well as water, solids, ash contents were determined. It was found that the optimal reaction temperatures for pyrolysis of SB, SL and ST were 499°C, 403°C and 402°C, which gave maximal oil yields of 64.6, 53.4, and 52.2 wt.% on a dry biomass basis, respectively.

Keywords: Sugarcane residues, Pyrolysis oil, Fast pyrolysis, Fluidized-bed reactor

1. Introduction

Thermochemical technologies for converting biomass into energy or chemicals are primary direct combustion, pyrolysis, gasification and liquefaction [1-2]. Pyrolysis is promising technology for production of liquid fuels from biomass. Rapid or fast pyrolysis involves thermal degradation of biomass at intermediate temperatures, e.g., 400-500°C, under oxygen-free conditions [3]. A high yielding process for production of pyrolysis oil includes a medium pyrolysis temperature (~500°C), short hot vapour holding times (<2s), very fast heating rates of 10^3-10^5 °C/s, very high heat transfer coefficients, fast removal of char products and fast cooling of pyrolysis products [4]. Yields pyrolysis oil or bio-oil can be up to 80 wt.% on a dry biomass basis [5] depending on the biomass and reactor used. There are many fast pyrolysis reactors to for the production of bio-oil. These include fluidized-bed reactor, circulating fluidized bed, rotating cone pyrolyzer, vacuum pyrolysis, auger reactor, free-fall reactor, fixed-bed reactor and microwave reactor [6-7]. The fluidized-bed reactor is the most common reactor type for biomass pyrolysis. This reactor has very good heat/mass transfer and subsequently fast heating of biomass particles takes place. High pyrolysis oil yield (i.e. 75-80 wt.%) is typical [8]. Pyrolysis oil is of sufficient quality for use in furnaces and engines. Additionally, pyrolysis oil can be used as a feedstock for manufacture of other chemicals [9-11].

The primary sugarcane producing countries are Brazil, India, China, Thailand, Mexico and Australia. Typically, sugarcane is grown for production of sugar. Lately, sugarcane has been used a feedstock for production of ethanol. The demand for sugar and industrial ethanol is increasing. Therefore, it is expected that crop residues will increase accordingly. Residues from sugarcane processing include bagasse, leaves and tops. Sugarcane bagasse is traditionally used as a boiler fuel in sugar factories to generate electricity for their own use. The sugarcane leaves and tops are usually burned before harvest. A small portion sugarcane leaves and tops are used as a feed or to make compost. In 2014, Thai sugarcane production is currently about 103.7 million tonnes [12]. Crop to residue ratios of 0.291 and 0.302 have been reported for sugarcane bagasse (SB) and 0.302 for sugarcane leaves (SL)/tops (ST), respectively [13]. Using these ratios, approximately 61.5 million tonnes of sugarcane residues are produced annually. Using rapid pyrolysis to convert sugarcane bagasse, leaves and tops to pyrolysis oil could produce valuable fuel and lessen environmental impacts. The main product is liquid fuel (bio-oil), which is different from direct combustion (heat) and gasification (gas).

In this study, a fluidized-bed pyrolysis reactor was used to process sugarcane residues (bagasse, leaves and tops). Particular attention was given to the effect of reaction temperature on the product yields. Additionally, pyrolysis oil samples were analyzed to determine their basic physical and chemical properties.

2. Materials and methods

2.1 Biomass samples

The samples used in the current study were sugarcane residues. These included sugarcane bagasse (SB), sugarcane

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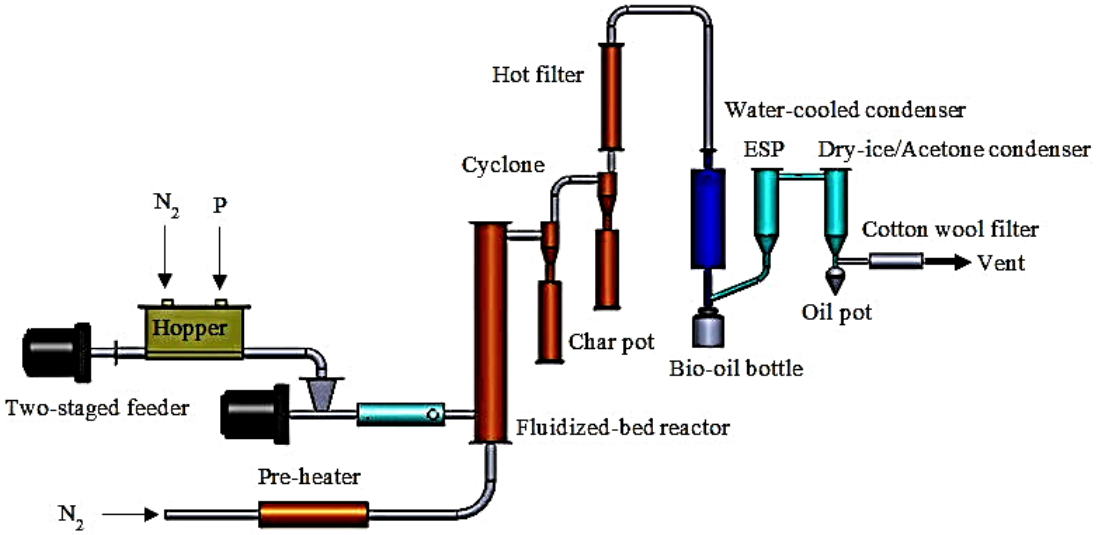

Figure 1 Schematic of the fast pyrolysis unit used in the current study.

leaves (SL) and sugarcane tops (ST). Sun dried residues were ground and passed through a sieve stack to select for particle sizes from 250-500 µm. Chemical analyses were done and the heating values of the samples were determined. Proximate analysis was used to determine the moisture, volatile matter and ash contents of the samples according to ASTM standard methods (E1756-01, E872-82 and E1755-01). Fixed carbon was determined as a difference. Ultimate analysis was used determine the carbon, hydrogen, nitrogen, sulfur and oxygen contents using a Carbon Hydrogen Nitrogen & Sulfur Analyzer. The higher heating value (HHV_{dry}) of biomass was calculated from equation (1) below [14]. The lower heating value (LHV_{dry}) was determined from equation (2) [15]. Each analysis was performed in triplicate.

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

where C and H are percentages of carbon and hydrogen, respectively, on a dry basis and O* is 100-C-H-Ash. HHV_{dry} is the higher heating value on a dry matter basis.

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

where LHV_{dry} is the lower heating value on dry matter basis.

2.2 Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was done in conjunction with differential thermal analysis (DTA). Concurrent measurements were made using TGA/DSC, SDT 2960 (V3.0F), and TA Instruments. Nine mg of each sample was heated under nitrogen from 25-800°C at a rate of 10°C/min.

2.3 Fast pyrolysis unit

As shown in Figure 1, the experimental device was constructed with a pre-heater, hopper, two screw-feeders, a fluidized-bed reactor, two cyclones, a hot filter and a bio-oil collection device. The pre-heater brought nitrogen to approximately 450°C prior to entering the reactor. The biomass hopper was used to contain the SB, SL and ST feedstocks. The screw-feeders were of the same configuration and size. The first controlled the feeding rate and the second prevented jamming of the feeding system. SUS 304 stainless-steel pipe was used to make the reactor. It was 45 mm in diameter and 450 mm in height. Silica sand with particle diameters ranging from 250–500 µm was used in the reactor. It served as a fluidizing and heat transfer medium. The fluidized-bed was heated by heating wires. PID controllers maintained temperature (SESTOS™ Model: D1S). K-type thermocouples were used as temperature sensors. The temperatures in the reactor were monitored using two thermocouples, and the pyrolysis temperatures were averaged. The errors in the average pyrolysis temperatures were within ±5°C. The resulting hot vapour was cleaned using two cyclones and the hot filter before condensation. The first and second cyclones were 30 and 20 mm in diameter, respectively. They were used to separate solid particles (char) from the hot gas. The temperatures in the cyclones and hot filter were maintained at 420°C. A liquid was condensed from the hot vapour using a heat exchanger, electrostatic precipitator (ESP), a dry ice/acetone condenser and a cotton wool filter. Figure 2 shows the fast pyrolysis equipment used in this study.

Figure 2 Fast pyrolysis equipment used in the current study.

Table 1 Characteristics of sugarcane residues

Analysis	Sugarcane residues		
	SB	SL	ST
Proximate (wt.%, dry basis)			
Moisture (wet basis)	4.45±0.03	4.12±0.04	3.88±0.08
Volatile matter	82.77±0.08	77.63±0.18	76.53±0.07
Fixed carbon*	10.62±0.09	14.67±0.12	15.16±0.10
Ash	6.41±0.01	7.53±0.06	8.16±0.04
Ultimate (wt.%, dry basis)			
Carbon	45.65±0.06	43.38±0.14	42.55±0.04
Hydrogen	7.10±0.03	4.29±0.04	4.10±0.04
Nitrogen	0.38±0.03	0.38±0.03	0.54±0.02
Sulfur	0.31±0.02	0.14±0.02	0.20±0.02
Oxygen*	40.82±0.10	44.78±0.10	45.17±0.10
H/C molar ratio	1.87±0.01	1.19±0.01	1.16±0.01
O/C molar ratio	0.66±0.00	0.77±0.01	0.78±0.00
Molecular formula	$CH_{1.87}O_{0.66}$	$CH_{1.19}O_{0.77}$	$CH_{1.16}O_{0.78}$
Heating value (MJ/kg, dry basis)			
HHV	19.23±0.04	16.67±0.06	16.29±0.03
LHV	17.68±0.03	15.74±0.05	15.40±0.02

*Determined by difference

2.4 Fast pyrolysis conditions

Fast pyrolysis was done using a fluidized-bed reactor with a feed rate of approximately 200–300 g/hr. The goal of this study was to elucidate the effects of pyrolysis temperature on process products. The types of biomass investigated were SB, SL and ST. Five temperatures were investigated. These were 350°C, 400°C, 450°C, 500°C and 550°C. The controlled variables included a hot nitrogen gas flow rate at 6 litres/min, a biomass feed rate of around 200-300 g/hr, temperature of the pre-heater, cyclones, hot filter and transfer line at 450°C, cooling water at room temperature, a dry-ice/acetone condenser at -75°C and a process duration of one hour. Fifteen trials were conducted to elucidate the impacts of these factors. The optimal reaction temperatures of SB, SL and ST will be considered from maximum obtained yield (pyrolysis oil).

2.5 Mass balance calculation

The fast pyrolysis products of sugarcane residues include pyrolysis oil, char and non-condensable gases. Yields were determined by measuring the masses all components of the reactor. This included feed samples, sand in the reactor, and the reactor itself, along with the cyclones, hot filter, condenser unit and bio-oil collection unit. This was done prior to and after each run. Pyrolysis oil yield was the mass of liquid from the bio-oil collection unit. The char yield was determined from the combined masses of the solids in the reactor, the cyclones and the hot filter. The gas yield was calculated as a difference.

2.6 Pyrolysis oil analysis

The properties of pyrolysis oil were determined from its elemental composition, water, solids and ash contents, as well as its density, pH, viscosity and heating values. Measurement of the elemental composition of bio-oil determined its carbon, hydrogen, nitrogen and oxygen content as described in Section 3.1. The water content of pyrolysis oil was determined using a Karl-Fischer Moisture Titrator (MKC-520). The solids content of bio-oil was measured by vacuum filtration. Pyrolysis oil solids were ethanol insoluble materials. Approximately 2 g of pyrolysis oil was dissolved in ethanol and filtered through pre-dried and pre-weighed Whatman No. 3 filter paper [16]. Bio-oil ash content was measured by determining the mass of residues formed when heating bio-oil samples to 775°C in the presence of oxygen. Directly heating of pyrolysis oil to 775°C resulted in foaming due to the high water content of the samples. Therefore, the samples were first heated to 105°C to gently evaporate water before fast heating to 775°C [16]. The density of pyrolysis oil was determined at room temperature using a density bottle [10]. A pH meter was used to measure the acidity of room temperature pyrolysis oil (AMT620 Lab PH meter). Its kinematic viscosity at 40°C was determined according to standard methods (ASTM D 445 and D446) with a ViscoClock Viscometer in an SI Analytics temperature bath (CT 72/P). HHV_{dry} was determined based on the elemental analysis according to equation (3) [17]. Equation 2 was used to predict LHV_{dry} [15]. HHV_{wet} and LHV_{wet} are given by equations (4) and (5) [15]. This accounts for the water (H_2O) in pyrolysis oil. Each analysis was carried out in triplicate.

$$HHV_{dry}\left(\frac{MJ}{kg}\right) = 0.3491C + 1.1738H + 0.1005S - 0.1034O - 0.0211A \quad (3)$$

C, H, S, O and A in equation (3) are percentages of carbon, hydrogen, sulfur, oxygen and ash, respectively, in pyrolysis oil on a dry basis.

$$HHV_{wet}\left(\frac{MJ}{kg}\right) = HHV_{dry}\left(1 - \frac{H_2O}{100}\right) \quad (4)$$

$$LHV_{wet}\left(\frac{MJ}{kg}\right) = LHV_{dry}\left(1 - \frac{H_2O}{100}\right) - 2.442\left(\frac{H_2O}{100}\right) \quad (5)$$

3. Results and discussion

3.1 Characteristics of sugarcane residues

The characteristics of sugarcane residues (SB, SL and ST) are given in Table 1. The volatile matter contents of SB, SL and ST were 82.77, 77.63, and 76.53 wt.% on a dry biomass basis, respectively. This showed that fast pyrolysis of SB resulted in a higher yield than for either SL or ST. This result is consistent with Pattiya et al. [18]. The calculated higher heating values (HHV_{dry}) of SB, SL and ST were 19.23 MJ/kg, 16.67 MJ/kg and 16.29 MJ/kg, respectively, on a dry biomass basis.

3.2 Thermogravimetric analysis

Thermogravimetric (TGA) and differential thermal analyses (DTA) of the SB, SL and ST were performed with a thermogravimetric analyzer under nitrogen with a 10°C/min heating rate. TGA and DTA curves of SB, SL and ST are shown in Figures 3 (a) and (b), respectively. The initial weight losses of the SB, SL and ST were due evaporation of water. Initial decomposition began at ambient temperature and finished with a shoulder at 220°C. The TGA curves show that about 76%, 72% and 70% by weight of the SB, SL and ST, respectively, were decomposed at a temperature below 500°C. DTA curves of the SB, SL and ST showed a maximum decomposition rate from 220–380°C, with peaks at 350°C, 332°C and 322°C, respectively. This result is consistent with Pattiya and Suttibak [10] and Jung et al. [19]. Usually, lignin pyrolysis happens at temperatures greater than 400°C [19-21]. Therefore, yields observed between 400 and 800°C are likely associated with lignin and protein decomposition. Sample comparison at 400°C on the TGA curve reveals that the weight of the SB was less than that of the SL and ST. This indicates that rapid pyrolysis of SB can produce a higher pyrolysis oil yield than the rapid pyrolysis of SL and ST.

3.3 Impacts of pyrolysis temperature on product formation

Figures 4, 5 and 6 show the influence of reaction temperatures on the yields of fast pyrolysis products of SB, SL and ST, respectively. The optimal pyrolysis temperatures for SB, SL and ST were 499°C, 403°C and 402°C, yielding maximal pyrolysis oil levels of 64.6, 53.4, and 52.2 wt.%, respectively, on a dry biomass basis. At increased temperatures, pyrolysis oil and char yields from SL and ST decreased with increasing gas yields. These results were due to secondary cracking of pyrolysis vapours at elevated temperatures [19].

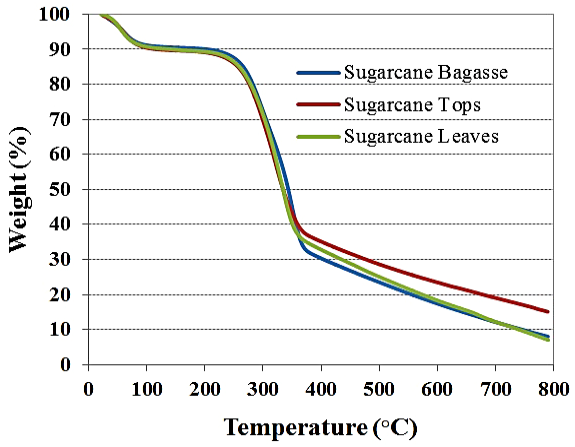

Figure 3 (a) TGA and (b) DTA curves for sugarcane residues.

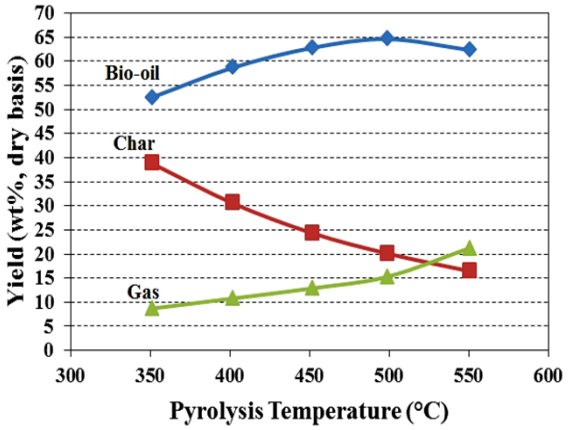

Figure 4 Impacts of reaction temperature on fast pyrolysis product yields of sugarcane bagasse (SB).

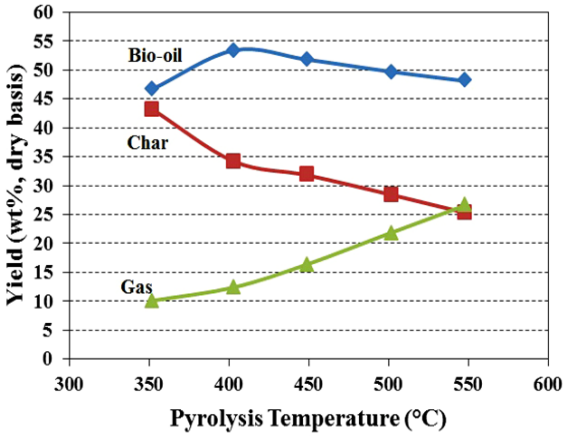

Figure 5 Impacts of reaction temperature on fast pyrolysis product yields of sugarcane leaves (SL).

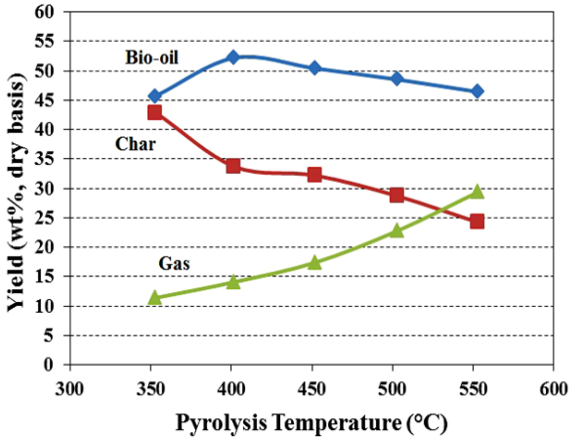

Figure 6 Impacts of reaction temperature on fast pyrolysis product yields of sugarcane tops (ST).

Table 2 Properties of oils from fast pyrolysis of sugarcane residues.

Analysis	Sugarcane residues			Typical*
	SB	SL	ST	
Pyrolysis temperature (°C)	499	403	402	N/A
Elemental composition (wt.%, dry basis)				
Carbon	68.79±1.58	68.32±1.81	65.81±0.96	55-58
Hydrogen	6.43±0.18	5.27±0.17	4.61±0.16	5.5-7.0
Nitrogen	0.75±0.06	0.71±0.05	0.66±0.04	0-0.4
Sulfur	0.27±0.03	0.22±0.03	0.22±0.03	0-0.05
Oxygen [a]	23.74±1.46	25.44±1.71	28.65±0.81	35-40
H/C molar ratio	1.12±0.06	0.93±0.05	0.84±0.04	N/A
O/C molar ratio	0.26±0.02	0.28±0.03	0.33±0.01	N/A
Molecular formula	$CH_{1.12}O_{0.26}$	$CH_{0.93}O_{0.28}$	$CH_{0.84}O_{0.33}$	N/A
Water content (wt.%)	30.03±1.58	33.56±1.75	32.67±1.09	20-30
Solids content (wt.%)	0.62±0.04	0.46±0.07	0.51±0.08	0.01-0.5
Ash (wt.%)	0.02±0.01	0.03±0.01	0.02±0.01	0.01-0.2
pH value	3.46±0.05	3.32±0.02	3.19±0.02	2-4
Density (g/ml)	1.12±0.01	1.11±0.01	1.11±0.01	1.10-1.30
Viscosity @ 40°C (cSt.)	18.97±1.19	13.45±0.68	10.26±1.77	15-35
Heating value (MJ/kg)				
HHV (dry basis)	29.15±0.49	27.44±0.62	25.48±0.23	N/A
HHV (wet basis)	20.39±0.12	18.22±0.07	17.16±0.13	16-19
LHV (dry basis)	27.75±0.53	26.29±0.65	24.47±0.26	N/A
LHV (wet basis)	18.68±0.10	16.64±0.07	15.68±0.12	13-18

[a] Determined by difference.
*Krutof & Hawboldt. [3]; Oasmaa et al. [24]; Balat et al. [25]

3.4 Comparison of product yields

Figure 7 shows a comparison of the product yields from fast pyrolysis of SB, SL and ST in a fluidized-bed reactor. These results showed that fast pyrolysis of SB gave a higher pyrolysis oil yield than SL and ST. The fast pyrolysis of SB gave a high bio-oil and low char yield due to the greater primary decomposition of the biomass at higher temperature or secondary thermal decomposition of the char formed before being entrained out of the pyrolysis zone. The high pyrolysis oil yield was predicted from both the proximate analysis and from the thermogravimetric analysis (TGA) experiments. The proximate analysis showed that the SB contained more volatile matter than the SL and ST. TGA showed fewer residues from SB breakdown than for SL and ST. This indicates that the SB pyrolysis can produce a higher bio-oil yield than the SL and ST pyrolysis. Comparing the bio-oil yields of SB, SL and ST pyrolysis oils with rice straw (50 wt.%) [22] and rice husks [23], the bio-oil yields of SB, SL and ST pyrolysis oils were higher than that of rice straw and rice husks pyrolysis oils because the volatile matter contents of SB, SL and ST were higher.

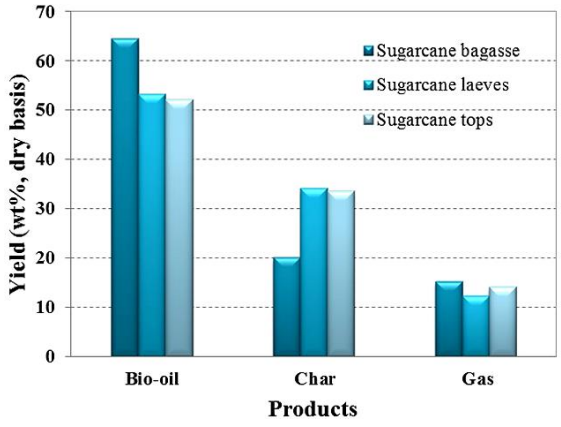

Figure 7 Comparison of product yields from fast pyrolysis of sugarcane residues.

3.5 Characterization of pyrolysis oil

In the current study, pyrolysis oil samples were characterized. This was done by determining their elemental composition, water, solids, and ash contents, as well as their density, pH value, viscosity and heating values. Table 2 lists some of the distinct features of the SB, SL and ST pyrolysis oils obtained at 499°C, 403°C and 402°C, respectively. The carbon contents of SB, SL and ST pyrolysis oils were 68.79, 68.32, and 65.81 wt.%, respectively, on a dry basis. The oxygen contents of SB, SL and ST pyrolysis oils were 23.74, 25.44, and 28.65 wt.% on a dry basis, respectively. The carbon content increased with the heating value whereas the oxygen content was reduced with the heating value. The water contents of SB, SL and ST pyrolysis oils ranged from 30.03-33.56 wt.%. In the current study, the water content of pyrolysis oil derived from SB, SL and ST was higher than the previous studies [3, 24-25]. The differences were possibly related to the oxygen contents of the various pyrolysis oil samples. The solids contents of SB, SL and ST pyrolysis oil were 0.62, 0.46 and 0.51 wt.% on a dry basis, respectively. This is within the acceptable range for use as fuel [24]. The ash contents of pyrolysis oil produced using SB, SL and ST were 0.02, 0.03, and 0.02 wt.%, respectively. Ash contents of oil produced from SB, SL and ST were quite similar to those of previous studies [24-25]. The densities of pyrolysis oil from fast pyrolysis of SB, SL and ST were 1.12, 1.11 and 1.11 g/ml, respectively. These are typical values for liquids obtained from fast pyrolysis. The pH values of the SB, SL and ST pyrolysis oils were in the range of 3.19-3.46, which is in agreement with Oasmaa et al. [24]. The viscosity of SB, SL and ST pyrolysis oils ranged from 10.26-18.97 cSt. These values were low due to elevated water content of the pyrolysis oils, but within the acceptable range for use as fuel [3, 24]. Comparing the kinematic viscosity of SB, SL and ST pyrolysis oils with rice husk [26] and wood pyrolysis oils [8], the kinematic viscosity of SB, SL and ST pyrolysis oils was lower than that of rice straw and wood pyrolysis oils because of the higher water content of the SB, SL and ST pyrolysis oils. The heating values of SB pyrolysis oil were higher than

for SL and ST pyrolysis oils, which was likely due to its higher carbon and lower oxygen contents.

4. Conclusions

Fast pyrolysis experiments were conducted on sugarcane residues, namely sugarcane bagasse (SB) sugarcane leaves (SL) and sugarcane tops (ST), in a pyrolysis reactor. The pyrolysis temperature was varied from 350-550°C. It was found that the optimal pyrolysis temperatures for SB, SL and ST were 499°C 403°C and 402°C, yielding maximal bio-oil levels of 64.6, 53.4, and 52.2 wt.% on a dry biomass basis, respectively. These results showed that fast pyrolysis of SB gave a higher pyrolysis oil yield than SL and ST. The liquid fuel products were also tested for their basic properties. Results showed that the heating values of SB pyrolysis oil were higher than for SL and ST pyrolysis oils, which was likely due to its higher carbon and lower oxygen contents.

5. Acknowledgements

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