

Bio-Crude-Oil from Fluidized Bed Pyrolysis of Rice Straw and its Characterization

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ABSTRACT

Rice is grown abundantly in all of the south Asian countries. About 130 wt% of net rice production is generated as biomass solid waste in the form of rice straw. The biomass solid waste has been characterized to investigate its suitability as feedstock for thermochemical conversion. Considering this availability of rice straw, its favorable volatile content, and elemental and thermal characteristics, an attempt has been made to convert the solid waste into bio-oil. The solid rice straw in particle form was pyrolysed in an externally heated 1443 cm³ volume fluidized bed reactor with nitrogen as the fluidizing gas and silica sand as the bed material. A renewable energy biomass source cylindrical heater and a gravity feed type reactor feeder were used to heat and to feed the reactor, respectively. The pyrolytic products were oil, char and gas. The product yields were found to be significantly influenced by the process conditions. The physical properties, calorific value, elemental (CHNOS) analysis and chemical composition using Fourier Transform InfraRed spectroscopy (FTIR) of the product oil were determined. The oil obtained at maximum liquid yield condition was considered for these analyses. The optimum reaction condition was at 450°C reactor bed temperature and 31 l/min fluidizing gas flow rate. The results of the analyses showed that the oil was highly oxygenated, acidic in nature with moderate heat value and favorable pour and flash points.

1. INTRODUCTION

Energy is a strategic input necessary for socio-economic development. It helps in meeting basic human needs. Actually, energy propels the society. The standard of living is correlated with per capita energy consumption. The more the energy is consumed the higher the standard of living is considered. By the year 2100, the world population is expected to be more than 12 billion and it is estimated that the demand for energy would increase by five times the current demand [1]. Thus, the demand of energy will continue to outstrip its supply. As a result engineers, scientists and technologists are searching for new, alternative and promising source of energy. Rice straw represents an important source of alternative potential energy, fuel and value-added chemicals, which is abundantly available in south Asia, particularly in Bangladesh.

Bangladesh is an agricultural country where rice is the main food crop. Rice is grown on about 80% of cultivable land in the country. The country's topography (flat land), temperature (16°C to 30°C) and the amount of rainfall (100 to 200 cm) are very suitable for rice production. Rice grows two times a year and rice straw is the major residue of rice production. The quantity of rice straw is 130% of rice production [2]. Thus, huge amount of rice straw is obtained every year in the country. This residue is traditionally used as fuel for cooking and cattle feed. Large amount is unused and wasted, creating disposal problem [3].

Pyrolysis is the thermal degradation either in complete absence of oxidizing agent, or with such a limited supply that gasification does not occur to an appreciable extent. According to Bridgwater and

Bridge [4], pyrolysis currently seems to be the most promising thermochemical conversion technology for the production of pyrolysis liquid oil. The liquid is of moderate heating value, is easily transported, can be burnt directly in the thermal power plant, can possibly be injected into the flow of a conventional petroleum refinery, burnt in a gas turbine or upgraded to obtain light hydrocarbons for transport fuel [5]. The solid char can be used to make activated carbon. Besides the char has its potential to be used as fuel. The gas has high calorific value, sufficient to be used for the total energy requirements of the pyrolysis plant [6]. According to Soltes [7], both the product yield and chemical composition of pyrolysis oil can be varied according to the process conditions. Previously [6, 8] some works have been carried out in the laboratory of Universiti Teknologi Malaysia to derive liquid oil from fluidized bed pyrolysis of oil palm shell and rice-husk using electrical energy as source of heat in a 5 cm diameter and 30 cm high stainless steel reactor with a motor driven screw feeder. Recently, bio-crude-oil has been obtained by fluidized bed technology with rice straw as the feedstock at the Heat Engine Laboratory of Bangladesh Institute of Technology (BIT), Rajshahi, Bangladesh. Here rice straw is a new material as feedstock. In this study, biomass solid waste is used as the heat source, which is renewable. Also a gravity feed type reactor feeder is used for feeding the feedstock.

2. MATERIALS AND METHODS

2.1 Biomass

The rice straw was collected locally in Rajshahi, Bangladesh. It was ground and sieved to the size of 300 to 1180 μm and finally dried for 24 hours at 110°C prior to pyrolysis.

2.2 Characterization of Solid Rice Straw for Pyrolysis Process

For the purpose of investigating the suitability of rice straw locally available in significant amount as feedstock for pyrolysis so as to obtain value-added liquid product, the following analyses have been considered:

- Bulk density and gross calorific value, and
- Proximate and ultimate analysis

The bulk density of the oven-dried sample of particle size 300 to 1180 μm prepared by the commonly used hammer milling technique was 223.47 kg/m^3 . The particle size of the feedstock has an important bearing on the ability to be heated quickly in a given heat flux environment. The higher density particles take less volume in the pyrolysis reactor and hence, reduce reactor volume. The gross calorific value (GCV) of the sample was determined using Plain Oxygen Bomb Calorimeter of model PARR 1341. The test was carried out in the Heat Engine Laboratory of Bangladesh Institute of Technology (BIT) Rajshahi, Bangladesh. The gross calorific value of the feedstock was 14.774 MJ/kg. The gross calorific value of the solid biomass might be compared to that of the pyrolytic oil product.

Proximate analysis gives information about feedstock suitability for pyrolysis process in terms of moisture content, ash content, volatile matter content and fixed carbon. The test was carried out according to the American Society for Testing and Materials (ASTM) Standard D3172-73 (1984) test procedures for solid fuel, titled "Standard Method for Proximate Analysis of Coal and Coke". It was conducted in the laboratory of Institute of Fuel Research and Development, Bangladesh Council of Science and Industrial Research (BCSIR), Dhaka, Bangladesh. The amount of volatile matter indicates the suitability of waste as the feedstock for pyrolysis conversion to liquid product where high volatile content is desirable [4]. Fixed carbon content gives information of the amount of char formation in the pyrolysis process. Moisture content is important in determining drying cost and energy content of the feedstock. The test results of the proximate analysis for the sample are presented in Table 1.

The elemental composition by ultimate analysis, in terms of carbon, hydrogen, nitrogen, oxygen and sulfur (CHNOS) contents of the selected biomass is essential for their pyrolysis conversion upon which the pyrolysis product quality depends. The test was carried out by an Elemental Analyzer of model EA 1108 according to the ASTM D3176-84 standard test procedures in the laboratory of Analytical Research Division, BCSIR, Dhaka, Bangladesh. The technique used for the determination of CHNS was based on the quantitative “dynamic flash combustion” method. The oxygen content was determined by difference, knowing the ash content. The elemental analysis of the biomass is presented also in Table 1.

Table 1 Proximate and Ultimate Analysis of the Biomass

Proximate analysis		Ultimate analysis	
Contents	wt%	Elements	wt%
Volatility	64.45	C	41.48
Fixed carbon	12.67	H	3.60
Moisture	9.47	N	-
Ash	13.41	O	41.51
		S	-

2.3 Experimental System

The experimental system was a fluidized bed pyrolysis unit. Nitrogen gas was used as fluidizing gas to make the system inert and dry silica sand was used as bed material. The sand in the fluidized bed was of mean size 181 μm diameter with static bed height of 5 cm. Figure 1 shows the schematic diagram of the pyrolysis conversion system. The reactor was 7 cm (diameter) x 37.5 cm (height), constructed of stainless steel with full gas flow and temperature control. The reactor was heated externally. The incoming fluidizing nitrogen gas was pre-heated before entering the reactor in a pre-heating chamber. The fluidizing gas flow rate was measured and controlled by nitrogen gas flow meter. The reactor bed and gas pre-heating chamber were heated by means of renewable energy biomass burning cylindrical heater. A blower supplied air required for burning biomass in the heater. By varying the air supply that was directly related to the blower speed, the temperature of the reactor was controlled. The temperature in the fluidized bed reactor was measured by a digital pyrometer. The gravity feed type heater feeder supplied the heater biomass required for continuous heating of the reactor. The ash of burned biomass in the heater was disposed through the ash disposal system at the bottom of the heater due to gravitational force only. Exhaust gas due to burning of heater biomass was exhausted in the atmosphere through a pipeline at the top of the heater. The biomass solid waste particles were fed into the reactor by a gravity feed type reactor feeder. The feed rate was maintained near about constant by a feed control valve at the bottom of the reactor feeder. Gravitational force was the main agent for feeding the reactor. The system was maintained at a pressure slightly above atmospheric by a nitrogen gas pressure regulator. The char was collected from the reactor after completing a run. The vapors and gases were passed through a water-cooled condenser to a series of two ice-cooled collectors to trap the derived liquid product. The fluidizing gas and noncondensable vapor were flared to the atmosphere. In this experimental study, the influence of reactor bed temperature and fluidizing gas flow rate on the liquid and the solid char product yields were studied. The reactor bed temperature was varied between 400°C to 500°C at an interval of 25°C while the fluidizing gas flow rate was varied between 23 to 39 l/min at an interval of 4 l/min.

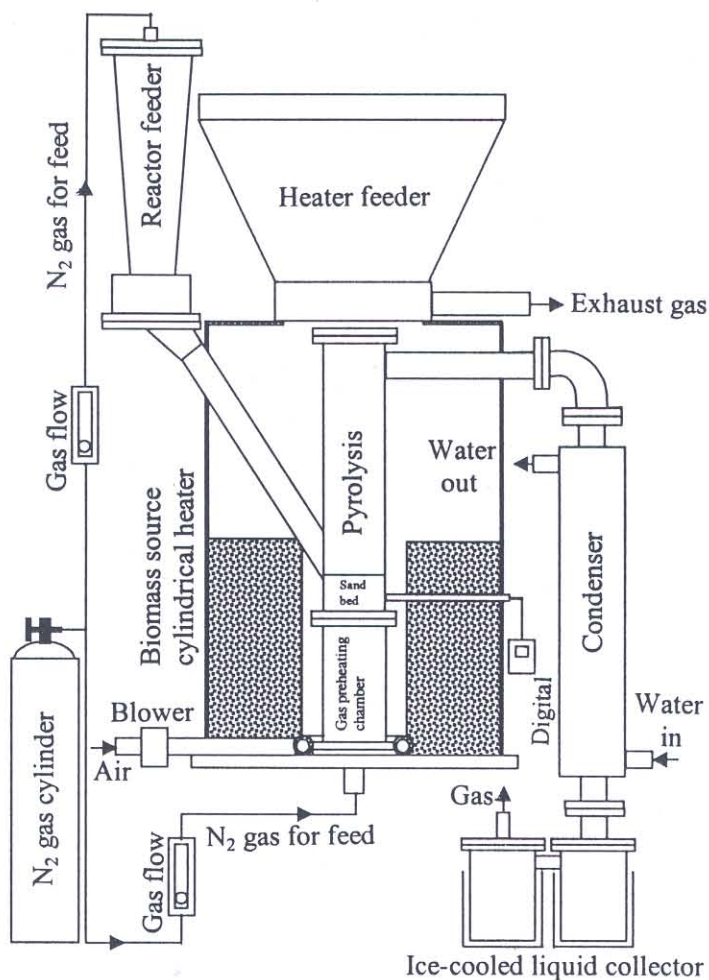


Fig. 1. Schematic diagram of fluidized bed pyrolysis system

2.4 Analysis of Pyrolysis Oil

2.4.1 Physical and chemical analyses

The pyrolytic oil at the maximum liquid yield condition was characterized for its physical properties. These properties were determined according to the American Society for Testing and Materials (ASTM) standard test methods. The properties determined were: kinematic viscosity, density, pH value, flash point, pour point and gross calorific value. The elemental analysis of the oil was conducted at Analytical Research Division of BCSIR, Dhaka. The elemental composition of the derived oil was determined using a Carbon, Hydrogen, Nitrogen and Sulphur (CHNS) elemental analyzer of model EA 1108. Oxygen was calculated by difference.

2.4.2 Compositional analysis

The functional groups of the pyrolysis liquid obtained at fluidized bed temperature of 450°C was analyzed by Fourier Transform InfraRed spectroscopy (FTIR) to identify the basic compositional groups.

The FTIR instrument of model SIMADZU FTIR 8400 was used to produce the ir-spectra of the derived oil. A thin uniform layer of the liquid was placed between two salt cells and exposed to infrared

beam. In the FTIR-8400, the infrared beam from light source was reflected by Collimator Mirror into the interferometer. A parallel beam entered the Michelson interferometer with a 30° incident angle. The beam was then divided by beam splitter, the resultant beams falling upon moving mirror and fixed mirror. Both beams were reflected back to the beam splitter and joined into one interference beam before proceeding to the collecting mirror. From the collecting mirror, the parallel infrared beam created an image of the light source at the center of the sample compartment. Another collecting mirror gathered the beam that passed through the sample and reflected it to the detector as the interferogram. After reaching the detector, the interferogram underwent several treatments before being sent to the computer. It was amplified by the preamplifier and the automatic gain amplifier, passed through high-pass and low-pass filters, and was digitized by the 20-bit A/D converter. After the signal was digitized into the interferometer memory, it traveled through the SCSI interface to the PC where Hyper-IR transformed the interferogram into a spectrum. It provided the absorption spectrum in percentage incident intensity, along the wave numbers 4000 to 500 cm^{-1} . The standard ir-spectra of hydrocarbons were used to identify the functional group of the components of the derived liquid. The test was conducted in the laboratory of Department of Chemistry of Rajshahi University, Rajshahi, Bangladesh.

3. RESULTS AND DISCUSSIONS

3.1 Characteristics of Solid Rice Straw

Table 1 shows that volatile content in the biomass was 64.45 wt% of the sample. This higher percentage is usually favorable to obtain liquid by fluidized bed pyrolysis technology [8]. The biomass also contains moderate amount of carbon that was 41.48 wt% of the sample. Sulfur and nitrogen were not found in the elemental analysis. Heat propagated into the solid drove off the inherent moisture at about 110°C, which was less than 4% of the total sample weight. At a heating rate of 60°C/min, devolatilization of solid rice straw was initiated at about 250°C and the rate was maximum between 300°C and 400°C. At around 450°C, devolatilization of the sample was completed. From these results, an idea was obtained to operate the pyrolysis system at a moderate temperature of around 450°C to obtain maximum percentage of liquid product.

3.2 Product Yields

Three products were obtained from the pyrolysis of rice straw. These were liquid, char and gas. The liquid obtained from rice straw waste was a single-phase dark brownish color product of acrid smell. No phase separation was found to take place. The liquid product was collected in two ice-cooled collectors in series. The char was collected from the reactor that retained the original geometrical shape of the rice straw feedstock particle. The gas was diluted with high fluidizing gas flow and was flared to the atmosphere. The water was not removed from the liquid since evaporation or distillation at normal temperature of around 100°C or higher can cause significant physical and chemical changes in the liquid [4].

3.3 Effect of Reaction Conditions on Product Yields

3.3.1 Effect of fluidized bed temperature

The yields obtained from different experimental run with rice straw as the feed materials of 300 to 1180 μm particle size at different fluidized bed reactor temperatures for an apparent vapor residence time of 2.80 seconds have been presented in Table 2 and Fig. 2. At lower and higher fluidized bed temperatures

the liquid yields were less in comparison to that at an intermediate temperature. However, the char yield was higher at lower temperature and this was found to be decreasing with increasing temperature. The gas yield was lower at lower temperature with an increasing trend at increasing temperature. The maximum liquid product yield was obtained at an intermediate temperature of 450°C. This was found to be 40 wt% of dry feed while the char yield was 45 wt% of dry biomass fed at this condition. At a lower temperature of 400°C the oil yield was found to be lower with a higher char yield. The char yield was 56 wt% of biomass fed with an oil yield of 31 wt%. On the other hand, at a higher temperature of 500°C, the char yield was found to be reduced with a reduction of liquid yield. The char yield was 36 wt% at 500°C with an oil yield of 30 wt%. The gas yield was found to increase with the increase of temperature.

The reason behind this is that the lower temperature was not sufficiently high enough for the pyrolysis devolatilization reaction to take place completely rendering reduced amount of liquid and gaseous products. Again the higher temperature was causing secondary cracking reaction of the vapors yielding more gas at the cost of the liquid product yield. However, the intermediate temperature was sufficient enough for complete pyrolysis reaction to take place and at the same time this temperature was not much high for secondary reaction to take place rendering maximum quantity of liquid product with less amounts of char residues and gaseous products.

Table 2 Effect of Fluidized Bed Temperature on Product Yields for Particle Size of 300 to 1180 μ m and Gas Flow Rate of 31 l/min

Run No.	Feed rate (kg/h)	Fluidized bed temperature (°C)	Liquid collected	Char collected	Noncondensable vapor
			wt% of biomass fed		
1	0.35	500	30	36	34
2	0.34	475	36	42	22
3	0.35	450	40	45	15
4	0.34	425	35	51	14
5	0.35	400	31	56	13

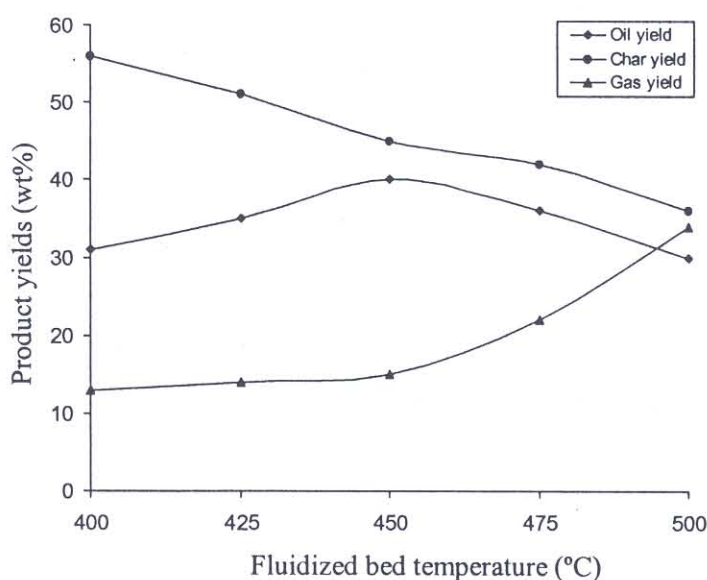


Fig. 2. Effect of reactor bed temperature on product yields for particle size of 300 to 1180 μ m and fluidizing gas flow rate of 31 l/min

3.3.2 Effect of fluidizing gas flow rate

The effect of fluidizing gas flow rate on product yields at a temperature of 450°C is presented in Table 3 and Fig. 3 which show the variation of the mass percentage of liquid, solid char and gas products for different gas flow rate at a reactor bed temperature of 450°C for feed particle of size 300 to 1180 μm . The maximum liquid product was 40 wt% of biomass fed while the solid char product was 43.5 wt% of dry fed at a fluidization gas rate of 31 l/min. It was observed that at the beginning with lower fluidization gas flow rate at 23 l/min, the liquid product was 29.5 wt% of biomass fed with a char product of 38.5 wt% of biomass fed and a gas yield of 32 wt%. With the increase of fluidization gas flow rate up to 31 l/min, the liquid product was increasing with a decreasing trend of gas yield. The char yield appeared to be increasing with the increase of gas flow rate.

This may be due to the fact that at lower fluidizing gas flow rate, the fluidization was not achieved completely and hence the fast pyrolysis reaction could not take place properly. At higher gas flow rate, the fluidization was good enough for high heating rate causing fast pyrolysis for maximum liquid yield. The vapor residence time was quite low which was not enough for secondary reaction to take place yielding less gas and more liquid product. When the fluidizing gas flow rate was increased to values higher than 31 l/min, char particles were found to be elutriated significantly into liquid collector, thus disturbing liquid collection.

Table 3 Effect of Fluidizing Gas Flow Rate on Product Yields for Particle Size of 300 to 1180 μm and Reactor Bed Temperature of 450°C

Run No.	Feed rate (kg/h)	Fluidized bed temperature (°C)	Liquid collected	Char collected	Noncondensable vapor
			wt% of biomass fed		
6	0.33	23	29.5	38.5	32
7	0.32	27	35	41	24
8	0.32	31	40	43.5	16.5
9	0.33	35	36	49	15
10	0.32	39	33.5	53	13.5

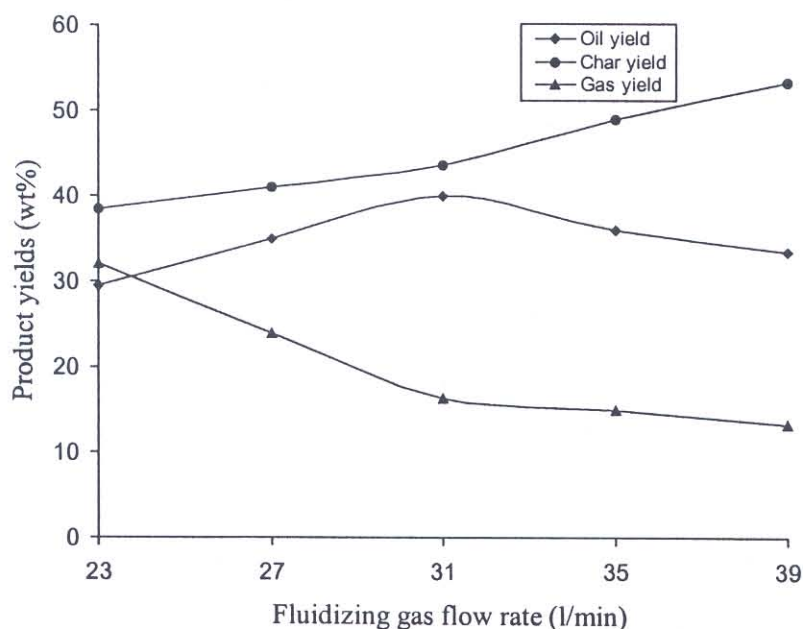


Fig. 3. Effect of fluidizing gas flow rate on product yields for particle size of 300 to 1180 μm and reactor bed temperature of 450°C

3.3.3 Effect of apparent vapor residence time

The product yield determination of rice straw pyrolysis at a fluidized bed temperature of 450°C for feedstock particle size of 300 to 1180 μm for different apparent vapor residence time has been presented in Fig. 4. It is evident from Fig. 4 that the maximum liquid product yield was obtained at an intermediate apparent vapor residence time of 2.80 seconds with a maximum solid char yield and a minimum gaseous product yield. Above and below this residence time, the oil yield is found to be reduced. With the decrease of apparent vapor residence time, char yield was increasing while gas yield was decreasing.

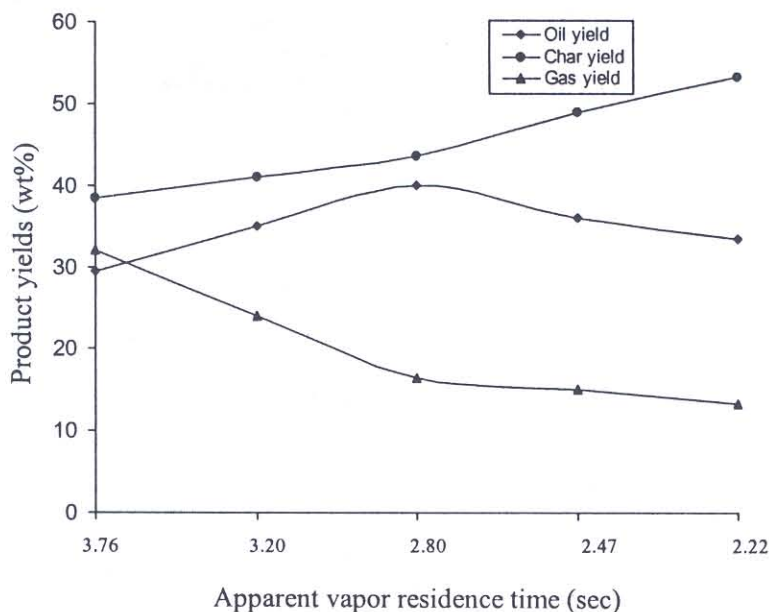


Fig. 4. Effect of apparent vapor residence time on product yields for particle size of 300 to 1180 μm and reactor bed temperature of 450°C

3.4 Characteristics of Product Oil

3.4.1 Physical and chemical characteristics

The elemental composition and the physical characteristics of the rice straw pyrolysis oil obtained at the maximum yield temperature of 450°C are shown in Table 4. The energy content of the oil was 18.340 MJ/kg, due to the presence of moisture and oxygenated compounds. However, it was significantly higher than that of raw rice straw. The oil was heavier than water with density of 1153.40 kg/m³ at 30°C and hence, results higher volumetric energy content. The flash point of the oil was 103°C hence, precautions are not required in handling and storage at normal atmosphere. The oil was easily pourable and the pour point was low, less than -8°C. The oil was found to have moderate viscosity. The liquid was very acidic with low pH value of 3.01 suggesting its corrosive nature.

The elemental analysis of the oil is an important criterion for the design of a combustion plant utilizing the oil with the help of the elemental analysis, the combustion calculation can be carried out from which the quantities of combustion air and flue gas and also the composition of the flue gas can be determined [12]. Bio-crude oils have some properties that are quite different from those of petroleum-derived oils. Bio-crude oils contain a large amount of oxygen. The oxygen content of bio-oils (excluding water) is a function of the oxygenated feedstock from which they are made, as well as the residence time and temperature of the pyrolysis step [10]. The oxygen content in the liquid was 50.96%, calculated by difference, and was close to that of feedstock composition. The sulfur content of bio-oils is naturally low, due to the low sulfur content of biomass. This low sulfur content is one of the positive aspects of

Table 4 The elemental composition and physical characteristics of biomass pyrolysis oil and its comparison

Analyses	Standard	Rice-straw*	Maple and oak wood* [10]	Fast diesel [11]
Elemental (wt%)				
C	ASTM D3176-84	42.30	44.80	86.10
H	ASTM D3176-84	6.53	7.20	12.80
N	ASTM D3176-84	Not detectable	0.1	-
S	ASTM D3176-84	Not detectable	0.01	0.5
Ash	ASTM D482-IP 4	0.21	0.09	0.01
O	By difference	50.96	47.80	-
Kinematic viscosity at 30°C (cSt)	ASTM D445-IP 71	10.81	70 [#]	1.3-3.3 [#]
Density (kg/m ³)	-	1153.40	1230	780
pH value	Digital pH meter	3.01	2.8	-
Flash point (°C)	ASTM D92-IP 36	103	>106	75
Pour point (°C)	ASTM D97-IP 15	-8	-9	-
GCV (MJ/kg)	DIN 51900	18.340	18.100	45.000-46.000

at 50°C; * Wet oil basis

bio-oils [10]. The amount of sulfur in the biomass-derived oils will indicate the quantity of the hazardous SO₂ emission in combustion processes. In this study, sulfur was not found and ash content was low at 0.21% of the oil weight. One of the variables with bio-oils will be the nitrogen content of the oil, which will reflect the variable protein content possible with biomass. Biomass which has a significant content of green, living plant cells (e.g., green grasses) will have a higher nitrogen content than biomass having a large content of dead plant cells (e.g., straw and wood) [10]. The nitrogen content in the bio-oils will contribute to NO_x emissions and it was not detectable in the rice straw pyrolytic product.

3.4.2 Compositional analysis

From the Fourier transform infrared spectroscopy of the derived pyrolytic oil, the FTIR functional groups and the indicated compositions of the liquid product are presented in Table 5. The presence of water impurities and other polymeric O-H in the oil were indicated by the broad absorbance peak of O-H stretching vibration between 3600 and 3200 cm⁻¹. The presence of alkanes was indicated by the strong absorbance peak of C-H vibrations between 3050 and 2800 cm⁻¹ and the C-H of deformation vibrations between 1490 and 1325 cm⁻¹. The absorbance peaks between 1775 and 1650 cm⁻¹ represented the C=O stretching vibration, indicating the presence of ketones, aldehydes and carboxylic acids. The possible presence of alkenes was indicated by the absorbance peaks between 1680 and 1575 cm⁻¹ representing C=C stretching vibrations. The sharp but less prominent absorbance peaks between 1550 and 1475 cm⁻¹ represented -NO₂ stretching vibration indicated the presence of small nitrogenous compounds. The overlapping peaks between 1300 and 950 cm⁻¹ were suggested due to the presence of primary, secondary and tertiary alcohols, ethers and esters due to the C-O stretching and O-H deformation vibration of these functional groups. Absorbance peaks between 900 and 650 cm⁻¹ indicated the possible presence of single, polycyclic and substituted aromatics groups. These functional groups and the indicated composition have been identified in the pyrolytic derived oils from palm shell and rice-husk from fluidized bed reactor by Ani and Zailani [13], and Islam and Ani [8], respectively.

The pronounced oxygenated functional groups of O-H; C=O; C-O and aromatic compounds showed that the oil was highly oxygenated and therefore very acidic, as have also been indicated by the elemental composition and the pH value. The high fraction of oxygenated compounds reduce the calorific value of the oil since C=O bonds do not release energy during combustion. The presence of hydrocarbon groups C-H; C=C; and alcohols indicates that the liquid has a potential to be used as fuel.

Table 5 FTIR Functional Groups and the Indicated Compounds of Biomass Pyrolysis Oil

Frequency range (cm ⁻¹)	Groups	Class of compounds
3600 - 3200	O-H stretching	Polymeric O-H, water impurities
3050 - 2800	C-H stretching	Alkanes
1775 - 1650	C=O stretching	Ketones, Aldehydes, Carboxylic acids
1680 - 1575	C=C stretching	Alkenes
1550 - 1475	-NO ₂ stretching	Nitrogenous compounds
1490 - 1325	C-H bending	Alkanes
1300 - 950	C-O stretching, O-H bending	Primary, secondary and tertiary alcohols, phenol, esters and ethers
900 - 650		Aromatic compounds

4. CONCLUSIONS

- Fluidized bed pyrolysis of solid rice straw had given maximum oil yield at 40 wt% of biomass fed at a reactor bed temperature of 450°C and at a gas flow rate of 31 l/min. With increasing fluidized bed temperature, the percentage mass of char production was decreasing while gas production was increasing.
- The liquid yield was found to be increasing with fluidizing gas flow rate up to 31 l/min at a reactor bed temperature of 450°C. The solid char was also found to follow a similar trend up to 39 l/min.
- The pyrolytic oil was a single-phase liquid product.
- The elemental composition of the oil was better than that of solid biomass.
- FTIR analysis showed that the liquid was dominant with oxygenated species.
- The physical property analysis showed that the oil was heavy and acidic in nature with moderate viscosity. The oil possessed favorable pour and flash points.
- The heating value of the oil was quite low, however, the value is similar to other biomass derived pyrolytic oil. This value was significantly higher than that of solid rice straw.

5. ACKNOWLEDGEMENTS

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6. NOMENCLATURE

CHNOS	=	carbon, hydrogen, nitrogen, oxygen and sulfur analysis
FTIR	=	Fourier transform infrared
ASTM	=	American Society of Testing Materials
TCD	=	thermal conductivity detector
O-H	=	hydroxyl stretching
C-H	=	carbon hydrogen stretching and bending
C=O	=	carbonyl stretching

C=C	=	carbon carbon double bonding stretching
-N ₂ O	=	nitrogen dioxide stretching
C-O	=	carbon oxygen stretching

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