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EXPERIMENTATION OF BIOMASS PYROLYSIS OF ORGANIC WASTE USING A VACUUM PYROLYSIS REACTOR

Ehsanul Kabir[1,*], Md. Abul Kalam Azad[2] and Sudip Kumar Pal[3]

[1,3]Department of Civil Engineering, Port City International University, Chittagong, Bangladesh
[2]Innovative Consultant Ltd, Chittagong, Bangladesh
[1,*]ehsanul_kabir@live.com; [2]akazad.civil@gmail.com;[3] sudip94@yahoo.com

Abstract- *This research presents the experimental results of biomass pyrolysis obtained with a laboratory-scale pyrolysis system consisting of a directly heated vacuum reactor, inserted in a high temperature furnace. Waste wood (in the form of wood chips and sawdust) and rice husk has been used as feedstock. Slow pyrolysis experiments on the feedstock have been conducted to determine the effect of pyrolysis temperature, heating rate and particle size on the pyrolysis product yields. The experimental activity includes the analysis of the yield amount of untreated bio-oil for different feedstock and also the physical and chemical characteristics of the extracted liquid and char. The optimum process conditions for maximum liquid yield were also identified. Pyrolysis experiments were performed at temperatures between 240°C and 340°C with particle size of 0.5-20 mm. The highest liquid yield was obtained at a pyrolysis temperature of 340°C, wood chips as feedstock with a particle size of around 20 mm, with an average heating rate of 5°C/min in a 305 mm (12 inch) length reactor. The obtained yield of liquid was found to be 24.3% by mass. Basic physical characteristics of the extracted liquid obtained at optimum conditions were determined. Density and apparent specific gravity of the liquid yield were 1020 kg/m^3 and 1.022 respectively. The conversion of liquid to usable biofuel had not occurred as the 27/3 test results were negative. Clouding point of the extracted liquid was found to be approximately 0°C. The liquid was nonflammable and insoluble in water. The extracted liquid requires further treatment like moisture removal and removal of fine carbon particles before it's ready to be used as biofuel.*

Keywords: Biomass pyrolysis, organic waste, vacuum pyrolysis reactor, untreated bio-oil.

1. INTRODUCTION

In the current energetic outlook, one of the strategies pursued is the energy generation from renewable sources, in order to reduce the fossil fuels dependence and to rely on environment compatible technologies. In this context, the biomass pyrolysis process is considered as a viable fuel upgrading path, allowing the transformation of a solid biomass into an energy vector. This chance is particularly interesting for the current scenario, in which small and medium size conversion plant technologies could be integrated in a distributed energy generation model that is expected to increase its diffusion. In this perspective the biomass pyrolysis represents an alternative to improve innovative energy processes, in particular for small rural territories, looks promising for bio-oil production as fuel for electric power generation by direct utilization in conventional internal combustion engines or, after clean up and reforming stages, for innovative generation systems as fuel cells. [1] This work presents the experimental results coming from a vacuum biomass pyrolysis system and the assessment of its performance.

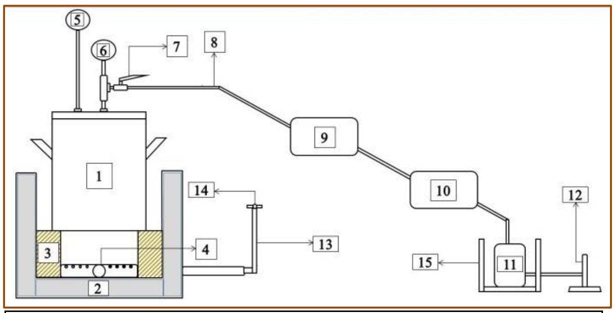
2. METHODOLOGY

The vacuum pyrolysis process in which the feedstock is heated from moderate to high temperatures produces considerable amount of bio-oil along with combustible gas or syngas. The production of bio oil is given most emphasis during this experimentation of pyrolysis process.

2.1 Experimental Setup

An experimental laboratory-scale apparatus for biomass pyrolysis has been designed for experimentation. The apparatus is designed for small scale slow pyrolysis process. It consists of a directly heated vacuum reactor operating at variable pressure. The reactor is set up within an external heating furnace capable of reaching temperatures up to 400^{0}C.

The reactor is inserted from above in the laboratory cylindrical furnace which is open on the top side. The control and acquisition devices are connected at the top of the reactor. There are condensers placed in series which are connected to the outlet and liquid collector and gas flaring devices are connected at the end. Also, flexible plastic pipes of small diameter are used to convey the liquid and gas produced from the reactor through the condensers and collectors.



LEGEND

1. Cylindrical Reactor
2. Furnace
3. Support
4. Gas Burner
5. Temperature Indicator
6. Pressure Indicator
7. Ball Valve
8. Outlet Tube
9. 1st Condenser
10. 2nd Condenser
11. Liquid Collector
12. Secondary Gas Outlet
13. Gas Supply Pipe for Furnace
14. Gas Control Valve
15. Ice-Bath

Fig.1: Schematic Diagram of Pyrolysis Equipment (not drawn to scale)

2.2 Feedstock Materials

Primary products of hemicelluloses and cellulose decomposition are condensable vapors (hence liquid products) and gas. Lignin decomposes to liquid, gas and solid char products. Extractives contribute to liquid and gas products either through simple volatilization or decomposition. [2]

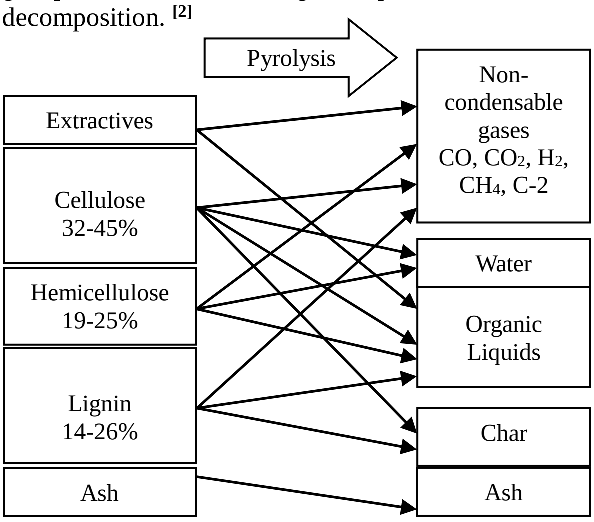


Fig.2: Simplified Representation of Biomass Pyrolysis

In the model, feed particle is considered to be spherical in shape. When the feed particle is exposed to medium temperature when being fed into the vacuum reactor, heating occurs immediately by conduction, radiation, and convection. The generated vapors diffuse through pore and out of particle to the bulk of the gas. Meanwhile, homogeneous secondary cracking occurs inside the porous particle and in the bulk of gaseous phase. Along with pyrolysis, particle shrinkage takes place, which may influence the transport phenomenon.

The experimentation was carried out with wood chips, wood dust or saw dust, and rice husk; each material individually used as feedstock. The wood chips and sawdust used in the experimentation were discarded wood particles and flakes form wood processing mills.

2.3 Preparation of Feedstock

It is necessary to have a relatively small particle for introduction to the reaction vessel adequately to exploit one or more of the heat transfer mechanisms. This ensures a high surface area per unit volume of particle. Another reason for small particles is the physical transition of biomass as it undergoes pyrolysis when char develops at the surface. Char has insulating properties that impede the transfer of heat into the center of the particle and therefore runs counter to the requirements needed for fast pyrolysis. The smaller the particle the less of an affect this has on heat transfer. [3]

But it also has to be noted that decrease in particle size leads to decrease of liquid yield because the residence time of the volatiles in the reactor is longer, favoring the cracking of hydrocarbons. More residence time of volatiles inside the reactor leads to cracking of heavier molecules (tar) into lower molecules at lower particle size ranges and it results in increase of gaseous product.

Size reduction of biomass however requires energy, and this in turn adds to the overall processing cost. As would be expected, the smaller the desired size the more expense added to the feedstock preparation costs.

Presence of free moisture affects the liquid yield of pyrolysis process severely by significant reduction of oil yield. The materials used for experimentation namely waste wood and rice husk contain a considerable amount of moisture. So, proper removal of surface moisture is essential for expected yields.

Therefore, the feedstock was prepared by the following way before experimentation:

(1) Cleaning the sample for any impurities or unwanted materials (e.g. grit).

(2) Reducing the size of feed particles. The wood chips were kept within 10-20 mm particle size and the sawdust and rice husk particle size was less than 1 mm.

(3) Oven drying the samples. During this experimentation, each batch of feed was oven dried for 24 hours at 1050C. The expected moisture content of feedstock was therefore expected to be less than 5%.

2.4 Experimental Parameters

The experiments were held within fixed parameters for different feedstock samples as described in Table 1.

Table 1: Experimental Parameters for Different Feedstock

Feed Material	Particle Size (mm)	Moisture Content %	Sample Weight (gm)	Residence Time (min)	Maximum Temp. (^{0}C)	Pressure (atm)
Wood Chips	10-20	<5%	700	75	340^{0}C	atm
Saw dust	< 1	<5%	1000	75	240^{0}C	atm
Rice husk	< 1	<5%	1000	75	315^{0}C	atm

2.5 Experimental Procedure

The feed particle is heated up rapidly to the reaction temperature when exposed to hot atmosphere in the vacuum bed by means of intensive conduction. As the particles heat up, hydro-cracking takes place and the material divides into liquid, gas and char. The hot liquid and gas come up through the conveying pipes as vapor. Produced pyrolysis vapor was passed through two sets of condenser tubes to quench into liquid and then collected into the liquid collector, which was also submerged in an ice-bath for better condensation. The uncondensed gases were flared into the atmosphere. The char product was pushed out from the reactor chamber manually after every complete experiment.

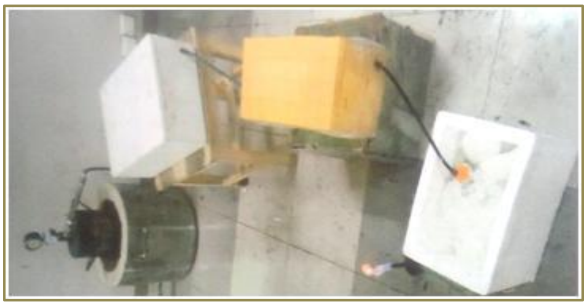


Fig.3: Actual Vacuum Reactor and Collection System

Finally, the liquid product was again condensed by ice and then filtered through simple filter papers to remove heavy condensate and impurities. The extracted liquid product was then allowed to rest for one day so the remaining fine char particles in the liquid settle at the bottom of the container and then relatively clean bio-oil was extracted.

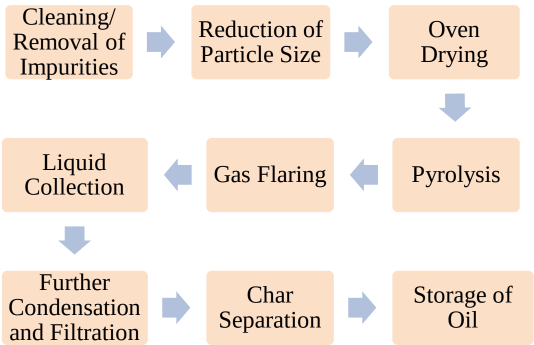


Fig.4: Flowchart of Experimental Procedure

2.6 Final Products

The bio-oil and the charcoal were sampled and stored. Quality and quantity of the product i.e. both bio oil and charcoal varied marginally due to different sources of feedstock.

2.6.1 Liquid Product

After separation of char, the extracted liquid is stored in sealed containers. The untreated bio oil is a dark black colored, highly viscous liquid with very strong odor. It's a high density liquid material contains a good amount of very fine suspended carbon particles.

2.6.2 Charcoal and Ash

The derived charcoals from wood chips are readily usable as fuel and need little processing because of the larger size of the charcoal. The char of ash derived from the sawdust and rice husk however need further processing before usage.

2.6.3 Secondary Gas or Pyrogas

The pyrogas consists of a number of combustible gases, such as Carbon monoxide, Methane, Hydrogen and minor quantities of other hydrocarbons. It also contains Carbon dioxide and may contain water vapor if it has not been previously condensed out of the stream.

3. RESULTS AND DISCUSSION

3.1 Experimental Results

Table 2: Process Parameters and Yield for Wood chips

Feed Particle	Feed Amount (gm)	Max Reactor Temp. (^{0}C)	Process Duration (min)	Avg. Heating Rate (^{0}C/min)	Liquid Yield (ml)	Solid Yield (gm)	Liquid Yield % by Mass	Solid Yield % by Mass	Gas Yield % by Mass
20 mm (SI)	700	340	75	5	170	148	24.3	21.1	54.6
10 mm (SI)	700	325	75	4.5	158	142	22.5	20.2	57.3

Table 3: Process Parameters and Yield for Sawdust

Feed Particle	Feed Amount (gm)	Max Reactor Temp. (^{0}C)	Process Duration (min)	Avg. Heating Rate (^{0}C/min)	Liquid Yield (ml)	Solid Yield (gm)	Liquid Yield % by Mass	Solid Yield % by Mass	Gas Yield % by Mass
0.5 mm	1000	240	75	3.1	227	150	23.4	15	61.6

Table 4: Process Parameters and Yield for Rice husk

Feed Particle	Feed Amount (gm)	Max Reactor Temp. (^{0}C)	Process Duration (min)	Avg. Heating Rate (^{0}C/min)	Liquid Yield (ml)	Solid Yield (gm)	Liquid Yield % by Mass	Solid Yield % by Mass	Gas Yield % by Mass
0.5 mm	1000	315	75	5	171	450	17.3	45	37.7

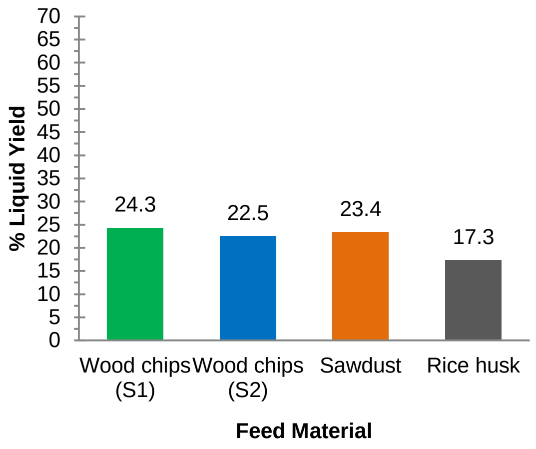


Fig.5: Variation of Liquid Yield for Different Feedstock

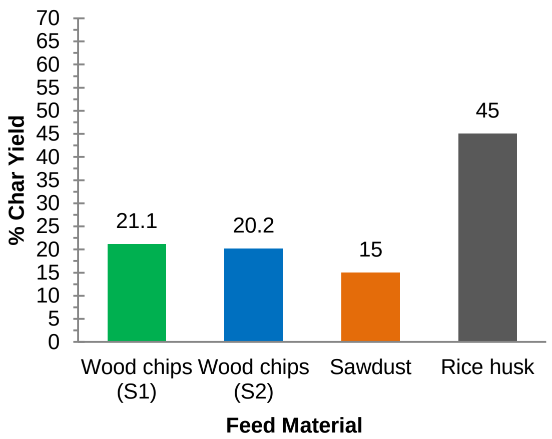


Fig.6: Variation of Char Yield for Different Feedstock

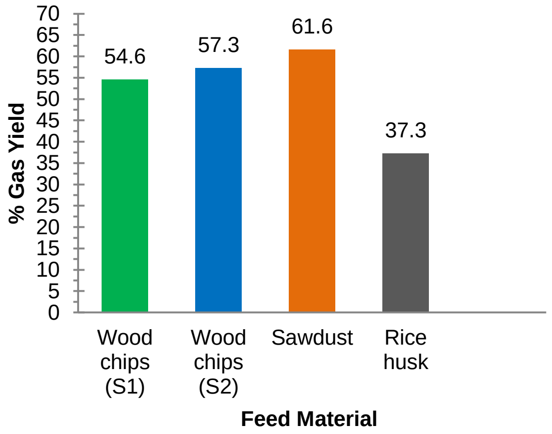


Fig.7: Variation of Gas Yield for Different Feedstock

3.2 Effect of Temperature

To a large extent, reactor temperature determines the yield of pyrolysis products. The heating system used in this project was able to provide maximum temperatures around 350^{0}C. Maximum temperatures differ for different feedstock, which could not be controlled as the heating was provided through natural gas.

3.3 Effect of Heating Rate

From the heating rate profiles of all feed materials it's apparent that,

(1) The heating rate is higher at first but keeps reducing after some intervals of time.

(2) It keeps reducing until it reaches a lowest value, at which the pressure or amount of secondary gas also starts to decrease.

(3) After reaching this point, the heating rate increases by some amount and ultimately becomes zero as the reactor reaches its maximum temperature.

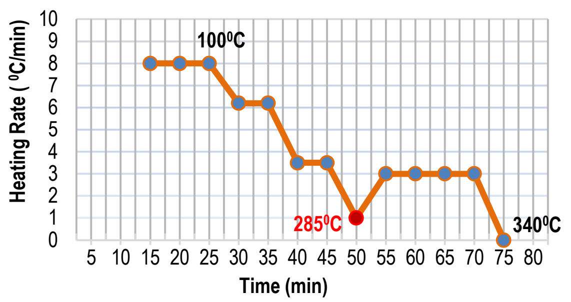


Fig.8: Heating Rate Profile for Wood chips (Sample 01)

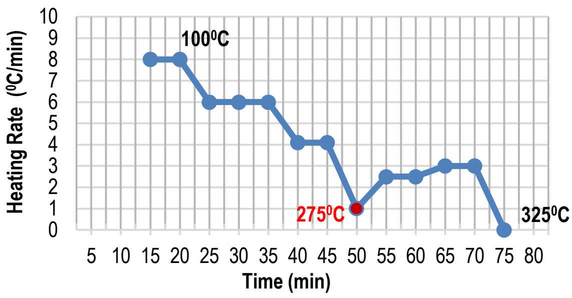


Fig.9: Heating Rate Profile for Wood chips (Sample 02)

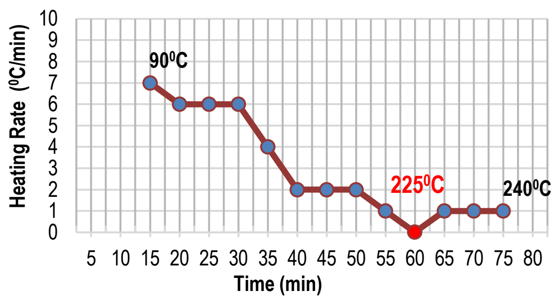


Fig.10: Heating Rate Profile for Sawdust

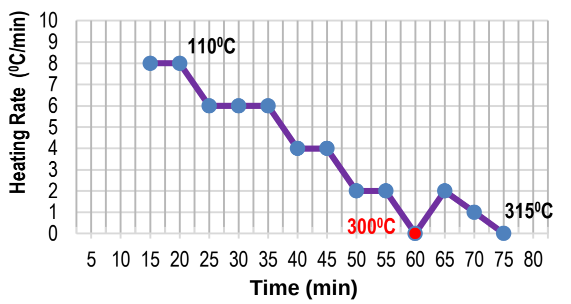


Fig.11: Heating Rate Profile for Rice husk

This behavior of heating rate and also the decrease of amount of secondary gas at the designated point indicate that the process of chemical cracking of material had been completed before the reactor reached its maximum temperature. So the particles have actually completed cracking at lower temperature than the reported maximum temperature of the process. This is significant for the comparatively lower yields and presence of high moisture in the extracted liquid. This factor is most significant for woody biomass with larger particles.

From the heating rate we can say that the hydro cracking process was completed at 285^{0}C for wood chips, 225^{0}C for sawdust and 300^{0}C for rice husk although the reported maximum temperatures of the process were 340^{0}C, 240^{0}C and 315^{0}C respectively.

3.4 Effect of Feed Particle Size

It was observed that the liquid yield increased as long as the particle size increased.

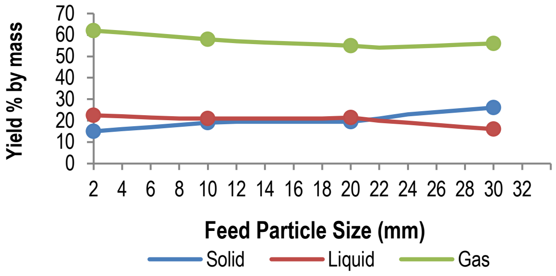


Fig.12: Relation between Yield Percentage and Feed Particle Size

This observation indicates that the lower density of feed with larger particles promote liquid yield, as the residence time is lower. Smaller particles for their long residence time promote more yields of char and gas. Feed with around 20 mm particle size gave maximum liquid yield. But once the particle size exceeds 20 mm, the void spaces are too large which hamper the circulation of heat. So the chemical cracking of particles remains incomplete, which results in very low yield of bio-oil. Also, it was observed by inspecting the wood shaving particles that char formation was on the outside only, as the particles in the middle of the reactor could not receive enough heat to transform and therefore, remained as wood, not char.

3.5 Properties of Extracted Liquid

The conversion of bio oil to usable fuel has been determined by the 27/3 test, which includes mixing 3 ml of oil sample with 27 ml methanol. If the solution remains relatively clear that means full conversion from bio oil to usable fuel has occurred. An unclear solution would indicate that the extracted fuel is not ready to be used as fuel and requires extensive treatment.

The clouding point test is used to determine the temperature at which the fuel is likely to plug the filter. It is an indicator of amount of paraffin in the fuel. Higher clouding temperature designates that the fuel is less suitable for low temperature operation. [4] The test was carried out by observing the temperature at which the liquid begins to "gel" when it's placed in a refrigerator.

Table5: Basic Properties of Untreated Bio-oil

Properties	Wood Chips	Sawdust	Rice Husk
Color	Dark brown	Dark brown	Dark brown
Density(kg/m^3)	1020	1030	1010
Apparent Sp. Gravity	1.022	1.032	1.012
Flammability	Non flammable	Non flammable	Non flammable
Solubility(H_2O)	Negligible	Negligible	Negligible
27/3 test results	-ve	-ve	-ve
Clouding point (^{0}C)	Approx. 0^{0}C	Approx. 0^{0}C	Approx. 0^{0}C

3.6 Optimum Conditions for Maximum Liquid Yield

The experimentations were carried out emphasizing on maximization of liquid yield for slow pyrolysis. Therefore the main objective of the study was to establish optimum conditions in which the vacuum reactor is able to give maximum liquid yield. Observing the experimental data, following facts were noted:

(1) Woody biomass was found to give more liquid yield in comparison to agricultural waste (rice husk). Chemical structure of woody biomass enables the chemical breakdown in the desired way for maximum liquid yield.

(2) Feedstock with higher particle size (wood chips) resulted in higher percentage of liquid than feedstock with smaller particle size (sawdust and rice husk) which resulted in more gas yield. [5] Although when the feed particle size exceeded 20 mm (wood shavings), pyrolysis process was not fully completed. The liquid yield was substantially decreased and extracted liquid was not qualified to include in the results.

(3) Oven drying the feed samples until the moisture content is close to zero resulted in a significant amount of increase in liquid yield than the air-dried raw feed sample from previous trials.

(4) Higher heating rate increases the liquid yield significantly.

(5) Lower residence time results in more liquid yield.

(6) Maximum temperature was found to be different for different feed materials due to the limitations of heating system. But liquid yield would increase with maximum temperature as long as it is around 400^{0}C.

Owing to the limitations of the heating system, it was difficult to assess some factors like proper time, proper heating rate, maximum temperature etc. Therefore, following conclusion for maximum liquid yield is drawn analyzing the experimentation results:

Table 6: Condition for Maximum Liquid Yield for SVPR

Feedstock Material	**Wood chips**
Feed particle size	15-20 mm
Oven drying temperature	105^{0}C
Oven drying duration	24 hours
Pyrolysis process duration	75 minutes
Ultimate reactor temperature	340^{0}C
Average heating rate	5^{0}C/min

The maximum liquid yield of 24.3% by mass was obtained at the optimum conditions. From the results it's apparent that the extracted liquid is not ready to be used as fuel as it is nonflammable. This is due to the presence of moisture in the oil. So, further treatment is required for the liquid which may include:

(1) Removal of suspended fine carbon particles

(2) Removal of moisture through distillations

(3) Further distillations for separating high density waste oil and low density bio-diesel.

It is difficult to separate moisture as normal distillations process is not adequate for the extracted liquid. Again, distillation for separating heavy and light oils requires advanced mechanisms. After separation, the heavier tar like oil which is better known as "Crude oil" is mostly used as additives for diesel or conventional fossil fuel. [6] The acquired "Bio-diesel" can be used directly or as fractional blends with conventional diesel.

3.7 Limitations of the System & Proposals for Further Improvement

The system used for the experimentation on pyrolysis was small laboratory scale equipment and thus had many limitations. Thus, following steps should be taken into consideration for further development of the project:

(1) From many studies, it is familiar that the optimum temperature for pyrolysis is approximately 450^0C while the system was able to provide a maximum temperature of about 350^0C. [7] Low temperatures are a cause of less oil yield and poor quality of oil. Increasing maximum temperature capacity would yield better results.

(2) As shown before, low and inconsistent heating is one of the major causes for poor quality of bio-oil. Low heating rate means more residence time which yields unsatisfactory results in pyrolysis project. Heating rate has to be improved for better results.

(3) The condensation of vapors was not done properly as the series ice-box condensers were not able to provide adequate condensation. A better system for condensation of vapors would result in more yield of bio-oil.

(4) The pressure meter was not able to give more than one reading as the oil clogged its inlet hole.

(5) The resultant liquid contained a large amount of suspended carbon particles or char because of the absence of a char removing system from the vapor. Presence of char decreased the oil quality by a large amount. Installation of a "Cyclone" system may be able to counteract this problem.[8]

(6) Absence of gas measuring equipment hampered the accuracy of the results as the calculation for gas yield percentage was done by subtracting the sum of liquid and solid yield from total mass of feedstock. Installation of a gas meter will be able to remedy this problem.

(7) The gas was flared using a gas burner. Therefore the secondary gas which had good calorific value was wasted for absence of a storage system. A gas storage system would not only utilize the pyrogas, but also help for further study of its properties.

4. CONCLUSION

The overall agreement between model predictions and experimental results is reasonably satisfactory. The simulation code, based on a thermo-chemical equilibrium approach, can then be considered as an useful tool in the design of biomass thermal conversion processes lasting long enough to achieve (or to approach) equilibrium conditions. However, the computed reaction product composition is representative only of processes where the residence time is long enough to establish thermodynamic equilibrium conditions. This would be the typical case of some continuous real reactors (e.g. fixed or fluidized bed type), while for other kinds of process (e.g. fast pyrolysis) the equilibrium conditions will be not achieved. Henceforth, this study must be considered as a preliminary one despite the satisfying results presented here.

Development and commercialization of pyrolysis oils will help meet the mandate in the demand renewable fuel, pyrolysis oils can also advance the Biomass Program's vision of a viable, sustainable domestic biomass industry that produces renewable bio-fuels, bio-products, and bio-power; reduces dependence on oil; provides environmental benefits including reduced greenhouse gas emissions; and creates economic opportunities across the globe. The applications of novel solid combined with pyrolysis fuels production, eventually will have a role in augmenting the petrochemical industry. The world's population growth is inexorably pushing us in that direction.

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