

Fast Pyrolysis Of Pakistani Cotton Stalks In Fluidized Bed Reactor: Design And Preliminary Results

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Abstract: Agricultural residues can be converted in the biofuels to fulfil the world energy demands and achieve the targets set by different countries to replace fossil fuels with renewable resources. Fast pyrolysis of cotton stalk, obtained from agriculture fields of Punjab, Pakistan, is being investigated. A Lab Scale bubbling fluidized bed fast pyrolysis system is designed and fabricated. The operating parameters of the fluidized bed reactor are selected on the basis of the study of hydrodynamics of cold model and TGA analysis. Experiments are conducted over a range of operating conditions. Yield of char and gases are measured. The products are analyzed on FTIR and GC-MS to investigate the functional groups and compounds present in the pyrolysis liquid derived from the cotton stalks. GC-MS analysis of pyrolysis liquid has shown number of peaks with the compounds identified as acetone, benzene, 3-methyl pentane, 2,2-dimethylbutane, 3,5- dimethyloctane, 2-methylhexanoic acid, 1,2- benzene dicarboxylic acid, Bis (2-ethylhexyl) phthalate whereas GC analysis of exhaust gas has shown three peaks containing gases H₂, CO₂ and n-butane. Effect of temperature is investigated on the yield of char and the no. of compounds present in the pyrolysis of liquid along with their functional groups.

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1. Introduction

Biomass is an alternate source of energy in the world. The consumption of fossil fuels has been increasing with the growth of population and causes the depletion of these resources and environmental threat. Ultimately, the production of sustainable energy has turned out to be the concern of today. Fluidized bed fast pyrolysis is one of the most promising technologies among the thermochemical conversion processes to convert waste biomass into liquid fuels due to high biomass conversion efficiency (Bridgwater and Bridge 1991; Meier and Faix 1999; Demirbas 2000). Pakistan is an agricultural based country and produces agricultural wastes such as wheat straw, rice straw, cotton stalks, maize stalks, sugar cane trash, corn stover, tobacco stalks etc in huge volume. The annual production of these residues is about 84 million tons and conventional use of this waste is for the purpose of heating, cooking and animal fodder (Koopmans and Koppejan 1997). This agricultural waste is an alternate potential resource for energy production and its exploitation into liquid fuel using different technologies should be considered. Consequently, the agricultural wastes which are useless and produce environmental problems can be made into more valuable energy rich products which are environment friendly.

Fast pyrolysis process is the thermal decomposition of biomass at moderate temperature in the presence of inert atmosphere and produces liquid (bio oil), solid (charcoal) and incondensable gas. The key objective of this fast pyrolysis technology is to convert solid biomass into liquid fuel (Goyal HB 2006). The proportions of these three products depend on the process conditions and feedstock used. The pyrolysis product fractions of liquid, charcoal and incondensable gas are 60-75, 15-25 and 10-20 wt.% (Mohan, Pittman et al. 2006). Pyrolyzed gas mainly consists of carbon monoxide (CO), methane (CH₄) and carbon dioxide (CO₂). Bio oil consists of the oxygenated compounds having functional groups such as carboxyl, carbonyl as well as phenolic. The main components of the liquid bio oil are water, insoluble lignin fractions, aldehydes, carboxylic acids, carbohydrates, phenols, ketones, alcohols and furfural (Balat, Balat et al. 2009). Fast pyrolysis of biomass can be performed with a variety of reactors such as entrained flow reactors, vacuum furnace reactors, vortex reactors, rotating cone reactors, circulating fluidized bed reactors and fluidized bed reactors (Bridgwater 1999). These fast pyrolysis reactors are identified by their specific characteristics and possess many problems of poor heat and mass transfer, difficult structure, working cost and even not one

reactor among them can be thought an ideal fast pyrolysis reactor.

Therefore, fluidized bed reactors are considered most suitable for fast pyrolysis of biomass due to high heat and mass transfer rates, rapid devolatilization, easy control, ready scale up and are the main focus of this research.

This research paper states the design and fabrication of a laboratory scale fluidized bed fast pyrolysis system for cotton stalks and preliminary test results including characterization of the products.

In this research, focus is on the experimental study of fast pyrolysis of Pakistani cotton stalks using fluidized bed reactor with different pyrolysis conditions.

2. Fast Pyrolysis System

2.1 Description of the Fast Pyrolysis System

The fast pyrolysis system (figure 1) is designed with working capacity of 2 kg/hr feedstock. The major components of the system are biomass feeding system, fluidized bed reactor, heating system, gas solid separation system, pyrolysis vapors condensation system and instrumentation and control system.

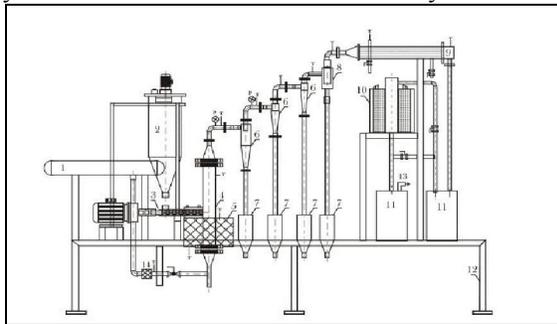


Figure 1: Schematic Diagram of Fast Pyrolysis System: 1. Nitrogen Gas Header 2. Feed Hopper 3. Screw Feeder 4. Fluidized Bed Reactor 5. Reactor Heater 6. Cyclone Separators 7. Char Collectors 8. Hot Filter 9. Water Condenser 10. Dry Ice Condenser 11. Bio Oil Collectors 12. Frame 13. Exhaust Gas 14. Nitrogen Preheater

In the fast pyrolysis process, screw feeder is used to transport the regulated biomass feedstock into the fluidized bed reactor and preheated inert nitrogen gas is passed through the bottom of the reactor as a fluidization gas for the sand bed. Pyrolysed volatiles along with charcoal leave the reactor from the top. The solid char particles are separated from the gas in the cyclone separators and pyrolysis gas is first condensed in the water cooled condenser and then in dry ice condenser to obtain pyrolysis liquid. The non-condensable gas is passed through a filter to remove fine solids and then samples of gas are taken for analysis. The remaining gas is burnt in the flare.

2.2 Design and Fabrication of Rig

2.2.1 Biomass Feeding System

The screw feeding is considered as the best method to transport the biomass materials. The key advantages of this feeding system are simple structure, good sealing efficiency, low cost fabrication and convenient operation (Ren, Wang et al. 2011). The feeding system consists of feed hopper, mechanical stirrer and screw feeder. The feed hopper is designed conical at the bottom to contain the biomass feedstock with capacity of 30 L volume. It is made of stainless steel 316 and provided with mechanical vibrator. Mechanical vibrator is installed to avoid the biomass feed from suspending and bridging during operation.

The biomass feed drops on the moving screw and fed into the fluidized bed reactor. The part of the carrier inert gas is used in the feed line to entrain the biomass feed. The screw is made of stainless steel 316 L and rotates in water cooled jacket. The cooling jacket is fixed to prevent the smelting of the biomass feed. In the screw, a gasket made of teflon / graphite is fixed as a sealing material. The feeder is calibrated for max 2.0 to min 0.5 Kg/hr.

2.2.2 Fluidized Bed Reactor

The vapor residence time in the fluidized bed reactor is a fundamental parameter to carry out the fast pyrolysis process for biofuel liquid production. The short vapor residence time of less than 2 seconds is recommended for this process in order to maximize the liquid product and avoid secondary cracking reactions (Bridgwater 2012). The vapor residence time is decided from the volume of the reactor and flow rate of inert fluidizing gas. Ultimately, the size of the fluidized bed reactor is reliant on the gas flow rate. The reactor is designed and fabricated in cylindrical shape with top and bottom conical made of stainless steel 316 L pipe of wall thickness 5 mm, internal diameter of 100 mm and length of 798 mm. At the bottom of reactor, distributor plate of mesh size 100 micron is fitted in the flange to retain the silica sand bed. Figure 2 describes the characteristics of the bubbling fluidized bed reactor used in this research.

The fluidized bed reactor is heated indirectly from outside at the bottom. The heater consists of four electric carbide rods of 2.5 KW power each and maximum heating temperature of 1000°C can be achieved. This heating system gives the quick heat input resulting in high heating rate required in the fast pyrolysis process. Fast pyrolysis of biomass is carried out at temperature of 450-550°C with inert fluidizing gas (Balat, Balat et al. 2009; Ma, Wang et al. 2012). The fluidizing gas is heated upto the temperature of 350°C in the preheater before entering into the reactor. The preheater is an insulated tubular heater containing four carbide electric heating rods of each 2KW capacity.

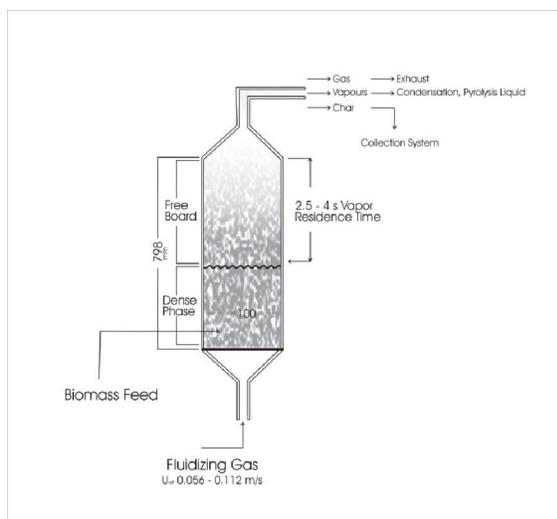


Figure 2: Characteristics of Fluidized Bed Reactor

2.2.3 Gas Solid Separation System

Pyrolysis gas leaving the fluidized bed reactor comprises the small size particles such as sand charcoal, ash and gas solid separation system is used to remove these particles. Charcoal particles are essential to separate from the pyrolysed gas because they can cause the secondary cracking reactions in the vapor phase at high temperatures reducing the yield of liquid product (Bridgwater, Meier et al. 1999) as well as reduce the efficiency of the condensation system by blocking the condenser tubes. Multi cyclone system is preferred for the separation of solid particles from the gas which unenviably affects the high pressure drop. Three stage cyclone separation system is designed for solids separation with diameter of first cyclone as 98 mm and other two with diameter of 68 mm.

The cyclones and connecting pipe from reactor exit to inlet of condenser are heated externally installing electric filaments to maintain the temperature of 350-400°C in the vapor phase. This vapor phase temperature is established so that prior condensation of tar vapors should not take place (Yanik, Kornmayer et al. 2007).

2.2.4 Vapors Condensation System

The condensation of pyrolysis vapors leaving the reactor affects the yield and quality of the bio oil and rapid quenching gives the higher amount of bio oil yield. The pyrolysis liquid derived from biomass contains tarry materials which can deposit in the condenser and pipelines (Islam, Islam et al. 2004). The shell and tube water condenser and simple dry ice condenser are designed to enable the easy cleaning. The condensers are made of stainless steel due to corrosive nature of the liquid. The diameter of the shell is 114 mm whereas dia of condenser tube is 13 mm and length is 693 mm. The dry ice condenser is

made of stainless steel and has shell dia of 324 mm and height 380 mm.

2.2.5 Instrumentation and Measurement System

Monitoring system of fast pyrolysis system includes temperature, pressure and flow rate measurement. Thermocouples are inserted to monitor the temperature in preheater, fluidized bed reactor, cyclones and condensers. Temperature controllers are installed in the preheater and reactor bed to maintain and measure the uniform temperature in the system. Three rotameters are fixed at the inlet of preheater, base of hopper and exhaust gas to measure the flow rates of nitrogen carrier gas and vent gas. Pressure gauges are placed at outlet of the reactor and at the cyclones to indicate the pressure. To monitor the feed rate of biomass, load cells are used which shows the weight consumed in the hopper.

Table 1. Characterization of Cotton Stalk

Proximate Analysis (wt %)	
Moisture	10.68
Volatile Matter	69.27
Fixed Carbon	6.19
Ash	13.95
Ultimate Analysis (wt %)	
Carbon	42.21
Hydrogen	5.59
Nitrogen	1.15
Sulphur	0.04
Oxygen	51.01
HHV (MJ/Kg)	17.40
Chemical Composition (wt %)	
Cellulose	26.90
Hemicellulose	19.10
Lignin	22.00

Nitrogen gas is measured from cylinder in two streams, one to the reactor and second to the biomass feed in order to purge the trapped air from the system and carry out the fast pyrolysis process in the absence of oxygen (Heo, Park et al. 2010). The flow rate of fluidizing nitrogen gas to the reactor and feed line is regulated through the needle valve. The maximum flow rate of nitrogen used is 8 m³/hr at 1bar measured by variable area flow meter. Biomass feed is conveyed to the reactor from hopper using screw feeder and feed rate is adjusted with the speed of feeder. The temperature of the fluidized bed reactor is controlled by a controller indicated using thermocouple inserted above the sand bed.

3. Materials and Methods

Shake down trials are carried out to identify and eradicate the fabrication faults of the fast pyrolysis system and to operate the system smoothly. The experiments are performed to optimize the various

operating parameters such as temperature, fluidizing gas flow rate, vapor residence time and feed rate of biomass. Inert nitrogen gas is used as a carrier / purge gas to fluidize the silica sand bed of particle size 350-450 μm in the fast pyrolysis system. The silica sand is practiced as heat transfer medium in the fluidized bed reactor. Nitrogen gas is preheated at temperature of 342- 444 $^{\circ}\text{C}$ before feeding into the reactor to get steady state condition.

Cotton stalk is used as a feedstock to test and optimize the fast pyrolysis system. The ultimate analysis, proximate analysis, heating value and chemical composition of cotton stalk is described in table 1.

Since bio oil contains a wide range of low molecular weight as well as high molecular weight hydrocarbon compounds, therefore complete analysis is not possible. The qualitative analysis of bio oil and exhaust gas are carried out with GC- MS and GC analyzers. FTIR spectroscopy is applied to identify the functional groups and bonds in the pyrolysis liquid at the reactor bed temperature. Water content in the bio oil is determined by the Karl Fischer Titrator using methanol as a solvent and KF titration reagent. The mass of each fraction in the experiment is achieved by weighing the feed and product (liquid, char and gas). In the product, pyrolysis liquid and char is weighed and exhaust gas is calculated by difference of total input and output. Figure 3 shows the mass balance of the fluidized bed reactor.

Table 2. Test Conditions for Fast Pyrolysis

Test	1	2	3	4
Nitrogen Preheater Temp($^{\circ}\text{C}$)	432	444	342	396
Reactor Bed Temp($^{\circ}\text{C}$)	537	524	366	401
Reactor				
Top Temp($^{\circ}\text{C}$)	430	362	462	390
Cyclone1 Temp($^{\circ}\text{C}$)	454	404	409	465
Cyclone2 Temp($^{\circ}\text{C}$)	458	502	302	434
Cyclone3 Temp($^{\circ}\text{C}$)	411	449	337	397
Nitrogen flow rate (m^3/hr)	8	4	4	4
Feed rate (g/min)	16	10	6	4

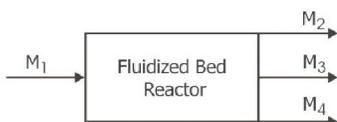


Figure 3: Mass Balance along Reactor

Mass balance and yields of pyrolysis products achieved from the experimental runs are defined in the equations (1-7). The yields are designated as mass of individual pyrolysis product to the mass of input biomass. The pyrolysis liquid is defined as the sum of each fraction collected from the water condenser (WC) and dry ice condenser (DIC). The charcoal is the amount of each part from cyclones 1, 2, 3 and filter respectively.

Overall Mass balance is

$$M_1 = M_2 + M_3 + M_4 \quad (1)$$

$$Y_C = 100 * M_4 / M_1 \quad (2)$$

$$M_4 = M_{C1} + M_{C2} + M_{C3} + M_F \quad (3)$$

$$Y_L = 100 * M_3 / M_1 \quad (4)$$

$$M_3 = M_{WC} + M_{DIC} \quad (5)$$

$$Y_G = 100 - Y_L - Y_C \quad (6)$$

$$\text{Mass Efficiency} = (M_2 + M_3 + M_4 / M_1) * 100 \quad (7)$$

Table 3. Mass Balance of Pyrolysis Products

Test	Liquid (wt. %)	Char (wt. %)	Gas (wt. %) by difference
1	21	44	35
2	18	10	72
3	16	13	71
4	14	8	78

4. Results and Discussion

The pyrolysis product consists of three constituents such as liquid, char and gas which are collected after each experiment. The pyrolysis oil obtained is a single phase almost homogenous liquid with dark brown color and acidic odor as shown in figure 4 and figure 5. The pH of pyrolysis liquid is 2.84-2.94 which shows that it is acidic in nature.



Figure 4: Bio Oil from Water Condenser Collector .

The results of mass balance show that yield of pyrolysis liquid is 14-21 % which is low in all the runs whereas yield of char is from 8-44 % and yield of produced gas is 35-78% which is high. These results may be due to secondary cracking reactions as well as the operating conditions of the reactor. In the first test result, yield of char is higher in comparison with result of other tests which may be due to greater fluidization velocity (i.e. 0.3 m/s) carrying forward the sand

In this IR Spectra, absorbance vs wavenumber peaks obtained are analysed to identify the functional groups or bonds present in the pyrolysis liquid and are shown in the table 4.

Table 4. FTIR Functional Groups and Bonds

Wavenumber (cm ⁻¹) experimental	Wavenumber (cm ⁻¹) Range	Functional Group	Bond
3353 (s, b)	3500-3200	Phenols, alcohols	O-H
2929 (m, n)	3300-2500	Carboxylic acids	O-H
1702 (s, n)	1750-1625	Ketones	C=O
1639(m, n)	1750-1625	Aldehydes	C=O
1454 (m, n)	1470-1450	Alkanes	C-C
1365(m, n)	1370-1350	Alkanes	C-C
1021(s, s)	1300-1000	Alcohols, carboxylic acids, ethers, esters	C-O
747(m, s)	680-860	Aromatic	C-H

The alcohols and phenols in the pyrolysis liquid are specified by the broad absorbance peak of O-H stretching vibration of 3353 cm⁻¹ in the range between 3500 and 3200 cm⁻¹ whereas carboxylic acids are found in the medium absorbance peak of O-H of 2929 cm⁻¹ wavenumber in the range of 3300-2500 cm⁻¹. The presence of ketones and aldehydes is characterized by the strong and medium narrow absorbance peaks of C=O between the frequency range of 1625-1750 cm⁻¹. The alkanes are indicated by the medium narrow absorbance peaks of C-C vibrations of 1454 and 1365 cm⁻¹. The possible absorbance peak of C-O of wave number 1021 cm⁻¹ in the range of 1000-1300 cm⁻¹ represents alcohols, carboxylic acids, ethers and esters. The aromatic compounds are categorized by presence of the absorbance peak of 747 cm⁻¹ wavenumber in 680-860 cm⁻¹ range. The similar functional groups and bonds have been found in the bio oil derived from rice husk and sunflower pressed bagasse in the literature (Islam and Ani 1998; Gercel 2002; Zhou, Yang et al. 2012).

The qualitative analysis of pyrolysis liquids are carried out by GC-MS using acetone as a solvent and chromatograms are shown in fig. 7. Test 1 chromatogram yields about 10 number of peaks with different retention times and searching in the library the compounds identified are acetone, benzene, 3-methyl pentane, 2,2-dimethylbutane, 3,5-dimethyloctane, 2-methylhexanoic acid, 1,2- benzene dicarboxylic acid whereas test 2 chromatogram gives one significant peak with retention time of 47.475 indicating the compound as 1,2- benzene dicarboxylic acid, Bis (2-ethylhexyl) phthalate. The similar compounds are found in the bio oil in the literature (Mullen and Boateng 2008; Heo, Park et al. 2010). There may be more than hundred compounds in the bio oil but due to pyrolysis conditions especially reactor temperature ($T_b - T_f > 100^\circ\text{C}$) and long vapor residence time only few compounds are present in the liquid resulting in lower bio oil yield.

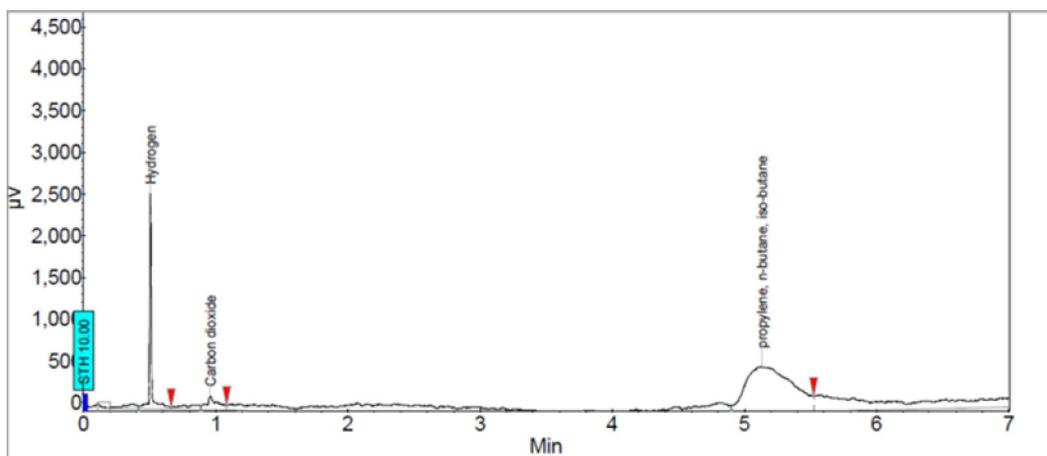


Figure8: Test 2 GC Chromatogram of exhaust gas

Gas Chromatograph (GC) is used to analyse the exhaust gas diluted with inert nitrogen gas and analysis are shown in the fig. 8. It is observed that there are three peaks of gas chromatograph indicating significant peak of H₂ and minor peak of CO₂ gas and third one of n-butane, iso-butane. The previous studies (Radlein 2002; Duman, Okutucu et al. 2010) on fast pyrolysis shows that CO₂ is the product of primary pyrolysis whereas CO and CH₄ are produced from secondary cracking reactions of volatiles followed by the reduction of CO₂ as well as secondary cracking also yields H₂ and CO.

5. Conclusions

1. Fluidized Bed Fast Pyrolysis System has been designed, fabricated and installed to carry out thermal conversion of Pakistani Cotton Salk into bio oil. About 14-21 % of pyrolysis liquid is obtained at bed temperature of 366-537°C. The yield of pyrolysis liquid can be increased by decreasing the residence time of volatiles from reactor outlet to condenser inlet that can be reduced by removing two intermediate cyclone separators.

2. The yield of pyrolysis liquid can be increased by achieving the temperature gradient of the reactor about 10°C to complete the fast pyrolysis reaction and to avoid more water contents in the bio oil.

3. The yield of pyrolysis liquid can be increased by achieving uniform temperature in the outgoing streams from reactor to the condenser inlet to avoid secondary reactions responsible for more H₂ gas production.

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