Pyrolysis of Rice Husk in a Fixed Bed Reactor

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Abstract—Fixed-bed slow pyrolysis experiments of rice husk have been conducted to determine the effect of pyrolysis temperature, heating rate, particle size and reactor length on the pyrolysis product yields. Pyrolysis experiments were performed at pyrolysis temperature between 400 and 600°C with a constant heating rate of 60°C/min and particle sizes of 0.60-1.18 mm. The optimum process conditions for maximum liquid yield from the rice husk pyrolysis in a fixed bed reactor were also identified. The highest liquid yield was obtained at a pyrolysis temperature of 500°C, particle size of 1.18-1.80 mm, with a heating rate of 60°C/min in a 300 mm length reactor. The obtained yield of, liquid, gas and solid were found be in the range of 22.57-31.78 %, 27.75-42.26 % and 34.17-42.52 % (all weight basics) respectively at different pyrolysis conditions. The results indicate that the effects of pyrolysis temperature and particle size on the pyrolysis yield are more significant than that of heating rate and reactor length. The functional groups and chemical compositions present in the liquid obtained at optimum conditions were identified by Fourier Transform-Infrared (FT-IR) spectroscopy and Gas Chromatography/ Mass Spectroscopy (GC/MS) analysis respectively.

Keywords-Slow pyrolysis, Rice husk, Recycling, Biomass.

I. INTRODUCTION

THE availability of fossil fuels is limited, but the demand I of energy from fossil is growing at high rate due to the development of all aspects of the world and the utilization of the fossil fuel generating the environmental problems. There is a need for identifying the sustainable energy options for energy production without polluting the environment. The renewable energy source can play a major role for sustainable development. Among the possible renewable energy options, agricultural and forestry residues (generally called biomass residues) can be used as raw materials to generate energy [1]. Pyrolysis is one of thermo chemical processes, which convert the solid biomass in to liquid (bio- oil), gas, and char. Biomass pyrolysis converts essentially 80-95% of the feed material to gases and bio-oil. The pyrolysis process is to be optimized to maximize the production of liquids (tar and bio-oil). The liquid products have high energy density, ease of transportation & storage and the potential to be upgraded as

fuels similar to refined premium-grade fuels [2]. The gross calorific value of the bio oil obtained from pyrolysis of rapeseed oil cake [3], empty fruit bunches [4] and cashew nut shell [5] were reported to be 33, 36 and 40 MJ/kg respectively. The objective of present work is to study the influence of the pyrolysis temperature, particle size range, reactor length and heating rate on the pyrolysis yield of rice husk and to find the optimum parameters for maximum liquid yield in a fixed bed reactor with slow pyrolysis process. The study also aims at determining the functional groups and chemical mixtures present in the liquid at optimum parameters.

II. MATERIALS AND METHODS

A. Raw Material – Rice Husk

Samples of rice husk studied in the present study were originated from Ponnari, Tiruvallur district, located in the Tamilnadu, India. Immediately after getting, the rice husks were sun dried for few days to remove the moisture content, after which they were ground in a high speed rotary cutting mill and screened by standard sieve (IS designation 460-1962) separator to give fractions of less than 0.15 mm, 0.15-0.30 mm, 0.30-0.60 mm, 0.60-1.18 mm and 1.18-1.80 mm. The proximate, ultimate and component analysis were done by ASTM standards. The components and elements present in the rice husk are given in Table I.

TABLE I
COMPONENTS AND ELEMENTAL ANALYSIS OF RICE HUSK

Components	
Volatiles	70.6%
Fixed carbon	2.97%
Ash	17.09%
Elemental analysis (%)	
Carbon	50.45%
Hydrogen	6.58%
Oxygen	41.46%
Nitrogen	1.49%
Sulphur	0.23%
Calorific value (KJ/kg)	19807
Moisture content (%)	9.45%

B. Experimental Apparatus and Procedure

The stainless steel tube with a length of 300 mm, 400 mm or 500 mm are taken as the reactors for conducting the pyrolysis process. The reactor is heated externally by an electric furnace, with the temperature being measured by a Cr-Al: K type thermocouple fixed inside the reactor and

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temperature is controlled by external PID controller. The condensable liquid products (bio-oil + water) were collected in a trap maintained at the temperature of 25° C by means of circulating the water in the condenser. The schematic diagram of the pyrolyser setup is shown in Fig. 1. The liquid product condensed in the condenser is weighed. After pyrolysis, the solid residue is removed and weighed. The gaseous phase is then calculated by the material balance. The rice husk input, liquid and solid char are measured by the weighing machine with an accuracy of 0.01g.



Fig. 1 Pyrolyser experimental setup

The effect of temperature on the pyrolysis product yields is investigated initially by varying the pyrolysis temperature from 400 to 600°C in a 300 mm length reactor and keeping the particle size and heating rate as constant values of 0.60 to 1.18 mm and 60°C/min respectively. The second is carried out to determine the effect of particle size on pyrolysis yield of the rice husk with a heating rate of 60°C/min, pyrolysis temperature of 500°C and particle size in the range of <0.15 mm to 1.80 mm.

The effect of heating rates on the pyrolysis yield is calculated by varying the heating rate from 20° C/min to 60° C/min with the constant particle size of 1.18-1.80 mm, at a pyrolysis temperature of 500°C in a 300 mm length reactor. Finally, the effect of residence time on pyrolysis yield is analyzed by conducting the pyrolysis process in three different reactors with a length of 300 mm, 400 mm and 500 mm with the constant temperature, particle size and heating rate of 500°C, 1.18-1.80 mm and 60°C/min respectively.

The functional groups present with the pyrolysis liquid obtained at optimum conditions were identified by Fourier Transform Infrared (FT-IR) spectroscopy analysis. A Perkin Elmer Spectrum device with a resolution of 1.0 cm⁻¹ was used for finding the functional groups presents in the pyrolysis liquid. The chemicals present with the pyrolysis liquid obtained at optimum parameters was analyzed by Gas Chromatography/Mass Spectroscopy (GC/MS).

III. RESULTS AND DISCUSSION

A. Effect of Temperature on the Pyrolysis Yield

Fig. 2 presents product distribution from slow pyrolysis of rice husk (particle size 0.60-1.18 mm) with the pyrolysis temperature from 400° C to 600° C and heating rate of 60° C/min. The results shows that the liquid yield increases from 27.19 to 30.11 % when the temperature is increased from 400 to 500° C then decreases to 29.05 % when the temperature is further increased to 600° C. The gas yield increases from 32.83 to 38.42 % when the pyrolysis temperature is increased from 400 to 600° C, but the solid yield significantly decreases from 39.98 to 32.53 % with the same temperature rising.

The decrease in the solid yield with the increasing temperature could be due to greater primary decomposition of the sample at higher temperature. The decrease of liquid yield and sudden increase of gas yield are observed at higher temperature, this may be due to secondary cracking of the pyrolysis liquid in to gaseous product at higher temperature. This results are found to be an agreement with the study of fixed-bed slow pyrolysis experiments conducted on a sample of rapeseed, safflower seed press cake and soybean cake [6-8]. However, the higher ash content in the rice husk may be the cause for higher gas and lower liquid yield compared with the other samples, as silica in the ash play a role of active catalyst.



Fig. 2 Yield of pyrolysis products at various pyrolysis temperatures

B. Effect of Particle Size on Pyrolysis Yield

Decrease in particle size leads to decreases of liquid yield because the residence time of the volatiles in the reactor is longer, favoring the cracking of hydrocarbons, when smaller particles are used [9]. More residence time of volatiles inside the reactor leads to cracking of heavier molecules (tar) in to lower molecules at lower particle size ranges and it results in increase of gaseous product.

From Fig. 3, the results shows that the char and liquid yield is significantly increased as the particle size is increased from less than 0.15 mm to1.80 mm and gas yield is found to decrease. The study found that solid and liquid yield increases from 34.17 to 36.37 % and 22.57 to 31.78 % respectively and gas yield decreases from 43.26 to 32.31 % when the particle size is increased from <0.15 to 1.80 mm.

The increases in the solid yield with the increasing particle size for the sample could be due to greater temperature gradient inside the particles, so that at a given time the core temperature is lower than that of the surface, which possibly gives rise to an increase in the solid yields. There is a significant effect of particle size on the char, oil and gas yield in the pyrolysis of olive bagasse, pine sawdust, wood birch [10-12].



Fig. 3 Yield of pyrolysis products with different particle size

C. Effect of Heating Rate on Pyrolysis Yields

Heating rate is one of the important parameter for the yield of different products from the pyrolysis process. At low heating rates, there may be some resistances to mass or heat transfer inside the particles of the sample, but increasing the heating rate breaks the heat and mass transfer limitation in the pyrolysis and increasing the liquid yield and decreasing solid formation.

To determine the effect of heating rate on the yields of the pyrolysis products of rice husk the experiments were conducted at different heating rates of either 20, 40, 60°C/min with the constant pyrolysis temperature of 500°C and the particle size of 1.18-1.80 mm in a 300 mm length reactor. The study found that the liquid yield is low at lower heating rate and increases with the increase of heating rate, the liquid yield is 29.73 % at the heating rate of 20°C/min and increases to the value of 31.78 % when the heating rate is increased to 60°C/min. The gas yield also increases with increase in heating rate, but the solid yield decreases with the increase in heating rate. The solid yield is found to decrease from 42.52 % to 36.04 % when the heating rate is increased from 20°C/min to 60°C/min (Fig. 4). The increasing of the liquid yield with the increase of heating rate may be due to higher heating rates that break the heat and mass transfer barriers in the particles [10].



Fig. 4 Effect of heating rate on pyrolysis yield

D. Effect of Residence Time on Pyrolysis Yield

The effect of residence time on the pyrolysis yield was investigated by varying the reactor length. Fig. 5 shows the influence of residence time on the pyrolysis yield. The gas yield from 500 mm reactor was higher than that from the 300 mm length reactor. The liquid yield from 300 mm reactor was higher than that from the 500 mm reactor. Higher reactor length results in lower liquid yield, due to the secondary cracking of the liquid yield in the 500 mm length reactor owing to prolonged residence time of the vapours inside the reactor. The study observed that there are low variations of solid yield in all the three reactors.



Fig. 5 Effect of residence time on pyrolysis yield

IV. PROPERTIES OF PYROLYSIS LIQUID

The IR spectrum of the liquid obtained from pyrolysis of rice husk at the pyrolysis temperature of 500°C, particle size of 1.18-1.80 mm, heating rate of 60°C/min and 300 mm length reactor is shown in Fig.6. The O–H stretching vibrations between 3200 and 3400 cm⁻¹ indicate the presence of phenols and alcohols. The C–H stretching vibrations between 2800 and 3000 cm⁻¹ and C–H deformation vibrations between 1350 and 1475 cm⁻¹ indicate the presence of alkanes. The C=O stretching vibrations with absorbance between 1650 and 1750 cm⁻¹ indicate the presence of ketones or aldehydes. The absorbance peaks between 1575 and 1675 cm⁻¹ represent C=C stretching vibrations indicative of alkenes and aromatics. The C–H stretching and bending vibrations between 1380 and 1465 cm⁻¹ indicates the presence of alkane groups in pyrolysis oils derived from biomass. Absorptions between 1300 and 900

 cm^{-1} indicates the carbonyl components (i.e., alcohols, esters, carboxylic acids or ethers). The absorbance peaks between 900 and 690 cm⁻¹ represent O-H stretching vibrations indicative of aromatic groups.

The Table II indicates the peaks obtained from FT-IR spectroscopy of the rice husk pyrolysis liquid and the corresponding functional groups and their transmittance percentage of the pyrolysis liquid obtained for rice husk pyrolysis.

S.N	Wave	Functional group	% of
0	number		transmittanc
			е
1.	3388	Alcohol/Phenol	7.84
2	2931	Alkyl/ Carboxylic	9.20
		Acid	
3	1706	Ketones	3.36
4	1463	amides	8.19
5	1267	Esters	4.73
6	1118	Alcohol/Phenol	16.61
7	1031	Alcohol/Phenol	26.81
8	810	Amine/ Benz	49.34
9	753	Amine/ Benz/	41.24
		Chlorides	
10	626	Bromides/ Chlorides	65.51

The chemicals present in the pyrolysis liquid obtained at optimum conditions was analyzed by Gas Chromatography-Mass Spectroscopy (GC-MS) analysis. The Fig. 7 indicates the GC analysis of pyrolysis liquid, the peaks with different retention Time (RT) indicates the presence of different chemicals in it. The chemicals with different retention time peaks were identified by the mass spectroscopy. The mass spectroscopy analysis indicates the presence of acetic acid, phenol, 1, 2–benzenediol, 1-hydroxy-2-butanone, furfural, phenol-3-methyl, 2,5-dimethyl-phenol, benzene, 1-ethyl-4methoxy and 2,5-dimethyl-benzoic acid present with different percentages in the rice husk pyrolysis liquid.

V. CONCLUSION

The present study, pyrolysis experiments of the rice husk are carried out in a fixed bed reactor with different pyrolysis temperature, particle size, heating rates and reactor length. The liquid obtained at the optimum parameters are tested for finding its properties. The following are the findings from pyrolysis experiments of coconut shell in the fixed bed reactor.

- The optimum process conditions for maximizing the liquid yield of slow pyrolysis of rice husk in a fixed bed are: pyrolysis temperature 500°C, particle size 1.18-1.80 mm, heating rate 60°C/min and the reactor length 300 mm. The maximum liquid yield of 31.78 % was obtained at the optimum condition.
- Employing higher particle size results in more amounts of liquid and solid yield and less amount of gas yield compared to lower particle size. The gas yield decreases from 43.26 to 32.31 % when particle size is increased from less than 0.15 to 1.8 mm.
- The liquid yield is increased from 29.73 to 31.78 % when the heating rate is increased from 20°C/min to 60°C/min.The effect of heating rate on the pyrolysis yield is not significant compared to other parameters in this study.
- The FTIR and GC/MS analysis showed that the liquid composition was dominated by oxygenated species. The high oxygen content is reflected by the presence of mostly oxygenated fractions such as carboxyl and Alcohol groups produced by pyrolysis of the cellulose and phenolic and methoxy groups produced by pyrolysis of the lignin.
- The utilization of liquid as fuels or sources of chemical feedstock require some form of upgrading to improve storage stability and heating value and it can be used directly or mixing with other conventional fuels after up gradation.



Fig. 6 FT - IR spectra of pyrolysis liquid

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Fig. 7 The GC analysis of rice husk pyrolysis liquid

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