



Laboratory Estimation of Bio-crude Production Potential from Selected Agricultural Residues

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Article Info

Manuscript received:
June, 2018

Revised manuscript accepted:
April, 2019

Keywords: Pyrolysis, bio-crude, calorific value, flash point, fire point

ABSTRACT

Pyrolysis is a better option for effective utilisation of surplus biomass and its utilization to liquid fuels. In this study, the technique of fast pyrolysis was adopted for the production of bio-crude from selected biomaterials viz. rice husk, coir pith, saw dust, cotton stalk, red gram stalk, groundnut shell and maize cob. Bio-crude production was done in a lab-scale fixed-bed pyrolytic system at six different temperatures (350°C, 400°C, 450°C, 500°C, 550°C and 600°C). Bio-crude yield was obtained in the range of 28 % to 52 %, and groundnut shell pyrolyzed at 550°C gave maximum percentage of bio-crude. The viscosity, pH, density, specific gravity, calorific value, flash point and fire point of bio-crudes were evaluated using standard methods.

Biomass is a promising renewable energy resource, and has attracted research interest in the development of new techniques for producing valuable chemicals and fuels. The total estimated biomass power potential in India is about 25,000 MW (MNRE, 2018). Surplus biomass available includes the residue from paddy such as paddy straw and rice husk (170 Mt.yr⁻¹), wheat stalks (112 Mt.yr⁻¹), cotton stalks and husk (53 Mt.yr⁻¹), maize stalks and cob (27 Mt.yr⁻¹), groundnut shell and stalks (15 Mt.yr⁻¹), coir pith (11 Mt.yr⁻¹) (Kumar *et al.*, 2015). The primary routes for biomass to power include combustion, gasification, pyrolysis and anaerobic digestion. The reactivity of biomass feedstocks in thermal conversion process depends not only on conditions of the thermal treatment, but also on the composition of the biomass feed.

Pyrolysis is the thermal treatment of biomass in the absence of oxygen, which results in the production of solid (charcoal), liquid (bio-crude) and gaseous fuel products. The liquid product may be stored, easily transported, upgraded to refined fuels, added to petroleum refinery feedstocks or contain chemicals in economically recoverable concentrations. It is a dark brown, free flowing organic liquid mixture, which generally comprises of a great amount of water (usually 15–35 % of weight) and hundreds of organic

compounds such as acids, alcohols, ketones, aldehydes, phenols, ethers, esters, sugars, furans, alkenes, nitrogen compounds and miscellaneous oxygenates (Rezaei *et al.*, 2014). Bio-crude fuels generate 50 % lower NO_x emissions than diesel oil in a gas turbine, and they are also CO₂/GHG neutral (Xiu and Shahbazi, 2012). The bio-crude oil can be used to produce high-value chemicals and bio-fuels, including both petrol and diesel replacement fuels.

In this study, bio-crude production was done through a fixed-bed reactor, and the abilities of selected agricultural residues for bio-crude production at different temperatures were studied.

MATERIALS AND METHODS

Experimental Procedure

A lab-scale pyrolysis experiment, bio-crude measurement and analysis of bio-crude properties were carried out in the Department of Bioenergy, Agricultural Engineering College and Research Institute, Tamil Nadu Agricultural University, Coimbatore. Raw biomass samples were used for the experimental study. The selected lignocellulosic materials included rice husk, coir pith, saw dust, cotton stalk, red gram stalk, groundnut shell and maize cob. These were selected

based on the availability and higher energy content. The experimental studies for assessing physical, chemical and biochemical properties of the biomass were carried out in triplicate.

Bio-crude production

Bio-crude production was carried out in a fixed-bed pyrolytic system (Fig. 1) available in the Department of Bioenergy, TNAU, Coimbatore. It consisted of a fixed-bed reactor, muffle furnace for external heating, temperature controller, and a condenser. The fixed-bed reactor was made of mild steel, and its diameter and height were 50 mm and 65 mm, respectively. The outer shell of the condenser (350 mm x 100 mm) was made of G.I. sheet, and the inner tube (20 mm) was made of copper. A mixture of water and ice (50:50) was used as condensing medium in the outer shell for cooling volatile vapour released from a biomass. The operating time for each experimental run was one hour.

Six different temperatures (350°C, 400°C, 450°C, 500°C, 550°C and 600°C) were used to study the variations in bio-crude production. The temperatures between 400°C and 600°C were suggested as optimum for bio-crude production from the common biomass (Gang *et al.*, 2007; Zhang *et al.*, 2009). Thirty grams of a selected biomass was placed in the fixed-bed reactor, and the temperature was set to the desired value with heating rate of 10°C.min⁻¹. The volatile vapour from the reactor was cooled in the shell and tube condenser, and bio-crude was collected in a conical flask. The quantity of bio-crude and char collected were measured using measuring cylinder and weighing balance, respectively. The experimental setup of bio-crude production is shown in Fig. 2. The optimum temperature of bio-crude production was observed by considering the maximum bio-crude of each biomass at specific process temperature.

Characterisation of Samples

The selected lignocellulosic materials were characterised for the content of moisture, volatile matter, ash content and fixed carbon. Biochemical analysis was also carried out, which included cellulose, hemicellulose and lignin content of biomass. The procedures followed for the analysis are given below.

Moisture content

Using a hot air oven (M/S ESCO India, Model: OFA-110-8, Max. Temp: 300°C, Volume: 110 L, Serial: 2017-T03231), moisture content of a biomass was determined (ASTM, 2006). About 5g of powdered sample of biomass was taken in a petri-dish and kept inside the oven with a set temperature of 105°C for 24 h till a constant weight was recorded.

Moisture content (w.b.) was calculated by the formula:

$$\text{Moisture content, \%} = \frac{\text{loss in weight of biomass, g}}{\text{initial weight of biomass, g}} \times 100 \quad \dots (1)$$

Volatile matter

Volatile matter of a dried biomass was determined using a muffle furnace (M/S, Bionics Scientific Technology, Temp. Range, 200-1400°C, Accuracy: +/- 1°C) (ASTM, 2006). To measure the volatile content, 3g of sample was taken in a closed crucible and kept inside the muffle furnace at 650°C for 6 min, and at 750°C for another 6 min. The loss in weight of the sample was determined and the percent of volatile matter was calculated as:

$$\text{Volatile matter, \%} = \frac{\text{loss in weight of biomass, g}}{\text{weight of moisture free biomass, g}} \times 100 \quad \dots (2)$$

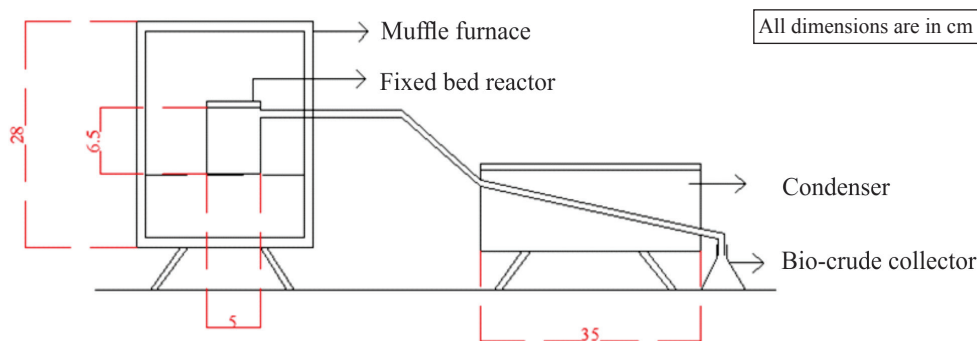


Fig. 1: Laboratory scale Bio-crude production system



Fig. 2: Pictorial view of laboratory scale bio-crude production system

Ash content

Ash content of selected biomass was found out as per ASTM (2013) procedure by taking 3g of sample in an open crucible and keeping it in a muffle furnace at about 750°C for 2-3 h. The ratio between the remaining weight of material left in the crucible and the initial sample taken was the fraction of ash content of tested material.

$$\text{Ash content, \%} = \frac{\text{weight of ash formed, g}}{\text{weight of initial dried biomass, g}} \times 100 \quad \dots(3)$$

Fixed carbon

Fixed carbon of a dried biomass was calculated using the formula (ASTM, 2009):

$$\text{Fixed carbon, \%} = 100 - [\text{volatile content (\%)} + \text{ash content (\%)}] \quad \dots(4)$$

Hemicellulose

Hemicellulose contents of the selected feedstocks were determined by NREL procedure (Sluiter *et al.*, 2004). One gram of a dried sample was taken in a test tube, and 10 ml of 0.5 M NaOH was added. The test tube was kept in a water bath at 80°C for 3 h, and cooled to room temperature. Sample solution was filtered through weighted filter paper (Whatman No. 42) until the colour disappeared. The hemicellulose content was calculated by keeping the filter paper with biomass solids at a temperature of 105±3°C in a hot air oven until reaching a constant weight.

Hemicellulose content was calculated as:

$$\text{Hemicellulose, \%} = [\text{Initial weight (g)} - \text{Final weight (g)}] \times 100 \quad \dots(5)$$

Lignin content

Acid soluble lignin and acid insoluble lignin content was determined based on the NREL procedure (Sluiter *et al.*, 2004). About 300 mg of dried sample was taken in a duran bottle, and 3 ml of 72 % sulphuric acid was added and mixed thoroughly. The sample was incubated for 60 min by stirring every 5 min for complete mixing during incubation. The sample was mixed by inverting the tube several times to eliminate phase separation between high and low concentration acid layers. The sealed samples were autoclaved for 1 h at 121°C. After completion of the autoclave cycle, hydrolysates were cooled slowly to room temperature before removing the caps. The autoclaved hydrolysis solution was filtered through a weighted filter paper, and collected in a filtering flask.

The filtrate sample was neutralized by adding calcium carbonate to determine the acid soluble lignin. Acid soluble lignin determination was carried out by using an UV-VIS spectrophotometer (M/S, Labtronics India, Model: LT 29, wavelength range: 190-1100 nm, photometric range: (-) 0.3 to 3.5 A, 0 to 220 % T, wavelength accuracy: (+) 0.3 nm) within 6 h of hydrolysis. Distilled water was used to transfer all remaining solids out of the duran bottle and was rinsed with distilled water until the colour disappeared. Hot distilled water was used to decrease the filtration time. The filter paper and acid insoluble residue was kept at 105 ± 3°C in hot air oven until a constant weight was reached.

Total lignin from acid soluble lignin (ASL) and acid insoluble lignin (AIL) was determined as:

$$\text{Lignin, \%} = \text{AIL(\%)} + \text{ASL(\%)} \quad \dots(6)$$

Cellulose content

Cellulose content of a biomass was calculated by subtracting the hemicellulose and lignin contents from 100.

$$\text{Cellulose, \%} = 100 - [\text{Hemi cellulose (\%)} + \text{Lignin(\%)}] \quad \dots(7)$$

Viscosity

Viscosity was determined using a Redwood viscometer (Model: LVDVE, Serial no: 8499218) (ASTM, 445-72). The viscometer bath was heated to 5°C above the testing temperature of 40°C. The bio-crude was filtered in B 200 mesh, and poured into the oil cup of the viscometer. The bio-crude was stirred continuously to make it homogeneous. A 50 ml measuring cylinder was kept below the jet of the viscometer. The time taken (t, sec) for filling of 50 ml oil sample after lifting the ball valve in oil cup of viscometer was recorded.

Viscosity of bio-crude was calculated using the following formula:

$$\begin{aligned} \text{Viscosity of bio-crude,} \\ cSt = 0.26 * t - 179/t \quad \text{For } 34 < t < 100 \text{ s} \\ cSt = 0.24 * t - 179/t \quad \text{For } t > 100 \text{ s} \end{aligned} \quad \dots(8)$$

Flash and Fire point

The flash and fire point of a sample were determined using Pensky Marten apparatus (M/S, Petrotest, Germany, Model: PMA-5, Application range: up to 450°C, Resolution: 0.1°C) (ASTM, 2018a). The bio-crude was filled up to the mark on the oil cup. The fuel was heated by a closed heating coil kept under water bath. A test flame was introduced in the fuel vapour at every 1°C rise of temperature. The temperatures observed during the flash and fire of bio-crude are the flash and fire point of the sample.

Density

Density of a bio-crude was determined using a digital density meter (Model: DDM 2910, Range: 0 to 3 g.cm⁻³, Accuracy: 0.0001 g.cm⁻³, measurement time: 30 to 60 s) following ASTM, 2018. The measurement was done by inserting the probe into a beaker containing bio-crude, and recording the result appearing on the screen.

Calorific value

Calorific value of a sample was determined using a

bomb calorimeter (M/s Aditya, India) following the procedure used by Jain and Jain (2016).

A known mass (1g) of a sample was taken in a clean crucible. The crucible was supported over the ring of the bomb of the calorimeter. A fine magnesium wire, touching the fuel sample, was stretched across the electrodes. The lid of the bomb was tightly screwed, and filled with oxygen to 25 atm. The bomb was lowered into the copper calorimeter, containing a known mass (1 litre) of water. The stirrer was operated and initial temperature of the water was noted. The electrodes were then connected to 6 V battery and circuit completed. The sample was burnt and hence heat was liberated. Uniform stirring of water was continued and the maximum temperature attained was recorded. The calorific value (CV) of the fuel was calculated by the formula given below.

$$\text{CV, cal g}^{-1} = \frac{(W+w)(t_2-t_1)}{x} \quad \dots(9)$$

Where,

- W = Mass of water in calorimeter, g,
- w = Water equivalent in g of calorimeter, stirrer, thermometer, bomb, Cal,
- t₁ = Initial temperature of water in calorimeter, °C,
- t₂ = Final temperature of water in calorimeter, °C, and
- x = Mass of fuel sample taken in crucible, g.

Specific gravity

Specific gravity of a bio-crude was determined using a pycnometer (ASTM, C 188) of a glass beaker of defined volume of 25 ml (M/S, Isolab GmbH). It was weighed without sample (w₁), and subsequently weighed by filling with water (w₂), and then filled with a bio-crude sample and weighed again (w₃). Specific gravity was calculated as:

$$\text{Specific gravity of bio-crude} = \frac{(w_3 - w_1)}{(w_2 - w_1)} \quad \dots(10)$$

Where,

- w₁ = Weight of empty specific gravity bottle, g,
- w₂ = Weight of water + specific gravity bottle, g, and
- w₃ = Weight of test sample + specific gravity bottle, g.

Statistical Analysis

Statistical Package of Social Sciences (SPSS 16.0) was used to find the effect of moisture content, volatile matter, cellulose, hemicellulose on bio-crude yield. Linear regression analysis was done with bio-crude yield as dependent variable. The independent variables included fixed carbon, volatiles, cellulose, hemicellulose, lignin and temperature.

RESULTS AND DISCUSSION

The observations made in the study for the optimization of bio-crude yield, and its analysis of properties as density, pH, viscosity, specific gravity, calorific value, flash and fire point are presented in this section.

Proximate Analysis

Volatile matter, fixed carbon, ash and moisture content are important components focussing on the characterization of fuel materials. Biomass residues have specific amount of moisture that directly affect their heating values (Singh *et al.*, 2013). Volatile matter, a key component of solid fuels, plays an important role in bio-crude production. The results of proximate composition of the selected feedstocks are given in Table 1.

Table 1. Proximate composition of selected feedstocks

Sl. No.	Biomass	Volatile matter, %	Ash content, %	Fixed Carbon, %
1.	Coir pith	75.90	6.70	17.40
2.	Rice husk	70.25	14.57	15.18
3.	Saw dust	76.68	7.51	14.82
4.	Cotton stalk	75.40	7.30	15.30
5.	Red gram stalk	73.20	7.94	15.86
6.	Groundnut shell	76.90	6.20	14.20
7.	Maize cob	76.60	4.10	16.30

Proximate analysis of the selected agricultural residues showed that the volatile matter ranged between 70.25 % and 76.90 per cent. Ash and fixed carbon contents were in the range of 4.10 % to 14.57% and 14.20 % to 17.40 %, respectively. The composition of cotton stalk was similar to the results reported by Jenkins and Ebeling (1985). The values obtained for coir pith, rice husk, red gram stalk and saw dust were similar to the results reported by Mythili *et al.* (2013). From Table 1 and Table 3, it was observed that the biomass with higher volatile content and lower ash content resulted in maximum bio-crude production.

Biochemical Analysis

The structural properties of cellulose, hemicellulose and lignin have been reported to influence the pyrolysis characteristics (Raveendran *et al.*, 1996). Biochemical compositions of the selected feedstocks are given in Table 2.

Table 2. Biochemical analysis of selected feedstocks

Sl. No.	Biomass	Cellulose, %	Hemi-cellulose, %	Lignin, %
1.	Coir pith	34.0	25.8	28.3
2.	Rice husk	36.5	22.9	13.1
3.	Saw dust	27.5	23.0	14.3
4.	Cotton stalk	25.9	12.0	20.0
5.	Red gram stalk	31.6	19.2	19.8
6.	Groundnut shell	46.2	20.1	20.3
7.	Maize cob	35.0	32.0	19.0

Cellulose and hemicellulose contents of the feedstocks were in the range of 25.9 % to 46.2 % and 12.0 % to 32.0 %, respectively; and lignin contents were in the range of 13.1 % to 28.3 per cent. These results were similar to the feedstock contents reported by Xie *et al.* (2013) for maize cob and groundnut shell, and Mythili *et al.* (2013) for coir pith, rice husk, saw dust, cotton stalk and red gram stalk. From Table 2 and Table 3, it was observed that the feedstocks with higher cellulose and hemicellulose contents resulted in maximum bio-crude production.

Bio-crude Production

Bio-crude productions from the selected biomaterials were carried out in the fixed-bed reactor set-up at six different temperatures (350°C, 400°C, 450°C, 500°C, 550°C and 600°C). The yields of bio-crude at six different temperatures are reported in Table 3. It was observed that bio-crude yield ranged from 28.3 % to 52.5 per cent. Groundnut shell yielded maximum bio-crude (52.1-52.5 %) at temperatures of 500°C and 550°C. Higher bio-crude yields were obtained for the feedstocks with more cellulose and hemicellulose contents compared to the other selected feedstocks. Bio-crude yield from red gram stalk was 29.7 % at 550°C. At temperatures of 500°C and 550°C, bio-crude yield of rice husk, maize cob, cotton stalk and coir pith were 39.4 (%)_w, 38 (%)_w, 28.7 (%)_w and 28.3 (%)_w, respectively. Bio-residues with lower cellulose and hemicellulose content and high lignin content produced lesser bio-crude. This might be due to maximized char production for the feedstocks with higher lignin content.

Table 3. Bio-crude production at different temperatures

Sl. No.	Biomass	350°C		400°C		450°C		500°C		550°C		600°C	
		Bio crude, %	Char, %	Bio crude, %	Char, %	Bio crude, %	Char, %	Bio crude, %	Char, %	Bio crude, %	Char, %	Bio crude, %	Char, %
1.	Rice husk	32.5	54.5	34.7	51.6	39.4	50.5	39.4	47.4	39.4	44.7	39.4	44.5
2.	Coir pith	22.0	46.0	23.6	45.0	28.0	38.4	28.3	38.8	28.3	38.0	28.3	37.7
3.	Saw dust	36.3	33.8	40.0	30.7	45.0	26.1	45.5	25.1	45.5	24.8	45.5	24.7
4.	Cotton stalk	20.0	49.2	21.0	47.0	25.0	44.1	28.7	43.6	28.7	42.9	28.7	42.3
5.	Red gram stalk	21.7	52.4	24.0	52.0	27.3	48.6	29.0	46.6	29.7	45.2	29.7	44.1
6.	Groundnut shell	32.5	40.3	35.1	30.9	50.1	29.3	52.1	26.8	52.5	27.2	52.5	25.6
7.	Maize cob	33.8	32.1	35.7	31.5	37.0	29.6	38.0	28.8	38.0	29.1	38.0	29.1

The optimized temperatures for the selected feedstocks are given in Table 4. The optimized temperature for better production of bio-crude ranged from 450°C to 550°C. The reduction in bio-crude production above 550°C indicated that the devolatilization of biomass was completed before 550°C. Higher pyrolysis temperature decreased the yield of char, and increased the yield of gaseous products (Zanzi *et al.*, 1996).

Bio-crude yield

Maximum bio-crude yield was obtained with the bio-materials containing higher volatile matter. Groundnut shell with highest volatile content (76.90 %) among the selected feedstocks yielded maximum (52.5 %) bio-crude. Biomass pyrolysis production process is divided into four individual stages: moisture evolution, hemicellulose decomposition, cellulose decomposition, and lignin decomposition. Higher cellulose and hemicellulose content leads to the formation of more bio-crude, while lignin favours char formation. Yang *et al.* (2007) also stated that cellulose decomposition occurred at 315°C to 400°C, and lignin decomposition went up to 900°C. From Table 1 and Table 3, it was observed that the presence of cellulose (46.2 %) and

hemicellulose (20.1 %) in groundnut shell favoured higher (52.5 %) bio-crude yield. It might be due to the presence of higher cellulose and hemicellulose content in biomass that led to the formation of more levoglucosan, which was the major constituent of bio-crude resulting in higher bio-crude yield.

Linear regression analysis was carried out using SPSS 16.0 software. Regression equation, considering the data of all biomasses under study, for the effect of cellulose (x_1), hemicelluloses (x_2), lignin (x_3) on bio-crude yield (y) was given as:

$$y = 0.673 x_1 + 0.195 x_2 + 0.151 x_3 + 7.459 \quad \dots(11)$$

The statistical analysis also indicated that the presence of higher cellulose and hemicellulose content favoured more bio-crude production.

An overall linear regression analysis was carried out with the characteristics of biomass, temperature and bio-crude yield. Bio-crude yield was taken as dependent variable and the independent variables included volatiles (x_1), fixed carbon (x_2), cellulose (x_3), hemicelluloses (x_4), lignin (x_5) and process temperature (x_6). The reliability of the model was 0.906. From the analysis, it was inferred that volatiles, fixed carbon, cellulose, hemicellulose and temperature had positive effects on bio-crude yield, while lignin had negative impact. Fixed carbon, hemicellulose and lignin had significance of 1 %, 1 %, and 10 %, respectively. The regression equation is represented as:

$$y = 1.742 x_1 + 4.702 x_2 + 1.381 x_3 + 0.532 x_4 - 0.283 x_5 + 0.038 x_6 - 224.21 \quad \dots(12)$$

Characterisation of Bio-crude

The analysis of bio-crude properties is important as

Table 4. Optimized temperatures for selected feedstocks

Sl. No.	Biomass	Temperatures
1.	Coir pith	500, 550
2.	Rice husk	450, 500, 550
3.	Saw dust	500, 550
4.	Cotton stalk	500, 550
5.	Red gram stalk	550
6.	Groundnut shell	550
7.	Maize cob	500, 550

Table 5. Characteristics of bio-crude

Sl. No.	Biomass	Density, kg.m ⁻³	pH	Viscosity, cSt	Specific gravity	CV, MJ.kg ⁻¹	Flash point, °C	Fire point, °C
1.	Rice husk	1140	2.8	11.52	1.53	14.9	102	105
2.	Coir pith	1067	2.9	23.52	1.84	13.7	109	114
3.	Saw dust	1063	2.8	5.74	1.41	14.8	97	102
4.	Cotton stalk	1160	3.3	10.57	1.72	16.8	108	115
5.	Red gram stalk	1260	2.6	6.87	1.60	14.3	106	110
6.	Groundnut shell	1026	2.7	5.62	1.39	14.2	101	106
7.	Maize cob	1144	2.8	5.37	1.18	18.8	94	99

the quality of any crude depends on its physical and chemical properties. The suitability of the crude for a specific use can be identified from its fuel properties. The properties of bio-crude including density, pH, viscosity, specific gravity, calorific value, flash point and fire point are reported in Table 5.

From Table 5, it was observed that the density of bio-crude was in the range of 1026 kg.m⁻³ to 1260 kg.m⁻³. As the density increased, the bio-crude energy content also increased. Bio-crude obtained from red gram stalk had highest density of 1260 kg.m⁻³ with energy content of 14.3 MJ.kg⁻¹ compared to other selected biomaterials. The pH value of the obtained bio-crude ranged between 2.6 and 3.3. The pH of cotton stalk bio-crude (3.3) was found to be more acidic than other bio-crudes.

Viscosity is an important property of bio-oil, which determines the flow quality or fluidity of the liquid. It has a major role on the design and manufacturing of engines where a liquid fuel is used (Bardalai and Mahanta, 2015). Viscosity of the bio-crudes ranged from 5.62 cSt to 23.52 cSt. Higher viscosity was found in coir pith bio-crude (23.52 cSt), and bio-crude obtained from groundnut shell had lower viscosity of 5.62 cSt.

Heating value of the bio-crudes ranged between 13.7 MJ.kg⁻¹ and 18.8 MJ.kg⁻¹, which was about half of conventional petroleum fuels (40 MJ.kg⁻¹ to 50 MJ.kg⁻¹). It might be due to the presence of water and higher oxygen content in the bio-crudes. Heating value of maize cob bio-crude (18.8 MJ.kg⁻¹) was higher than the other selected feed stocks. Flash and fire point of the bio-crudes were in the range of 94°C to 109°C, and 99°C to 115°C, respectively. Specific gravity of the obtained bio-crudes was in the range of 1.18 to 1.84.

CONCLUSIONS

Bio-crude production from selected biomass feedstocks through pyrolysis was assessed in fixed-bed reactor using muffle furnace. The study showed that bio-crude production through pyrolysis is an alternative method of energy generation from biomass. The temperature range for bio-crude production from selected biomaterials was 450°C to 550°C, and groundnut shell had highest bio-crude yield of 52.5 per cent. The range of bio-crude varied between 28.3 % and 52.5 percent. The heating values of bio-crudes ranged between 13.7MJ.kg⁻¹ and 18.8 MJ.kg⁻¹. Flash and fire point of bio-crudes were in the range of 94°C to 109°C, and 99°C to 115°C, respectively. After upgradation, bio-crude obtained from this process can be used as a substitute for furnace oil in industries.

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