Determination of Optimum Condition for The Production of Rice Husk-Derived Bio-Oil by Slow Pyrolysis Process

by

Tan Chiang Jinn

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

MAY 2013

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CERTIFICATION OF APPROVAL

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

TAN CHIANG JINN

ABSTRACT

This research study focuses on production of bio-oil through slow pyrolysis of rice husk (RH) by varying heating rates while maintaining other reaction parameters: Feedstock size, reaction temperature and nitrogen flow rate (carrier gas). The optimum reaction condition for the slow pyrolysis of husk (RH) giving the maximum liquid yield is found to be slow pyrolysis at heating rate of 20 ℃/min, feedstock size of 0.25-0.50 mm, reaction temperature of 500 ℃ and nitrogen flow rate of 100 ml/min. Highest average liquid yield of bio-oil obtained is 35.38 wt%. The bio-oil produced at the optimum condition is also characterized and found that it basically has higher pH, viscosity, density value, elemental compositions and higher estimated HHV compared to bio-oil produced at other heating rates. GC-MS analysis proved that bio-oil produced at heating rate of 20 ℃/min has more aldehydes, acids, ketones and phenol compounds.

ACKNOWLEDGMENTS

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ABBREVIATIONS AND NOMENCLATURES

FESEM	Field Emission Scanning Electron Microscope
GC-MS	Gas Chromatography Mass Spectroscopy
HHV	Higher Heating Value
RH	Rice Husk
wt %	Weight Percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The availability of fossil fuels is limited, but the demand 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. Figure 1.1 shows the pattern of rising oil consumption in this current emerging world. Realizing on this fact, 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.

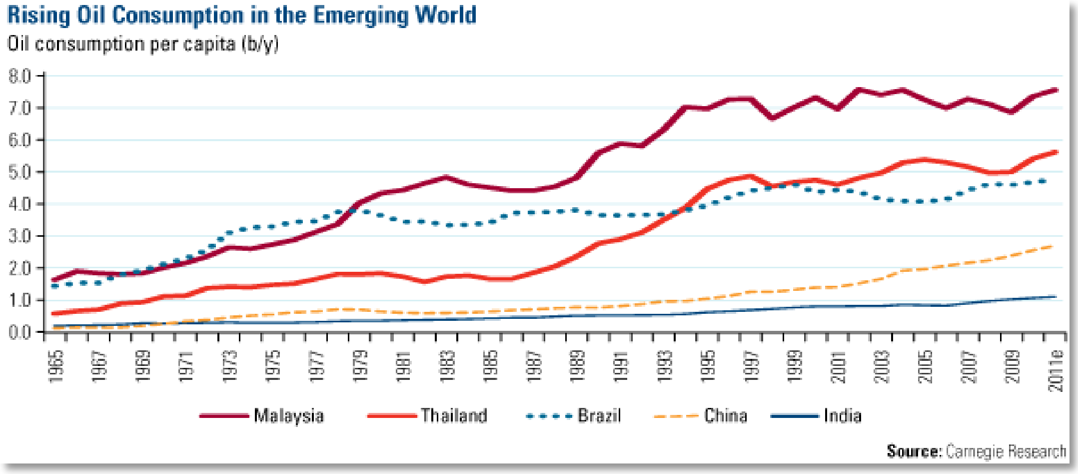


Figure 1.1: Rising Oil Consumption in the Emerging World

(Source: Advisor Analyst.com, 2011)

Among the possible renewable energy options, agricultural and forestry residues (generally called biomass residues) can be used as raw materials to generate energy. Biomass can generally be defined as any hydrocarbon material which mainly consists of carbon, hydrogen, oxygen and nitrogen, while sulfur is present in less proportions. According to Ragauskas et al. (2006), biomass is an abundant carbon-neutral renewable resource for the production of bioenergy and biomaterials. There are several methods to convert biomass into energy. As pointed out by Yaman (2004), biochemical and thermochemical conversion methods can be applied to biomass to make use of its energy potential for sustainable renewable energy option. Biochemical conversion methods are converting biomass into alcohols or oxygenated products by biological activity. On the other hand, thermochemical conversion methods such as pyrolysis, liquefaction, gasification and supercritical fluid extraction methods can be applied by converting biomass into energy.

Among all the thermochemical conversion methods, pyrolysis has attracted the most attention as highlighted by Dom ńguez, A., et al (2006). Pyrolysis is one of thermochemical processes, which convert the solid biomass in to liquid (bio- oil), gas, and char in absence of oxygen (Salleh, F., et al, 2011). Biomass pyrolysis converts 80–95% of the feed material to gases and bio-oil. The pyrolysis of biomass is a complex process due to differences in the chemical composition, particles structure within the biomass material, and operating condition. Natarajan. E, and Ganapathy Sundaram. E. (2009) noted in their research that the pyrolysis process is to be optimized to maximize the production of liquids (tar and bio-oil).

1.2 Problem Statement

There are various types of raw biomass materials can be used to undergo pyrolysis or other thermochemical conversion technologies to convert it into useful sustainable fuels. This kind of renewable energy source can be used as a substitute to current conventional fuels that is non-renewable and will be depleted one day in the future. Hence, investigation and researches have to be carried out on individual feedstocks in order to get more insight into them. Besides, the results obtained can also be used as a basis of comparison with bio-oil produced from pyrolysis of different operating conditions and reactor configurations using different feedstocks.

For this research project, the feedstock is chosen to be Rice Husk (RH). Rice husk is produced as a waste by-product from paddy production in large amount every year. Conducting research studies in converting rice husk from raw biomass into useful fuels is the best way rather than dispose this kind of agricultural waste by burning in the farmland every year. The rice husk waste produced can be utilized in some researches.

More attentions have been focused on fast pyrolysis for bio-oil production from rice husk but not slow pyrolysis. Hence in this project, the optimum condition for the production of rice husk-derived bio-oil by slow pyrolysis will be studied. The reaction parameter to be studied is heating rates and the bio-oil produced at different heating rates will be further analyzed. The heating rates of the slow pyrolysis will be varied in linear form to investigate its effects on the bio-oil liquid yield throughout pyrolysis as well as the product properties and composition. From the result obtained, reasoning and critical analysis will be done in attempt to explain the findings. Lastly, the optimum condition that will produce highest liquid yield of bio-oil and conversion will be determined.

1.3 Objectives

There are several relevant objectives that the author wishes to achieve in this research project. These objectives are:

1. To produce bio-oil from Rice Husk (RH) by slow pyrolysis process at different heating rates.
2. To discover the optimum operating conditions and parameters for the semi-batch reactor in UTP to produce highest liquid yield of bio-oil and conversion.
3. To analyze the bio-oil produced at optimum condition in terms of its physical properties and chemical composition.

1.4 Scope of Study

Pyrolysis of various biomass feedstocks has been greatly researched. However, most of the works done focus on the fast pyrolysis method while actually slow pyrolysis could be one of the ways to produce bio-oil, too. Besides, the reactor configuration used in the pyrolysis process is of larger scale (fluidized bed reactor) compared to the tube furnace reactor (semi-batch) reactor in UTP, with very few works focus on the influence of reaction parameters on the liquid yield and conversion.

Hence in this study, RH will be subjected to slow pyrolysis reaction to produce bio-oil. The operating conditions of the slow pyrolysis will be varied on different heating rates to investigate their effects on the bio-oil yield and conversion. The other 3 reaction parameters that remained constant are feedstock size, reaction temperature and nitrogen gas flow rate in the reactor. The responding aspect being studied is the liquid yield of bio-oil and conversion. Besides, the optimum condition giving the highest yield of bio-oil will be determined and the bio-oil produced at this optimum condition will be characterized for its physical properties and chemical composition. From the result obtained, reasoning and critical analysis will be done in attempt to explain the findings.

1.5 Feasibility of Project Within the Scope and Time Frame

This project is feasible within the scope identified and the time allocated. The first half of this project will be focusing on the through literature review of the related researches to see the areas of improvement and to obtain the basic understanding and knowledge about the project. After that, planning will be done in order to determine the subjects to be studied, the way to conduct experiment and the results to be collected from the experiment, with reference to the research journals studied. The second half of the project mainly focuses on conducting experiments and collected results and data. These results collected will then be analyzed and investigated critically.

In terms of scope of study, the project is feasible to be carried out in UTP as it has the required apparatus and equipment to run the pyrolysis reaction. The main equipment such as the reactor (tube furnace reactor) and characterization equipment are available. Furthermore, rice husk from the paddy farmland and methanol as a solvent for cleaning the reactor and apparatus can be easily obtained as well. The scope of study chosen is also similar to other related researches done. So, it is relevant and can be used to compare directly with other literature readings.

As such, this research project is feasible within the time frame and the scope of study. Strategic planning on the execution is needed for this research project to be completed on time and successfully.

CHAPTER 2
LITERATURE REVIEW / THEORY

2.1 Bio-Oil

Bio-oil, as shown in Figure 2.1, which is also known as pyrolysis oil or bio-fuel oil was produced by pyrolysis process without the additional of oxygen. According to Xiu et al (2012), bio-oil is a complex organic mixture with high oxygen content, containing more or less solid carbon particles, and its color is usually dark brown or black, having a strong pungent smell. The pyrolysis bio-oil is complex mixtures of oxygenated aliphatic and aromatic compounds (Meier et al, 1999).

Pyrolysis bio-oil consists hundreds of organic specific groups which include organic acids, esters, alcohols, ketones, aldehydes, phenols, aromatic hydrocarbons, nitrogen compounds, furan and furodrosugars (Abu Bakar et al, 2012).

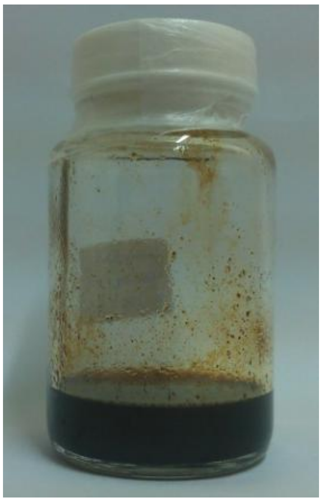


Figure 2.1: Bio-oil

Bio-oil has high potential to become substitute for fossil fuels as it can produce heat, electricity, and chemicals. However, Iliopoulou et al. (2012) studied that raw bio-oil that has not been upgraded yet has several undesired characteristics such as high water and oxygen content, corrosive, instable under storage and heating conditions, high viscosity, low calorific value and immiscible with petroleum fuels. The main reason for the instability of bio-oil is due to the presence of oxygenated compound in the bio-oil, they tend to further react during storage phase or in high temperature condition (Gopakumar et al., 2011). As a result of the instability, the bio-oil aging is occurring, causing the viscosity of the bio-oil to increase. This instability can actually be minimized or decreased either through the stabilization process after pyrolysis through upgrading methods such as emulsification with diesel oil (Jiang and Ellis, 2010), addition of antioxidants and solvents (Udomsap et al, 2011 and Wanasundara et al, 2005) and etc.

2.2 Biomass Rice Husk

Among the various biomass species, rice husk is a potential source of energy and value-added byproducts of the rice milling industry (Sharma and Rao, 1999). In the recent years, utilization of agriculture waste and residues (for instance, rice husk) for energy generation has gained a lot of attention. Rice is one of the major food crops in the world. The production of rice husk generates a great amount of waste in the world, namely rice husk. According to Bharadwaj et al. (2004), rice husk is the outer covering of paddy of paddy that produces approximately 20% of the annual gross rice production in the world, which currently stands at 672 million tons (Source: GeoHive, 2011). Rice husk is produced in the first step in the milling process when the husk is removed from the grain in the husking stage of the rice mill. Rice husk usually ends up being burned in open air, therefore causing environmental pollution and wastes the various biomass resources (Rafiee, et al., 2012).

Generally, rice husk is a cellulosic material, consisting of about 20-35% cellulose, 15-30% hemicelluloses, 5-10% lignin, and some materials. Rice husk is a renewable source of energy, with a high calorific value of about 4,260 kcal/kg on a dry basis.

Rice husk is characterized by low bulk density and high ash content (18–22% by weight). The large amount of ashes generated during combustion has to be continuously removed for a smooth operation of the system (Bharadwaj et al., 2004). A summary of the proximate analysis, ultimate analysis, calorific value and moisture content of rice husk are given in Table 2.1.

Table 2.1: Composition and Properties of Rice Husk

Characterization Parameters	Natarajan et al, 2009	Heo et al, 2010	Salleh et al, 2011	Fu et al, 2011	Guo et al, 2011	Abu Bakar et al, 2012
Proximate Analysis (wt%)						
Volatiles	70.6	78.8	79.70	61.23	60.98	68.25
Fixed carbon	2.97	-	3.38	14.95	14.46	16.92
Ash	17.09	11.9	12.31	17.09	12.26	14.83
Moisture content	9.45	9.3	8.27	6.73	12.30	8.43
Ultimate Analysis (wt%)						
Carbon (C)	50.45	53.5	38.42	50.47	40.00	39.48
Hydrogen (H)	6.58	7.0	5.09	6.85	5.03	5.71
Nitrogen (N)	1.49	1.4	1.01	0.59	0.53	0.665
Sulfur (S)	0.23	-	-	-	0.13	0.025
Oxygen (O)	41.46	38.1	55.48	42.09	29.75	54.12
Calorific Value (kJ/kg)	19807	-	14380	-	14570	17340

Somehow the chemical constituents of rice husk have been found to vary from sample to sample. Any of the differences in type of paddy, crop year, climatic and geographical conditions, soil chemistry, sample preparation, fertilizers used in the paddy growth or method of analysis could be a reason for this variation (Genieva, 2008).

2.3 Slow Pyrolysis

According to Natarajan, E. and Ganapathy Sundaram, E. (2009), biomass pyrolysis converts essentially 80–95% of the feed material to gases and bio-oil. The pyrolysis process converts biomass into high energy content biofuels, in the absence of oxygen/air, leading to the formation of solid (charcoal), liquid (tar and other organics) and gaseous products (Salman Zafar, 2012).

Pyrolysis can be divided into two types; fast pyrolysis and slow pyrolysis. These two pyrolysis reactions are different mainly in terms of heating rates and maximum reaction temperatures (Brown et al., 2011). Slow pyrolysis heating rates are typically below 100 K/min whereas fast pyrolysis can achieve rates exceeding 1000 K/min. Reaction temperatures are about 300 ℃ and 500 ℃ for slow and fast pyrolysis respectively. Slow pyrolysis requires several minutes or even hours, while fast pyrolysis is complete in as little as two seconds. This difference in time results in dramatic differences in product distributions: slow pyrolysis generates primarily gas while fast pyrolysis generates primarily bio-oil. This is because in slow pyrolysis, the vapor residence time is much longer than that of fast pyrolysis. As a result, the vapors undergo secondary cracking reaction, yielding more gas and less liquid. Whereas the short vapor residence time in fast pyrolysis and the rapid quenching of those vapors to produce bio-oil has greatly increases the liquid (bio-oil) yield. Table 2.2 summarizes the difference between fast and slow pyrolysis.

Table 2.2: Comparison between Slow and Fast Pyrolysis

(Source: The Pyrolysis of Sorghum Biomass webpage, 2011)

Type of Pyrolysis	**Heating Rate (K/min)**	**Reaction Temperature (℃)**	**Residence Time**	**Liquid**	**Char**	**Gas**
Slow Pyrolysis	< 100	300	Several minutes/hours	30%	35%	35%
Fast Pyrolysis	>1000	500	Few seconds	75%	12%	13%

CHAPTER 3
METHODOLOGY

3.1 General Research Methodology

1st Stage: Project Planning Phase

Problem identification and the significance of the project based on the current issue are determined. The optimum condition for the production of rice husk-derived bio-oil by slow pyrolysis is determined based on the difference of heating rates while other reaction parameters are remained constant.

2nd Stage: Characterization of Bio-oil and Slow Pyrolysis Studies

Literature review is conducted to have a basic understanding on biomass for slow pyrolysis reaction that had been published. Comparable studies between different types of operating conditions are conducted. Besides, characterization and component of the bio-oil is analyzed.

3rd Stage: Screening and Identification Phase

Basis for the selection criteria are outlined. In the experiment, biomass which is rice husk was obtained from local supplier. The rice husk is undergoes pre-treatment before further slow pyrolysis process. The pre-treatment of rice husk is done by grinding and sieving the rice husk into desired particle size. The rice husk sample is then put into oven overnight to remove the moisture and ready for use on next day.

4th Stage: Experiment Design

Decision on the equipments and chemicals involved for the project with clear view of methodology is conducted. Equipments will be identified specifically for analyzing product and chemical purposes.

5th Stage: Comparative Study

Statistical analysis and properties testing are conducted. The temperature difference data and the time when first drop of bio-oil produced are recorded manually while running the pyrolysis. After producing the bio-oil at different heating rates, the liquid yield and conversion are calculated. The bio-oil is then stored in glass bottle in refrigerator for further analytical testing purpose.

3.2 Research Methodology

The methodology for conducting this research project is mainly exploration and discovery. The approach needed is to carry out this research through hands-on experimental research on slow pyrolysis process.

Literature review firstly has to be done to have a firm and clear basic understanding about the background of the project and the related researches/works that were done previously to further plan the experimental procedure. Using the information gained from the journals reviewed that act as a base for this project; the slow pyrolysis process experiments will be conducted by using different configuration and setup of the equipment in order to produce bio-oil from rice husks as raw materials.

In order to conduct the experimental works, the author has to plan well the experimental procedure and taking into consideration the facilities and equipment available in UTP for the research project to be conducted.

Analyzing the experimental data is the next step to discuss the outcome of the projects. The results obtained from this research using different configuration and the setup of the equipment in the pyrolysis process to produce bio-oil from rice husk (RH) can be used as a basis of comparison with other literature results available. The results can hence, further enhance the research and development of slow pyrolysis of RH. The planning of this study such as project activities, Gantt chart and key milestones are shown in the Appendices.

Lastly, the author has to make conclusion by discuss the outcome of the results obtained and discussions in terms of statement. Figure 3.1 shows the flow of research methodology.

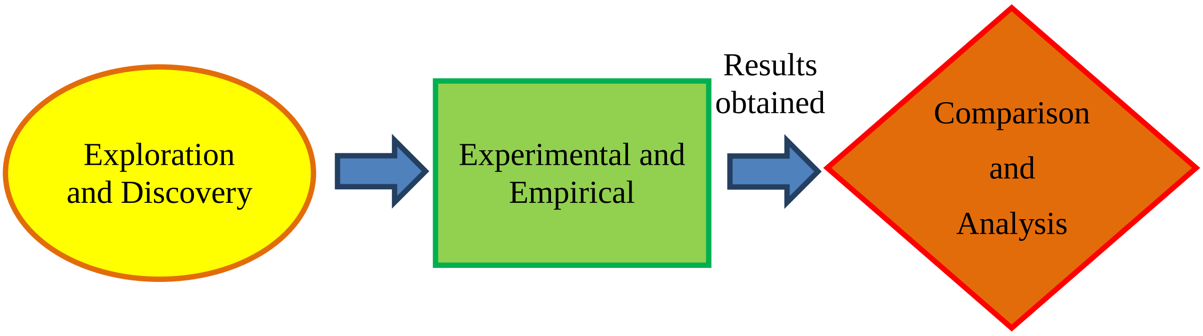


Figure 3.1: The Flow of Research Methodology

3.3 Experimental Procedure

The parameters are selected based on the literature review findings on most of the journals and articles. The particle size is chose to be 0.25-0.50 mm from three ranges of sizes: <0.25 mm, 0.25-0.50 mm and 0.50–0.71 mm. As for the reaction temperature, the optimum condition for pyrolysis process is around 500˚C. This is reported on the majority of the journals reviewed. Therefore, the level of the reaction temperature for this experiment is decided to be 500˚C. The nitrogen gas flow rate is chose to be 100 ml/min as it is suitable to be used in semi-batch reactor available in UTP. The experiment is decided to be run with heating rate of 5, 10, 15 and 20˚C/min as the heater available in the lab can only operate within this range.

3.3.1 Determination of Optimum Heating Rate

1. Conditions are remained constant at temperature of 500˚C, particle size 0.25-0.50 mm and nitrogen flow rate of 100 ml/min.

   **Note: The condition is assumed to be the best by referring to the journals and literature reviews. So the condition is chosen for the best condition for the determination of optimum heating rate that will give maximum liquid yield of bio-oil. The heating rates will be manipulated at 5, 10, 15 and 20 ℃/min.*
2. The borosilicate tube’s weight is recorded and the 15 g of rice husk sample is poured into the tube.
3. The experiment is set up and nitrogen gas is purged for 5 minutes at 500ml/min before the experiment.
4. The temperature of the heater and thermocouple is recorded every 2 minute.
5. The time of the first droplet of liquid yield first observed is recorded and the experiment is stopped when there are three constant readings of thermocouple readings or there are no more reactions.
6. The condenser before and after pyrolysis are weighed to calculate the liquid yield.
7. The bio-oil is collected for characterization.

3.3.2 Pyrolysis of Rice Husk into Bio-Oil

1. The rice husk sample is prepared in borosilicate tube as shown in Figure 3.2. Approximately 0.6g of glass wool is placed above the rice husk sample to avoid the rice husk from dispersed out of tube during pyrolysis reaction inside the furnace.

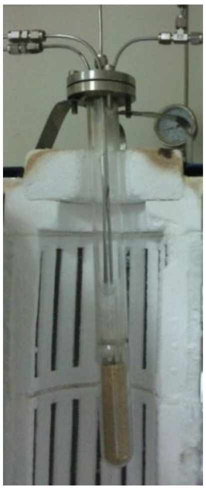


Figure 3.2: Preparation of Rice Husk Sample in Borosilicate Tube

2. The experiment is setup and nitrogen gas is purged for 5 minutes at 500 ml/min before the experiment.
3. The pyrolysis temperature is set at 500 ℃ and nitrogen gas flow rate at 100 ml/min.
4. The temperature of the heater and thermocouple is recorded every 2 minutes.
5. The time of the first drop of bio-oil formed is recorded.
6. When the temperature of the heater and thermocouple reached stable condition, the experiment is stopped after 10 minutes and let the reactor to cool down.
7. The condenser is weighed to calculate the liquid yield.
8. The bio-oil is collected for further characterization.
9. Steps 1-7 are repeated until enough bio-oil is produced (16 runs).
10. 16 runs will be based on 4 different heating rates: 5, 10, 15 and 20 ℃/min. Hence, 4 runs will be based on 5 ℃/min, 4 runs based on 10 ℃/min, 4 runs based on 15 ℃/min and 4 runs based on 20 ℃/min.

3.4 Raw Materials / Chemicals Needed

In the experiments that are going to be conducted, several raw materials and chemicals are needed. There are:

- i. Rice husk
- ii. Methanol – To clean up and dry the equipment and apparatus after experiment
- iii. Nitrogen flow – Used in the reactor as inert gas flow

3.5 Equipment

3.5.1 Reactor

Tube furnace reactor will be used to carry out the slow pyrolysis of rice husk. The layout of the reactor is shown as in Figure 3.3.

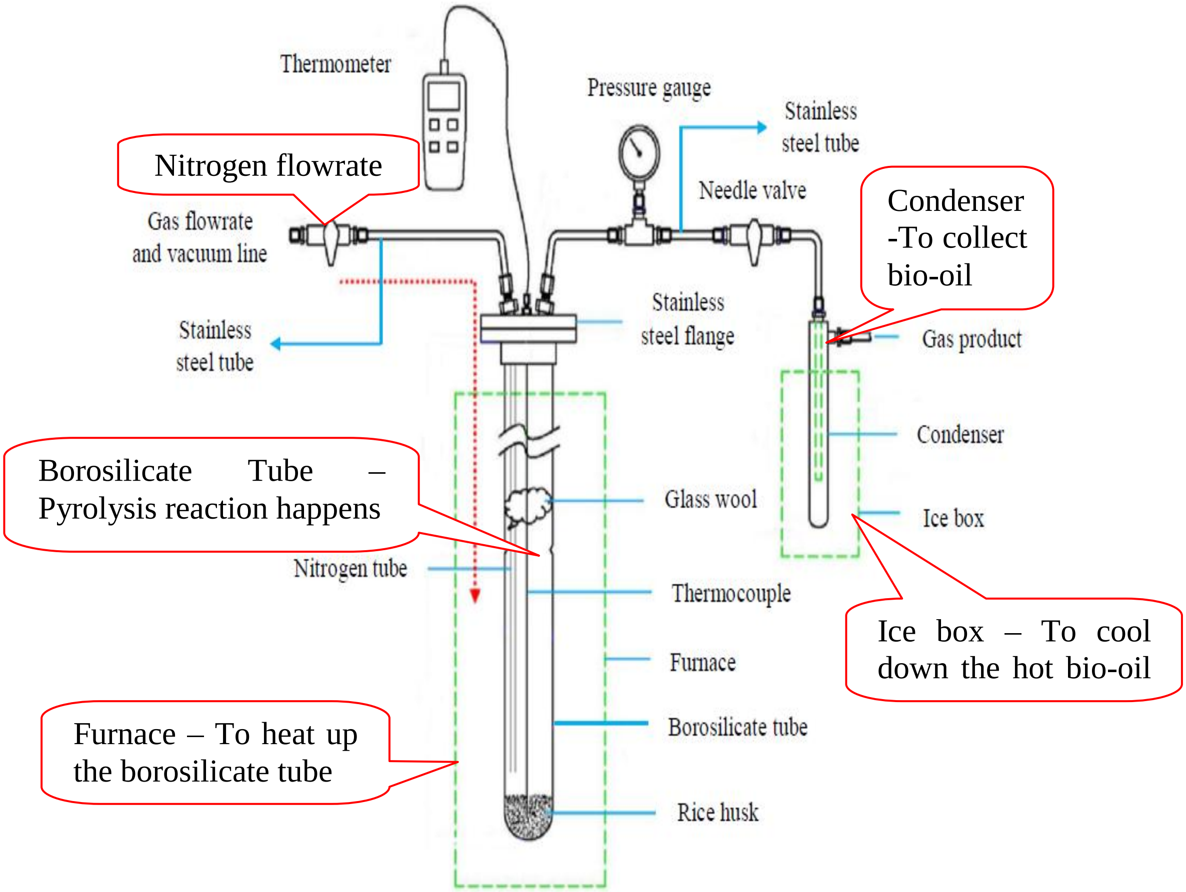


Figure 3.3: Schematic Diagram of Tube Furnace Reactor

Figure 3.4: Tube Furnace Reactor

Components of Tube Furnace Reactor (Figure 3.4) include:

1. Semi-batch Reactor (Lenton Thermal Designs with Split Tube Furnace)
2. RS 1319A K-Type Thermocouple
3. KI Key Instruments Nitrogen Flow Controller
4. Coleman Thermozone Insulator
5. Borosilicate Tube

Operating procedures of the reactor:

The raw material rice husk has to be pulverized, sieved and meshed into smaller size of particles before putting into the reactor for slow pyrolysis. Nitrogen gas is purged into the reactor for 5 minutes at 500 ℃/min to remove all the oxygen. After that, the slow pyrolysis process can be conducted by setting the desired temperature of the reaction and the flowrate of nitrogen gas. The reaction gas product will be collected in a gas bag and the liquid will be collected in a condenser. The condenser is stored in ice bath to condense the hot bio-oil.

3.5.2 Grinder

Figure 3.5: FRITSCH Cutting Mill Grinder

The grinder, FRITSCH Cutting Mill as shown in Figure 3.5, is been used to grind the raw rice husk sample into smaller sample size.

3.5.3 CISA Sieve Shaker

Figure 3.6: CISA Sieve Shaker

After grinding process, the grinded rice husk will be put into the sieve shaker (Figure 3.6) to obtain desired feedstock size (0.25-0.5 mm).

3.5.4 Oven

After pre-treatment of rice husk into desired feedstock size, they will be stored in the oven to remove the moisture content.

3.6 Characterization and Properties Measurement

Characterization of raw material (rice husk) is necessary as it can be used as a basis to compare with other types of biomass. Table 3.1 shows the lists of the equipments that will be used for sample product characterization:

Table 3.1: Testing Parameters

No	**Testing Parameters**	**Instruments**	**Methods**	**Sample Size**	**Venue**
1	Viscosity	Brookfield Viscometer	ASTM D445	13 mL	Block P
2	Higher Heating Value	IKA C5000 Bomb Calorimeter	-	0.4 g	Block 4
3	Density	Anton Paar Density Meter DMA 4500 M	ASTM D4052	2 mL	Block P
4	Morphology analysis	Field-Emission Scanning Electron Microscope (FESEM)	-	Vial tube	Centralized Analytical Laboratory
5	CHNS	LECO 932 CHNS Analyzer	ASTM D5291	2 mL	Block 4
6	Proximate Analysis	Perkin Elmer Pyris 1 TGA	-	3 g	Block P
7	Chemical Composition	Agilent GC/MS 5975 C	-	Vial tube	Centralized Analytical Laboratory
8	pH	EUTECH Instruments pH 510 pH/mV/ ℃ Meter	-	Few drops	Block 5

3.7 Justification of the Selection of Parameters

The bio-oil was produced from slow pyrolysis reaction at four different heating rates with other parameters chosen to be remained constant throughout the pyrolysis reaction. These parameters are chosen and determined by author based on certain valid justification. These parameters are set to be constant as they are not the factors under investigation, which are the particle size of the rice husk used in pyrolysis, temperature of pyrolysis reaction and the nitrogen gas flow rate into reactor. The manipulated variable for this project is the different heating rates at 5, 10, 15 and 20 ℃/min.

3.7.1 Feedstock Size

Three different set of sample sizes are used to study the properties of rice husk in order to select which range of particle size would result in the optimum condition of bio-oil. The result of the properties is obtained from a Master student, Dang Khanh Vi, who is currently working on her Master thesis in Universiti Teknologi PETRONAS (UTP) by using the same raw material rice husk. The result is shown as in Table 3.2 below:

Table 3.2: Sample Analysis at Different Feedstock Sizes

Size (mm)	**Moisture (wt%)**	**Combustible matter (wt%)**	**Ash (wt%)**	**Heating value (J/g)**	**Carbon (%)**	**Hydrogen (%)**	**Nitrogen (%)**	**Sulfur (%)**	**Oxygen (%)**
0.00-0.25	8.94	78.46	12.60	15238	37.88	5.34	1.41	0.06	55.31
0.25-0.50	8.79	79.95	11.26	15224	39.54	4.75	0.88	0.20	54.63
0.50-0.71	9.11	80.36	10.53	15271	39.12	4.58	0.93	0.20	55.17

Based on Table 3.2, it is shown that the feedstock size with the range 0.25-0.50 mm has the lowest moisture content and oxygen content. Both of these parameters should be showing low values because these are two major factors that affect the stability of bio-oil produced. Higher moisture content and oxygen content will lead to lower stability of bio-oil. Hence, the author has selected the feedstock size with the range 0.25-0.50 mm throughout the experimental works.

3.7.2 Temperature of Pyrolysis Reaction

The temperature of pyrolysis usually falls within the range of 400-600 ℃. According to Natarajan et al (2009) and Chen et al (2010), the optimum temperature of 500 ℃ for pyrolysis process gives maximum liquid yield of bio-oil at 34.17 wt% to 42 wt%. Hence, the temperature level that is chosen for this project is 500 ℃.

3.7.3 Nitrogen Gas Flow Rate

Nitrogen gas flow rate is directly related to the vapor residence time. As higher flow rate is used, the vapor residence time is shorter. Demiral and Şensöz (2006) reported that the high flow rate of carrier gas will cause the decreasing of the total yield. Similar effect was found by Şensöz and Angin (2008). This is because shorter residence time at higher gas velocity will lead the volatiles to leave the condenser without effective condensation. Hence, the reaction will not completely react. Moreover, Pütün et al (2007) found out that the liquid yield decreased when the gas flow rate increased from 100 ml/min to 400 ml/min. Therefore, by considering the operating range of nitrogen gas flow rate of the tube furnace reactor is 100-500 ml/min, the author decided to choose nitrogen gas flow rate into reactor to be 100 ml/min.

3.8 Calculation of Yield and Conversion of Slow Pyrolysis Reaction

The liquid yield or bio-oil is the desired product from the pyrolysis process and the conversion of the biomass to liquid yield can be calculated using the formula below:

$$Yield_{bio-oil} = \frac{m_{bio-oil}}{m_{biomass}} \times 100\%$$

Meanwhile, the overall conversion of biomass into gas and oil products can be determined by using the formula:

$$Conversion_{\%} = \frac{Decrease\ in\ weight\ of\ reaction\ mixture}{m_{biomass}} \times 100\%$$

Apart from that, the gas yield and residue yield can be calculated using the formula:

$$Yield_{gas} = Conversion - Yield_{bio-oil}$$

$$Yield_{residue} = 100\% - conversion$$

3.9 Pretreatment of Rice Husk (RH)

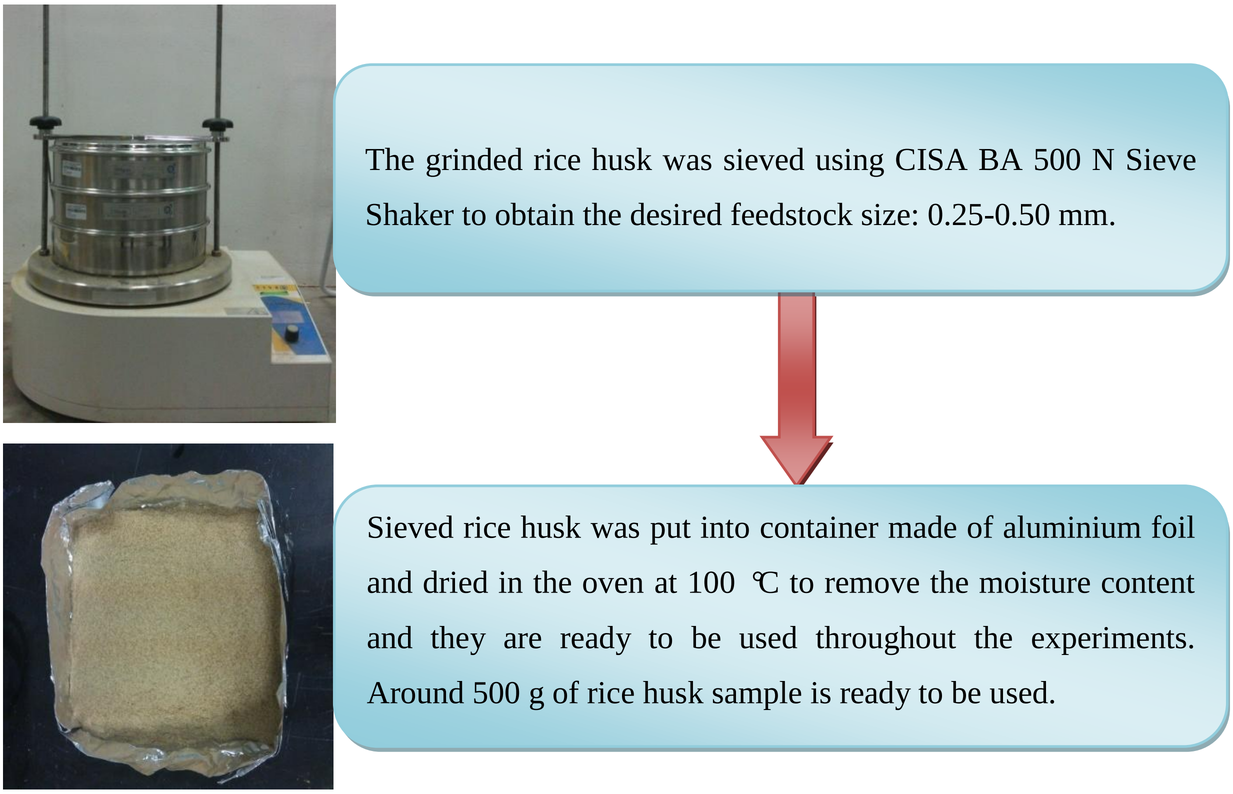
First of all, raw rice husk samples were obtained from local supplier. Before start-up experiment, the rice husk was pre-treated before they are grinded to smaller particle sizes. Figure 3.7 below shows the steps taken in treating the raw rice husk samples.

Raw rice husk samples were obtained from local supplier.

The rice husk is then grinded with FRITSCH Cutting Mill to smaller particle sizes.

After first grinding process, the rice husk has been reduced into smaller size but it still not reach at the desired particle size.

After few times grinding, the rice husk now has been reduced to desired feedstock size with the range of 0.25-0.50 mm.



The grinded rice husk was sieved using CISA BA 500 N Sieve Shaker to obtain the desired feedstock size: 0.25-0.50 mm.

Sieved rice husk was put into container made of aluminium foil and dried in the oven at 100 ℃ to remove the moisture content and they are ready to be used throughout the experiments. Around 500 g of rice husk sample is ready to be used.

Figure 3.7: Flow Chart Depicting the Pre-Treatment of Rice Husk

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Characterization of Rice Husk

The characterization of rice husk is done on its ultimate analysis, proximate analysis and higher heating value (HHV).

4.1.1 Ultimate Analysis

The ultimate analysis reports on the elemental composition of carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) and oxygen (O) by difference. The carbon, hydrogen, nitrogen, sulfur and oxygen content are shown in Table 4.1.

Table 4.1: Ultimate Analysis of RH

Characterization Parameter	Natarajan et al, 2009	Heo et al, 2010	Salleh et al, 2011	Fu et al, 2011	Guo et al, 2011	Abu Bakar et al, 2012	**This work**
Ultimate Analysis (wt%)							
Carbon (C)	50.45	53.5	38.42	50.47	40.00	39.48	**41.3400**
Hydrogen (H)	6.58	7.0	5.09	6.85	5.03	5.71	**3.5570**
Nitrogen (N)	1.49	1.4	1.01	0.59	0.53	0.665	**0.3675**
Sulfur (S)	0.23	-	-	-	0.13	0.025	**0.1485**
Oxygen (O)	41.46	38.1	55.48	42.09	29.75	54.12	**54.5870**

As compared to the literature readings, it shows that the ultimate analysis obtained in this work is quite similar with findings done by Salleh et al (2011) and Abu Bakar et al (2012). However, the ultimate analysis is actually depends on the source of raw biomass.

4.1.2 Proximate Analysis

The proximate analysis reports on moisture content, combustible matter and ash. The values of proximate analysis are shown in Table 4.2. Volatile matter and fixed carbon contents provided a measure of ease with which the biomass could be ignited, gasified and oxidized depending on the biomass utilization.

Table 4.2: Proximate Analysis of RH

Characterization Parameter	Natarajan et al, 2009	Heo et al, 2010	Salleh et al, 2011	Fu et al, 2011	Guo et al, 2011	Abu Bakar et al, 2012	**This work**
Proximate Analysis (wt%)							
Volatiles	70.6	78.8	79.70	61.23	60.98	68.25	**77.13**
Fixed carbon	2.97	-	3.38	14.95	14.46	16.92	**2.12**
Ash	17.09	11.9	12.31	17.09	12.26	14.83	**12.25**
Moisture content	9.45	9.3	8.27	6.73	12.30	8.43	**8.50**

In this work, the volatile matter content is high with low ash content. According to Salleh et al (2011), the liquid yield obtained from the pyrolysis process increased with an increase of their volatile matter and with a decrease in ash content. Therefore, the author could make a prediction that the liquid yield obtained from this work will range around 35 wt% to 40 wt%

4.1.3 Higher Heating Value (HHV)

Due to testing equipment (IKA C5000 Bomb Calorimeter) breakdown, hence the author is not able to obtain the HHV of RH. However, based on literature review, HHV can be predicted based in the ultimate analysis (C, H, N, O, S values). According to Friedl et al (2005), the HHV of biomass can be estimated using the following formula:

$$\mathbf{HHV = 3.55C^2 - 232C - 2230H + 51.2C \times H + 131N + 20{,}600}$$

Hence, by substituting the ultimate analysis obtained into formula, the author can predict the HHV of rice husk is **16720 kJ/kg.** (≈16.72 MJ/kg)

Table 4.3: Higher Heating Value of RH

Characterization Parameter	Natarajan et al, 2009	Salleh et al, 2011	Guo et al, 2011	Abu Bakar et al, 2012	**This work**
Calorific Value (kJ/kg)	19807	14380	14570	17340	**16720**

From Table 4.3, the HHV estimated using the above formula based on the elemental composition of C, H, N, S, O is comparable with the literature findings.

4.2 Result of 16 Runs (Calculation of Yield and Conversion)

The sixteen runs were successful and the bio-oil produced was kept in sample bottle, as shown in Figure 4.1. The liquid yield and conversion were calculated and tabulated in Table 4.4. Apart from that, the gas yield and residue were calculated and tabulated in Table 4.5. In these runs, the time that the first drop of bio-oil produced is observed and recorded in Table 4.5 as well.

Figure 4.1: The Bio-Oil Produced Through Catalytic Pyrolysis in 16 Runs

Table 4.4: Liquid Yield and Conversion

Run	Heating Rate (℃/min)	Weight of RH (g)	Weight of Bio-Oil Produced (g)	Decrease in Weight of Reaction Mixture (g)	Liquid Yield (wt%)	Conversion (wt%)
1	5	15.01	4.15	9.07	27.65	60.43
2	5	15.00	4.07	8.84	27.13	58.93
3	5	15.00	4.54	8.85	30.27	59.00
4	5	15.01	4.51	8.91	30.05	59.36
5	10	15.03	4.69	9.21	31.20	61.28
6	10	15.03	4.53	9.14	30.14	60.81
7	10	15.02	4.65	9.45	30.96	62.92
8	10	15.03	4.62	9.15	30.74	60.88
9	15	15.02	4.96	9.26	33.02	61.65
10	15	15.03	4.40	9.52	29.27	63.34
11	15	15.00	4.88	9.33	32.53	62.20
12	15	15.03	4.68	9.43	31.14	62.74
13	20	15.04	5.42	9.54	36.04	63.43
14	20	15.02	5.26	9.49	35.02	63.18
15	20	15.01	5.24	9.66	34.91	64.36
16	20	15.02	5.34	9.66	35.55	64.31

Table 4.5: Gas Yield and Residue Yield

Run	Heating Rate (℃/min)	First Drop of Bio-Oil Observed (min)	Gas Yield (wt%)	Residue Yield (wt%)
1	5	46.26	32.78	39.57
2	5	45.30	31.80	41.07
3	5	45.54	28.73	41.00
4	5	45.15	29.31	40.64
5	10	23.05	30.07	38.72
6	10	21.59	30.67	39.19
7	10	23.04	31.96	37.08
8	10	21.15	30.14	39.12
9	15	15.35	28.63	38.35
10	15	16.29	34.07	36.66
11	15	16.40	29.67	37.80
12	15	16.22	31.60	37.26
13	20	11.50	27.39	36.57
14	20	12.13	28.16	36.82
15	20	12.20	29.45	35.64
16	20	12.15	28.76	35.69

From the result above, it shows that as the heating rate increases, the time that first drop of bio-oil observed is getting shorter and shorter, in minute range. For further analysis, the average values of liquid yield, gas yield, residue yield and conversion are calculated and tabulated in Table 4.6

Table 4.6: Average values of Liquid, Gas, Residue Yield and Conversion

Run	**Heating Rate (℃/min)**	**Liquid Yield (wt%)**	**Gas Yield (wt%)**	**Residue Yield (wt%)**	**Conversion (wt%)**
1	5	28.77	30.66	40.57	59.43
2	10	30.76	30.71	38.53	61.47
3	15	31.49	30.99	37.52	62.48
4	20	35.38	28.44	36.18	63.82

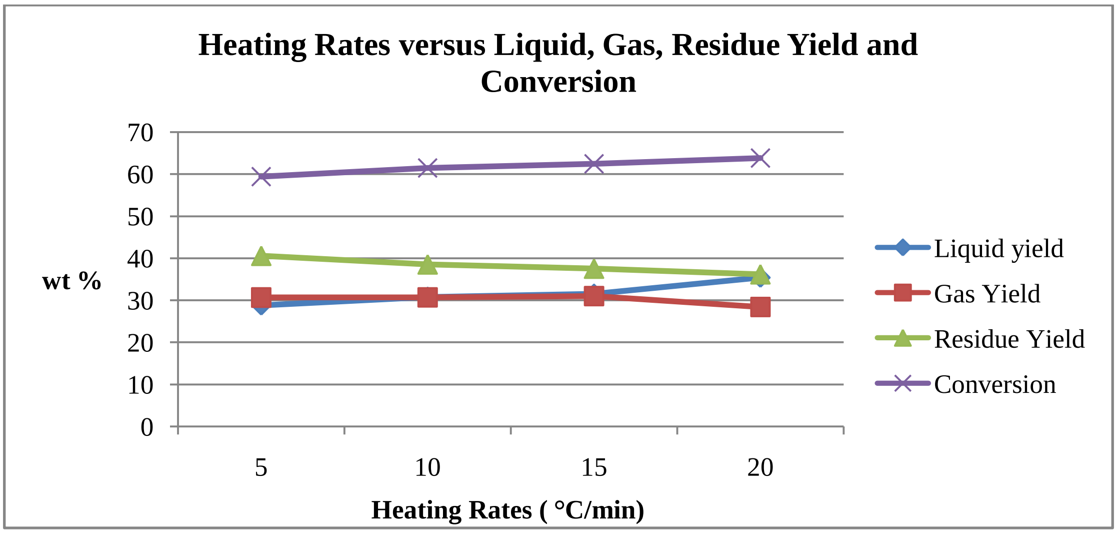


Figure 4.2: Heating Rates versus Liquid, Gas, Residue Yield and Conversion

Figure 4.2 shows that the liquid yield and conversion are increasing as increasing of heating rates. While as increasing the heating rate, the gas yield is slightly increasing but then decreased when heating rate is set at 20 ℃/min, this is because when higher liquid yield and conversion produced, the gas yield will decreased. However, the residue yield is decreasing with the increasing of heating rates. Hence, the author can conclude that at heating rate of 20 ℃/min, the maximum liquid yield of **35.38 wt%** could be obtained. The highest conversion value of **63.82 wt%** also obtained at heating rate of 20 ℃/min.

4.3 Characterization of Bio-Oil Produced

4.3.1 Water Content of Bio-Oil

The water content of bio-oil that produced at different heating rates is measured and tabulated in Table 4.7.

Table 4.7: Water Content of Bio-Oil

Heating Rates (℃/min)	**Water content (wt%)**
5	73.73
10	65.15
15	51.18
20	44.84

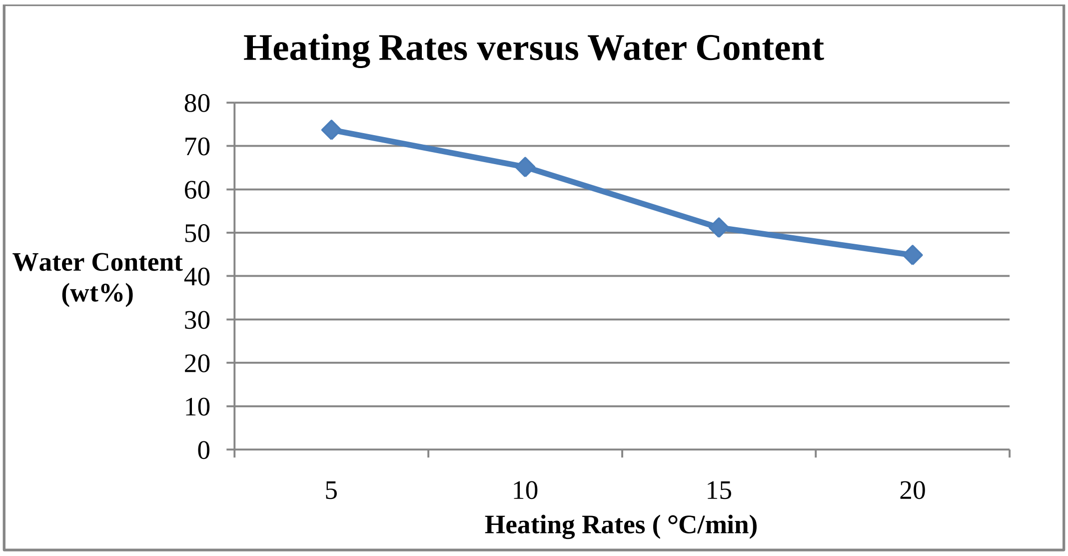


Figure 4.3: Heating Rates versus Water Content

Water is the most abundant compound in bio-oil. It is usually formed during pyrolysis when the breaking up of functional groups from the volatile compounds. Water content in the bio-oil is resulting from the original moisture in the feedstock and as a product of the dehydration reactions occurring pyrolysis (Czernik and Bridgwater, 2004). In this work, the water content is getting decreased as increasing of heating rates, as shown in Figure 4.3. Higher water content in bio-oil is not favored as this will lower the higher heating values of bio-oil and may cause phase separation in storage.

4.3.2 pH of Bio-Oil

The pH of bio-oil that produced at different heating rates is measured and tabulated in Table 4.8.

Table 4.8: pH of Bio-Oil

Heating Rates (℃/min)	pH
5	2.83
10	2.76
15	2.71
20	2.68

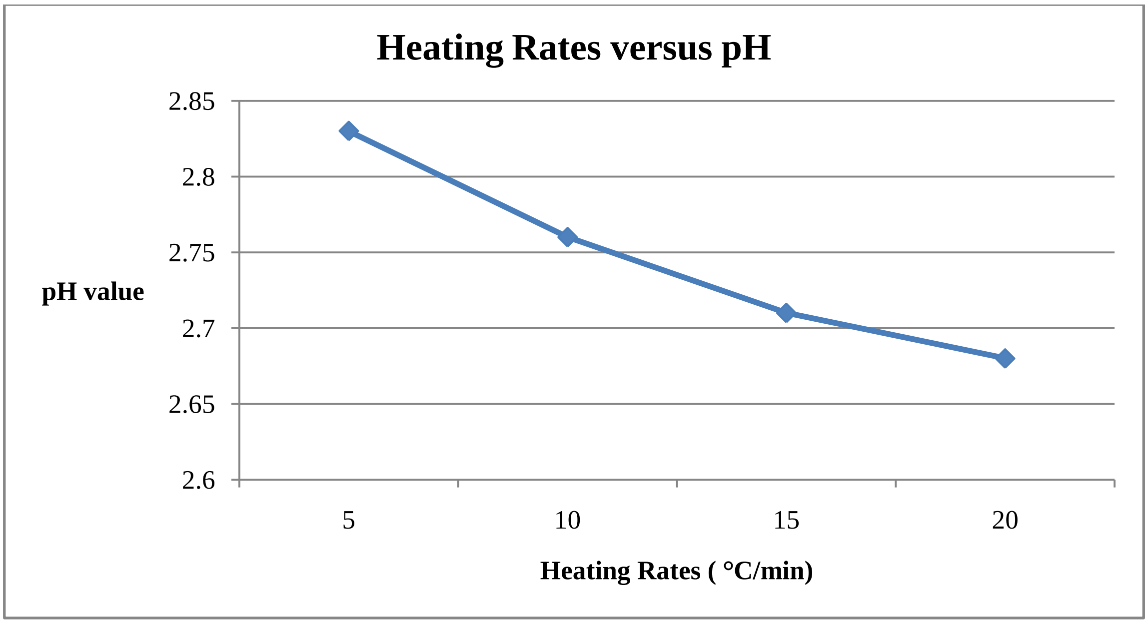


Figure 4.4: Heating Rates versus pH

Normally bio-oil is highly acidic, the pH of bio-oil is reported between the range of 2 to 3. The most acidic pH value obtained is 2.68 at heating rate of 20 ℃/min. In this work, the pH value is getting decreased as increasing of heating rates, as shown in Figure 4.4. Hence, higher the heating rate, the bio-oil produced will be more acidic. Most of the acidity may arise from the presence of acetic acid, but other carboxylic acids, phenols and other acidic compounds will have a significant contribution as well (Abu Bakar et al, 2012).

4.3.3 Viscosity of Bio-Oil

The viscosity of bio-oil is often the most important parameter that is always being reported as it directly shows the stability of the bio-oil. The viscosity of bio-oil that produced at different heating rates is measured and tabulated in Table 4.9.

Table 4.9: Viscosity of Bio-Oil

Heating Rates (℃/min)	**Viscosity (cP at 40 ℃)**
5	42.35
10	44.56
15	45.21
20	47.83

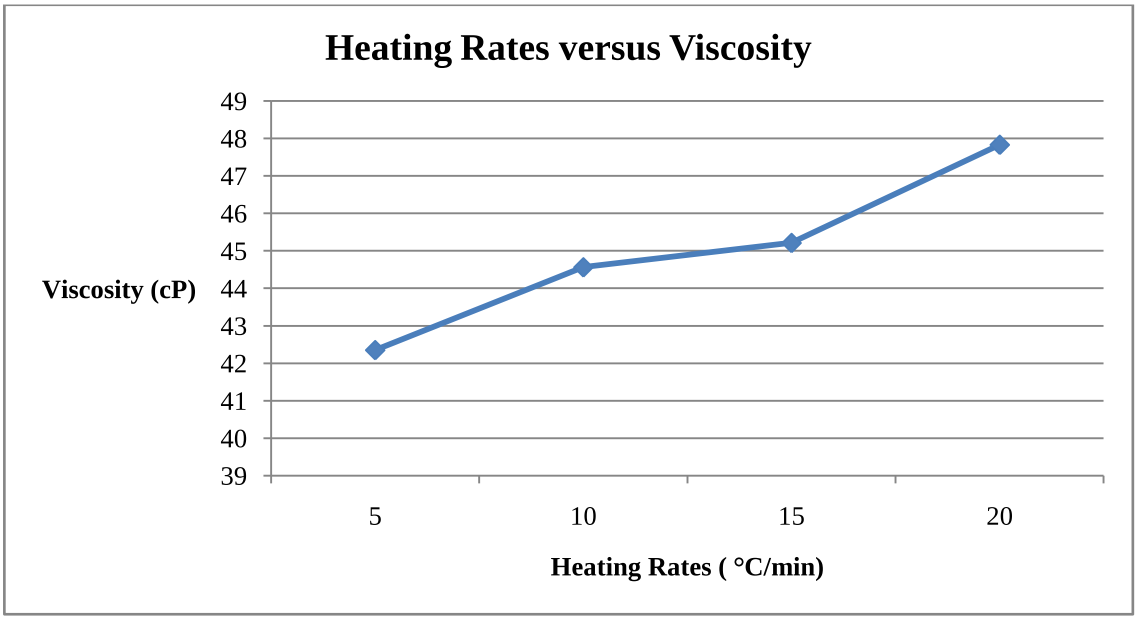


Figure 4.5: Heating Rates versus Viscosity

As seen from Table 4.9 and Figure 4.5, the viscosity of bio-oil produced is increasing with the increment of heating rates. The highest viscosity value of 47.83 cP measured at 40 ℃ is obtained at heating rate of 20 ℃/min. The increase of bio-oil is due to slow polymerization and condensation reaction, which leads to unstable storage condition (Zheng, 2007).

4.3.4 Density of Bio-Oil

The density of bio-oil that produced at different heating rates is measured at 20 ℃ and tabulated in Table 4.10.

Table 4.10: Density of Bio-oil at 20 ℃

Heating Rates (℃/min)	Density at 20 ℃ (kg/m^3)
5	1060.14
10	1064.85
15	1073.80
20	1082.69

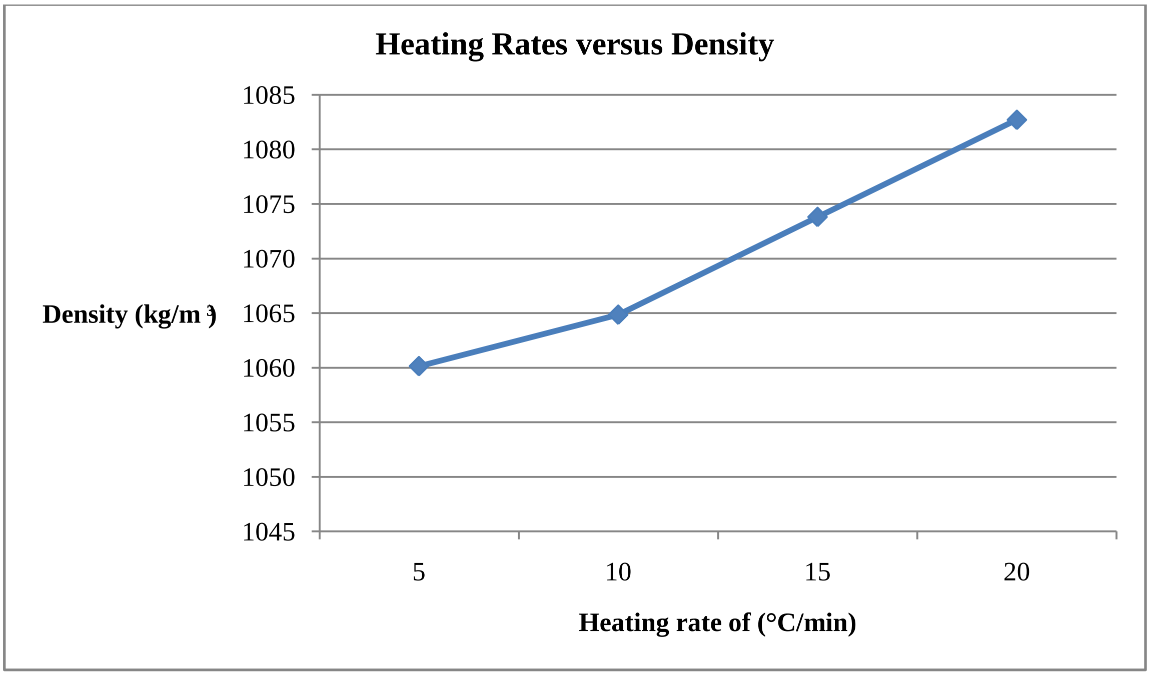


Figure 4.6: Heating Rates versus Density

As shown in Table 4.10 and Figure 4.6, the measured density of bio-oil getting higher as the heating rates increasing. The highest density value of 1082.69 kg/m^3 measured at 20 ℃ is obtained at heating rate of 20 ℃/min. The higher density value will attribute to the increase in the water content, which is non-favored in the production of bio-oil (Abu Bakar et al, 2012).

4.3.5 Ultimate Analysis of Bio-Oil

The ultimate analysis of the bio-oil produced is measured and the content of elemental carbon, hydrogen, nitrogen and sulfur is shown in Table 4.11.

Table 4.11: Ultimate Analysis of Bio-Oil

Ultimate Analysis (wt%)	**Heating Rates (℃/min)**			
	5	**10**	**15**	**20**
Carbon	20.45	22.13	23.87	26.76
Hydrogen	8.53	8.98	9.03	9.22
Nitrogen	0.18	0.22	0.25	0.31
Sulfur	0.27	0.35	0.41	0.44
Oxygen	70.75	68.32	66.44	63.27

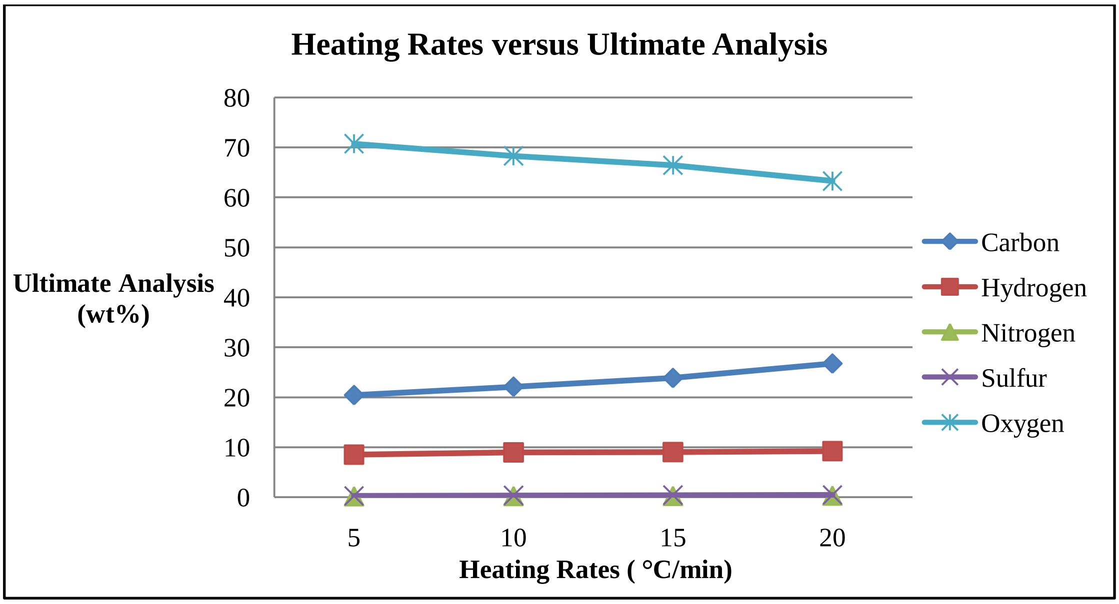


Figure 4.7: Heating Rates versus Ultimate Analysis

From Table 4.11 and Figure 4.7, it can be seen that the carbon, hydrogen, nitrogen and sulfur content are increasing as rising of heating rates. The nitrogen and sulfur contents were still considered low in all of the bio-oils, showing their potential as a clean fuel when used for combustion purposes. The elemental carbon, hydrogen, nitrogen, sulfur and oxygen contents of bio-oil basically have low carbon content and high oxygen content. It is noted that the slow pyrolysis bio-oil is highly oxygenated, with 63.27 wt% of oxygen present in the bio-oil produced at 20 ℃/min.

4.3.6 Higher Heating Value (HHV) of Bio-Oil

The heating value of bio-oil is gives the energy content information of bio-oil, which has the potential to be upgraded to transportation fuels. According to Parikh et al (2005), the HHV of bio-oil can be estimated using the following formula:

$$\mathbf{HHV = 0.3491C + 1.1783H + 0.1005S - 0.1034O - 0.0151N - 0.0211A\ (MJ/kg)}$$

Note: There are assumptions needed to be considered such as the ash content of the bio-oil which is assumed to be 0.5 wt%. The values obtained takes into account the water content in the bio-oil.

Table 4.12: Higher Heating Value of Bio-Oil

Heating Rates (℃/min)	**HHV (MJ/kg)**
5	9.89
10	11.26
15	12.13
20	13.69

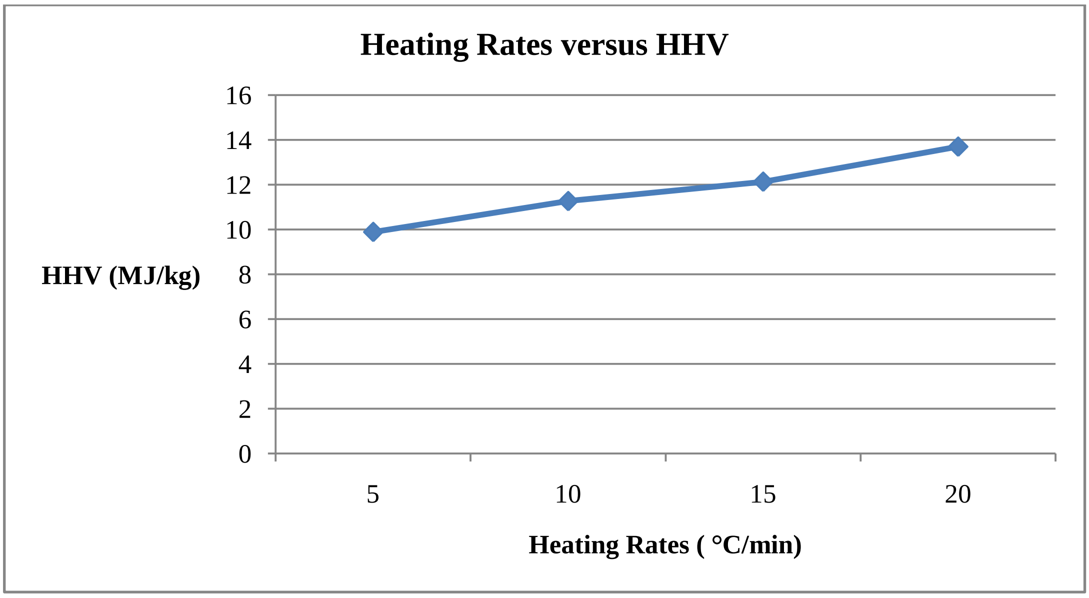


Figure 4.8: Heating Rates versus HHV

HHV is a measurement of the total energy content released when fuel is burnt in air or the maximum amount of energy potentially recoverable from a given biomass. From Table 4.12 and Figure 4.8, it can be shown that the estimated HHV for the bio-oil produced slightly lower compared to the estimated HHV of raw rice husk. The HHV is getting higher as increasing of heating rates. The highest HHV of 13.69MJ/kg is obtained at the heating rate of 20 ℃/min.

4.3.7 Comparison of Characterizations of Bio-Oil Produced with Literature Findings

The characterizations of bio-oil produced at optimum heating rate of 20 ℃/min are compared to the literature findings and the findings are tabulated in Table 4.13.

Table 4.13: Comparison of Characterizations of Bio-Oil Produced at 20 ℃/min with Literature Findings

Characterization Parameters	Zheng, 2007	Guo et al, 2011	Abu Bakar et al, 2012	**This work**
Water Content (wt%)	25.2	33.8	52.6	**44.84**
pH	2.8	3.36	3	**2.68**
Viscosity (cP)	152.32	82.43	1.79	**47.83**
Density (kg/m^3) at 20 ℃	1190	1210	1065	**1082.69**
Ultimate Analysis (wt%)				
Carbon	41.7	35.63	23.38	**26.76**
Hydrogen	7.7	7.00	10.39	**9.22**
Nitrogen	0.3	-	0.51	**0.31**
Sulfur	0.2	-	0.09	**0.44**
Oxygen	50.3	57.37	65.63	**63.27**
HHV (MJ/kg)	17.42	13.36	13.61	**13.69**

As shown in Table 4.13, the author can conclude that the findings from this work are quite similar and comparable with the literature findings. The highlighted point here is estimated HHV of bio-oil produced based on the ultimate analysis measured in this work is almost the same as other findings. Hence, the slow pyrolysis bio-oil produced from this work has high potential to become substitute of conventional fossil fuel as compared to those produced from fast pyrolysis process.

4.3.8 Field Emission Scanning Electron Microscope (FESEM)

To gain insight to particle’s structure, the morphology of raw rice husk and the residue left after slow pyrolysis at heating rate of 20 ℃/min was examined by field emission scanning electron microscope (FESEM). FESEM micrographs of raw rice husk are presented in Figure 4.9.

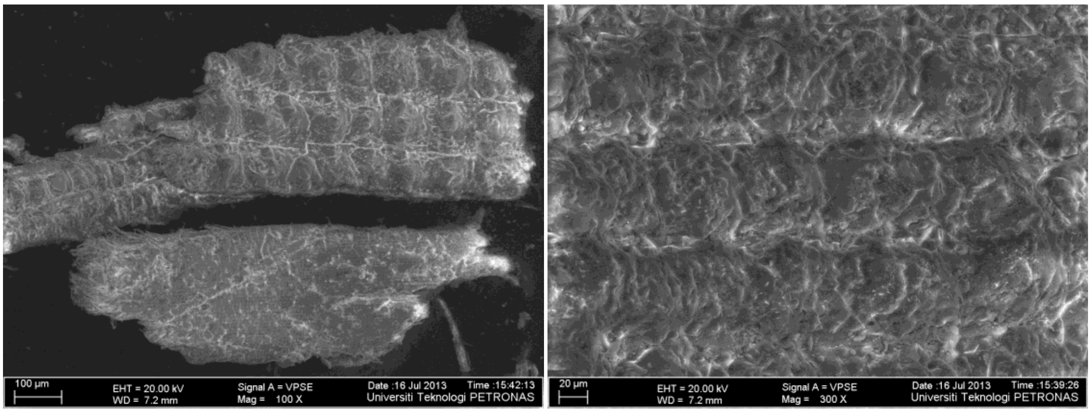


Figure 4.9: Field Emission Scanning Electron Microscope of Raw Rice Husk

As shown in Figure 4.9, the structure of raw rice husk was well-organized and originally in globular shape. The transverse section is dense, with no existence of pores. On the other hand, FESEM micrographs of residue left after slow pyrolysis in nitrogen atmosphere at heating rate of 20 ℃/min are presented in Figure 4.10.

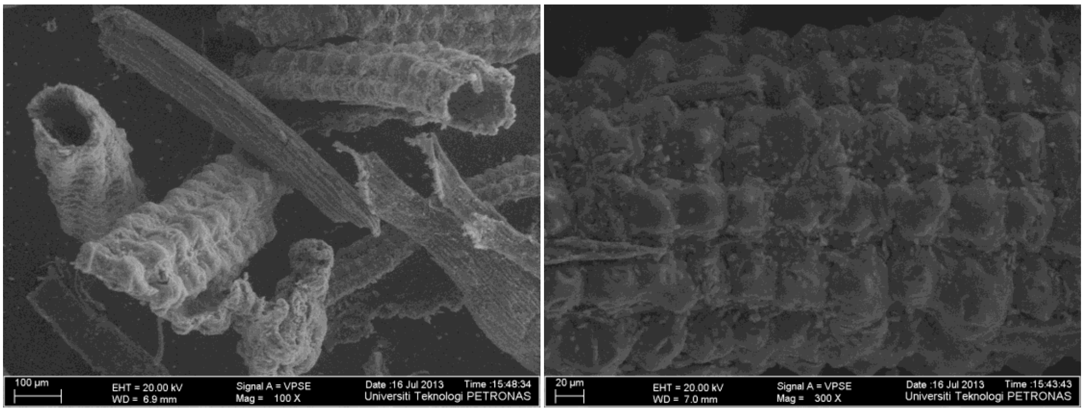


Figure 4.10: Field Emission Scanning Electron Microscope of Residue After Slow Pyrolysis at Heating Rate of 20 ℃/min

However, after slow pyrolysis in nitrogen atmosphere at heating rate of 20 °C/min as shown in Figure 4.10, the surface texture of rice husk residue changed. The globules were shrinked and densified due to the release of volatile products (Vlaev, L. T., et al, 2003). Evaporation of volatile materials creates large numbers of button-like structures or bumps interspaced with small pores formed on the particles with rough surface. The pores appear as channel from where the cellulose material was preferentially removed during pyrolysis. (Bharadwaj, A., et al., 2004).

4.3.9 Chemical Composition (GC-MS)

The chemical compound of bio-oil product was analyzed by using Gas Chromatography-Mass Spectroscopy (GC-MS). The bio-oil produced at different heating rates is analyzed for its chemical composition. According to literature (Heo et al, 2010; Salleh et al, 2011; Tsai et al, 2007; Lu et al, 2012; Lu et al 2011), the components of the bio-oil can be classified into aldehydes, acids, alcohols, ketones, phenols, furan and sugar. In this work, the area % of the GC-MS chromatogram was used to indicate the amount of the various chemical compounds in the bio-oil.

The components that are contained in the bio-oil produced at different heating rates with area % readings are summarized in Table 4.14 to Table 4.23.

Table 4.14: Aldehydes

Chemical Compound	**5 ℃/min**	**10 ℃/min**	**15 ℃/min**	**20 ℃/min**
Aldehydes	**% Area**			
Butanedial	-	-	-	1.12
(E)-2-butenal	-	-	-	0.16
Ethyl vanillin	0.20	0.19	0.18	0.16
Furfural	3.67	3.70	3.52	3.34
Heptanal	0.29	-	-	-
Hydroxy-acetaldehyde	0.54	0.97	1.31	1.68
Vanillin	0.67	0.62	0.66	0.69
1H-pyrrole-2-carboxaldehyde	0.10	0.09	0.11	0.09
2-methyl-propanal	-	-	0.14	-
2,3-dihydroxybenzaldehyde	0.20	-	-	0.22
2,4-dihydroxy-benzaldehyde	-	0.19	-	0.22
2,5-dihydroxybenzaldehyde	-	-	0.21	-
3-furaldehyde	0.04	-	-	0.08
3-hydroxy-butanal	-	-	1.21	-
3-methyl-2-cyclopenten-1-one	0.23	0.23	0.25	0.25
3,5-dimethyl-benzaldehyde	-	-	-	0.15
4-hydroxy-benzaldehyde	0.21	0.21	0.24	0.26
4-hydroxy-2-methoxycinnamaldehyde	-	0.25	0.36	-
4-methyl-2,5-dimethoxybenzaldehyde	-	-	-	0.29
5-dimethoxybenzaldehyde 4-methyl-2	-	-	0.25	-
5-(hydroxymethyl)-2-furancarboxaldehyde	0.36	0.44	0.48	0.50
5-methyl-2-furancarboxaldehyde	0.27	0.26	0.27	0.27

Table 4.15: Alkanes

Chemical Compound	**5 ℃/min**	**10 ℃/min**	**15 ℃/min**	**20 ℃/min**
Alkanes	**% Area**			
Cis-2,3-epoxyheptane	0.85	0.81	-	0.73
Heptane	-	-	-	0.16
Methyl-cyclohexane	-	-	0.46	0.49
Oxepane	-	-	0.22	-
1-bromo-butane	-	0.14	-	-
1-bromo-pentadecane	0.28	-	-	-
1-ethyl-2-methyl-cis-cyclopentane	-	0.35	-	-
1-methyl-7-oxabicyclo[4.1.0]heptane	-	-	0.33	-
2-methyl-hexane	-	0.36	-	0.21
2,2,7,7-tetramethyl-4,5-dimethyleneoctane	0.17	-	-	-
2,3,5-trimethyl-heptane	-	-	0.16	-
3-cyclopropylcarbonyloxytetradecane	-	-	0.22	-
3,5-dimethyl-heptane	-	-	-	0.15
3,5-dimethyl-octane	-	-	0.31	-

Table 4.16: Alcohols

Chemical Compound	**5 °C/min**	**10 °C/min**	**15 °C/min**	**20 °C/min**
Alcohols	**% Area**			
Cyclopropyl carbinol	0.60	-	0.59	0.56
Eugenol	0.23	0.22	0.25	-
Homovanillyl alcohol	1.64	-	1.60	-
Methyl alcohol	-	-	-	2.79
N,n-diethyl-2-aminoethanol	-	-	0.29	-
Isopropyl alcohol	-	-	0.60	-
1,2-benzenediol	2.25	2.33	2.06	1.93
1-2,ethanediol	0.04	0.03	0.04	-
1-(2-nitropropyl)-cyclohexanol	-	-	0.25	-
2-chloroethanol	3.30	3.22	-	-
2-furanmethanol	3.06	0.08	3.67	3.39
2-methoxy-1,4-benzenediol	0.33	-	0.21	0.47
2-methoxy-4-[[[2-(4-hydroxyphenyl)ethyl]amino]methyl]-1-benzenol	0.24	-	-	-
2-methylene-6,6-dimethyl-bicyclo[3.2.0]hepten-3-ol	-	0.27	-	0.26
(2-methyloxiranyl) methanol	0.67	1.06	-	-
2-phenoxy-ethanol	-	0.18	0.37	0.17
2-pentyn-1-ol	-	-	0.12	-
2,3-dihydro-2,3,7-benzofurandiol	-	-	0.17	-
2,6,6-trimethyl 2-cyclohexen-1-ol	-	-	-	0.24
3-ethyl-3-pentanol	-	0.15	-	-
3-hydroxy-benzenemethanol	-	0.36	-	-
3-methoxy-1-butanol	0.15	0.13	-	-
3-methoxy-1,2-benzenediol	0.39	0.33	0.30	0.29
3-pyridinol	0.23	0.38	0.40	0.18
4-methyl-1,2-benzenediol	0.37	-	-	-
4-amino-2,3-xylenol	-	1.60	-	1.62
5-cyclopropyl-1,3,4-oxadiazole-2-thiol	0.22	-	-	0.13

Table 4.17: Phenols

Chemical Compound	**5 °C/min**	**10 °C/min**	**15 °C/min**	**20 °C/min**
Phenols	**% Area**			
Phenol	0.78	0.72	0.66	0.62
2-methoxy-4-propyl-phenol	-	0.14	0.24	0.46
2-methoxy-phenol	2.21	2.10	2.08	2.24
2-methoxy-4-methyl-phenol	0.83	0.87	0.93	3.39
2-methoxy-4-vinylphenol	1.92	1.88	2.10	2.30
2-methoxy-4-(1-propenyl)-phenol	0.69	0.52	0.68	1.11
2-methyl-phenol	0.42	0.43	0.48	-
2,4-dimethyl-phenol	-	0.19	-	-
2,6-dimethoxy-phenol	0.99	0.94	0.91	0.93
2,6-dimethoxy-4-(2-propenyl)-phenol	0.23	0.17	0.24	0.27
4-(ethoxymethyl)-2-methoxy phenol	-	-	-	0.18
4-ethyl-2-methoxy-phenol	1.12	1.08	1.28	1.62
4-ethyl-phenol	0.67	0.59	0.56	0.54
4-methyl-phenol	-	-	-	0.46
4-(methylthio)-phenol	0.33	-	-	-
4-((1E)-3-hydroxy-1-propenyl)-2-methoxyphenol	-	-	-	0.29

Table 4.18: Alkenes

Chemical Compound	**5 °C/min**	**10 °C/min**	**15 °C/min**	**20 °C/min**
Alkenes	**% Area**			
(Z)-3-hexene	0.57	-	-	-
(Z)-5-decene	-	0.24	-	-
[1-(2,2-dimethylhydrazino)ethyl]ethyl-diazene	-	-	-	4.38
1,3,5,7-cyclooctatetraene	-	-	0.33	-
2-hexene	-	-	0.17	-
2-methyl-3-heptene	0.22	-	-	-
2,6-dimethyl-, [S-(E)]-4-octene	0.26	-	-	-
3-ethyl-thiophene	0.14	-	-	-
3-hexene	-	-	1.30	-
4-ethyl-3-heptene	-	-	-	0.26
4,4-dimethyl-2-pentene	0.48	-	-	-

Table 4.19: Carboxylic Acids

Chemical Compound	**5 ℃/min**	**10 ℃/min**	**15 ℃/min**	**20 ℃/min**
Carboxylic Acids	**% Area**			
Acetic acid	20.87	18.72	19.05	17.86
Butanoic acid	-	2.43	-	1.08
Formic acid	0.51	0.80	0.93	0.90
Octanoic acid-tert butyl ester	0.22	-	-	-
Oxalic acid	-	-	-	0.22
Propanedioic acid	0.16	-	-	-
Propanoic acid	0.58	0.22	-	-
.Beta.-(4-hydroxy-3-methoxyphenyl)propionic acid	0.13	-	-	-
Spirohexane-1-carboxylic acid	-	-	-	0.30
Trichloroacetic acid	-	-	-	0.32
1-methylethyl ester hexanoic acid	0.81	0.78	-	0.22
2-acetyl-hexanoic acid	0.25	0.22	0.24	0.22
2-hydroxy-propanoic acid	0.16	0.17	0.20	-
2-methyl-4-oxo-pentanoic acid	0.56	0.48	-	-
2-methyl-propanoic acid	-	-	0.29	-
2-nonenoic acid	-	0.28	-	-
2-nonynoic acid	0.17	-	-	-
2-oxo-n-valeric acid	-	-	1.48	-
2-oxo-propanoic acid	-	-	0.81	0.85
2,5-dimethylterephthalicdihydroxamic acid	-	-	-	0.08
3-hydroxy-butanoic acid	0.44	-	-	-
3-methyl-4-oxo-pentanoic acid	-	-	-	0.45
3-(3-hydroxyphenyl)-2-propenoic acid	-	-	-	0.05
3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid	-	-	-	0.03
3,5-dimethoxy-4-hydroxyphenylacetic acid	0.25	0.20	0.22	0.16
4-hydroxy-3-methoxy-benzeneacetic acid	-	0.16	0.19	-
4-methyl-3-pentenoic acid	-	-	0.09	-
4-methoxy-4’,5’-methylenedioxybiphenyl-2-carboxylic acid	-	-	0.07	0.10
4-methylpentyl ester 2,2-dimethylpropionic acid	-	0.18	-	-
4-oxo- pentanoic acid	0.42	-	-	-
5-amino-pyrazole-3-carboxylic acid	-	0.21	0.20	-

Table 4.20: Ketones

Chemical Compound	5 ℃/min	10 ℃/min	15 ℃/min	20 ℃/min
Ketones	**% Area**			
Acetone	0.58	0.82	0.92	0.96
Butyrolactone	1.32	1.35	-	-
Diethylaminoacetone	-	-	-	0.33
Dihydro-2H-pyran-3(4H)-one	0.21	0.13	-	-
Dihydro-4-hydroxy-2(3H)-furanone	-	5.26	4.55	-
(E)-3-penten-2-one	-	-	0.29	-
.alpha.-amino-3’-hydroxy-4’-methoxyacetophenone	-	-	0.15	-
1-(acetyloxy)-2-butanone	0.25	0.24	0.23	-
1-(acetyloxy)-2-propanone	1.50	1.52	1.50	1.47
1-hydroxy-2-butanone	-	1.29	1.22	1.16
1-hydroxy-2-propanone	6.10	7.73	7.08	8.78
1-(2-furanyl)-ethanone	0.25	0.23	0.22	0.23
1-(2,2-dimethylcyclopentyl)-ethanone	-	0.32	-	0.30
1-(2,4-dihydroxyphenyl)-ethanone	-	0.27	0.29	0.30
1-(3-hydroxy-4-methoxyphenyl)-ethanone	-	-	-	0.29
1-(4-hydroxy-3-methoxyphenyl)-ethanone	0.15	0.28	0.32	0.33
1-(4-hydroxy-3-methoxyphenyl)-2-propanone	0.12	-	-	-
1-(4-hydroxy-3,5-dimethoxyphenyl)-ethanone	0.25	0.21	0.24	0.23
2-acethylcyclopentanone	0.31	-	-	-
2-acetoxy-5-hydroxyacetophenone	0.29	-	-	-
2-cyclopentene-1,4-dione	-	-	-	0.20
2-ethyl-4-propyl-1,3-cyclopentanedione	-	0.18	-	-
2-hydroxy-2-cyclopenten-1-one	2.31	2.62	2.64	2.76
2-hydroxy-3-methyl-2-cyclopenten-1-one	1.56	-	1.56	1.59
2-methyl-3-hexanone	-	1.04	0.95	-
2-pentanone	-	0.18	0.20	0.20
2-pyrrolidione	-	-	-	0.34
2,3-butanedione	1.98	2.38	2.38	-
2,3-dimethyl-2-cyclopenten-1-one	0.18	0.17	0.26	0.25
2,3-pentanedione	0.19	0.28	0.41	0.29
2,3,7,7a-tetrahydro-7a-methyl-1H-indene-1,5(6H)-dione	-	-	-	0.14
2,4’-dihydroxy-3’-methoxyacetophenone	0.28	-	-	-
2,5-dihydro-3,5-dimethyl 2-furanone	0.32	-	-	0.26
2(5H)-furanone	-	-	0.44	1.39
3-ehylidene-heptan-2,6-dione	0.15	-	-	-
3-ethyl-2-hydroxy-2-cyclopenten-1-one	1.19	1.08	1.00	1.19

3-hexanone	0.24	0.23	0.22	0.23
3-hydroxy-2-butanone	-	0.63	-	0.57
3-methyl-1,2-cyclopentanedione	-	1.63	-	-
3-methyl-2-cyclopenten-1-one	-	-	0.25	0.25
3-methyl-2-butanone	-	-	-	2.38
3-methyl-2, 4-hexanedione	0.09	-	-	-
3-methyl-2(5H)-furanone	0.14	0.13	0.14	0.15
3-methyl-2,5-furandione	-	-	-	0.13
3,6-heptanedione	-	0.08	-	-
4-hydroxy-2-methoxycinnamaldehyde 1-(2-ethoxyphenyl) acetone	0.25	-	-	0.45
4-methyl-5H-furan-2-one	0.19	0.18	0.21	0.23
4,4,5-trimethyl-2-cyclohexen-1-one	-	-	0.27	-
5-acethyldihydro-2(3H)-furanone	0.26	0.23	-	-
5-butyldihydro-4-methyl-2(3H)-furanone	-	0.33	-	0.35
5-methyl-2(5H)-furanone	0.11	0.10	0.12	0.13
6-methoxy-3(2H)-pyridazinone	-	-	0.18	-
6,7-dimethyl-1H-pyrrolo [3,4-c] pyridine-1,3,4(2H,5H)-trione	0.14	0.12	0.13	-
8-hydroxy-2H-1-benzopyran-2-one	-	0.10	0.13	-

Table 4.21: Esters

Chemical Compound	**5 °C/min**	**10 °C/min**	**15 °C/min**	**20 °C/min**
Esters	**% Area**			
Ethyl acetate	-	-	0.50	-
Ethyl homovanillate	0.09	0.07	0.08	0.09
Methyl 2-furoate	0.07	0.05	-	-
O-methyl n-tert-butyllformimidate	-	-	-	0.19
Vinyl butyrate	1.40	-	-	-
2-ethoxyethyl acetate	-	-	0.16	-
2-isopropoxyethyl butyrate	-	-	0.32	-

Table 4.22: Furans

Chemical Compound	**5 °C/min**	**10 °C/min**	**15 °C/min**	**20 °C/min**
Furans	**% Area**			
(S)-(+)-3-Hydroxytetrahydrofuran	1.25	-	1.22	-
Tetrahydro-2,4-dimethyl-,trans-furan	-	-	0.38	-
Tetrahydro-2,5-dimethoxy-furan	0.24	0.17	0.28	0.30
2-methoxytetrahydrofuran	0.09	0.09	0.11	-
2,3-dihydro-benzofuran	1.15	1.12	1.40	1.90

Table 4.23: Sugar

Chemical Compound	**5 ℃/min**	**10 ℃/min**	**15 ℃/min**	**20 ℃/min**
Sugar	**% Area**			
1,4:3,6-dianhydro-.alpha.-d-glucopyranose	0.94	0.83	0.73	0.74
2,3-anhydro-d-mannosane	0.43	0.49	0.46	0.48
Levoglucosan	2.84	3.49	3.29	2.95

As listed in from Table 4.14 to Table 4.23, the bio-oil produced at heating rate of 20 ℃/min has more aldehydes, acids, ketones and phenol compounds and more compounds of heavy molecular weight which cause of high viscosity reading. The higher number of phenol and acids compounds contribute to the higher pH number of bio-oil that produced at heating rate of 20 ℃/min

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In conclusion, this study serves to enhance the research and development of the renewable energy sources towards the sustainability of energy. In this context, biomass is gaining more significance as an alternative renewable energy source in view of the global depletion of fossil fuels. Bio-oil produced from biomass pyrolysis reaction can be a promising substitute to the conventional fuels that we are heavily depending on. Hence, the slow pyrolysis of rice husk (RH) at different heating rate is studied in this project. The properties of bio-oil produced were further tested. FESEM analyses of raw rice husk and rice husk residue left after pyrolysis were done to study the morphology of particles. GC-MS analysis also performed to identify the chemical compounds available inside the bio-oil. The physical properties of bio-oil produced at the optimum condition are summarized in Table 5.1.

Table 5.1: Summary of the Physical Properties of Bio-Oil Produced at 20 ℃/min

Properties	Measured Value
Water content	44.84 wt%
pH	2.68
Viscosity	47.83 cP (40 ℃)
Ultimate Analysis • Carbon • Hydrogen • Nitrogen • Sulfur • Oxygen	 26.76 wt% 9.22 wt% 0.31 wt% 0.44 wt% 63.27 wt%
Density	1082.69 kg/m^3 (20 ℃)
Higher Heating Value (HHV)	13.69 MJ/kg

From this work, the optimum reaction condition giving the maximum liquid yield is determined at heating rate of 20 °C/min, temperature of 500 °C and nitrogen flow rate of 100 ml/min. The maximum average liquid yield of bio-oil that obtained is **35.38 wt%**. The highest conversion value of **63.82 wt%** also obtained at heating rate of 20 °C/min. Hence, the objectives of this project are achieved.

5.2 Recommendations

As the research of pyrolysis is indefinitely wide, various future works can be done to further expand and continue the research in various aspects of pyrolysis. In order to commercialize the production of bio-oil and its upgraded products using pyrolysis reaction in a big scale, the optimum reactor and reaction parameters has yet to be established. As such, the researches in the laboratory scale are being carried out actively in order to explore the possible and best ways to utilize biomass. Some of the recommendations that can be expanded in the future are:

i. Explore the possibility of upgrading the bio-oil using various stabilization methods in the slow pyrolysis and different reactor configuration and operating parameters.
ii. Explore other biomass feedstocks (may be the same type of feedstock but from different regions or countries) and the results can be compared with the literature readings available.
iii. Detailed study of the reaction mechanism of the 3 main components; cellulose, hemicellulose and lignin of RH during slow pyrolysis.

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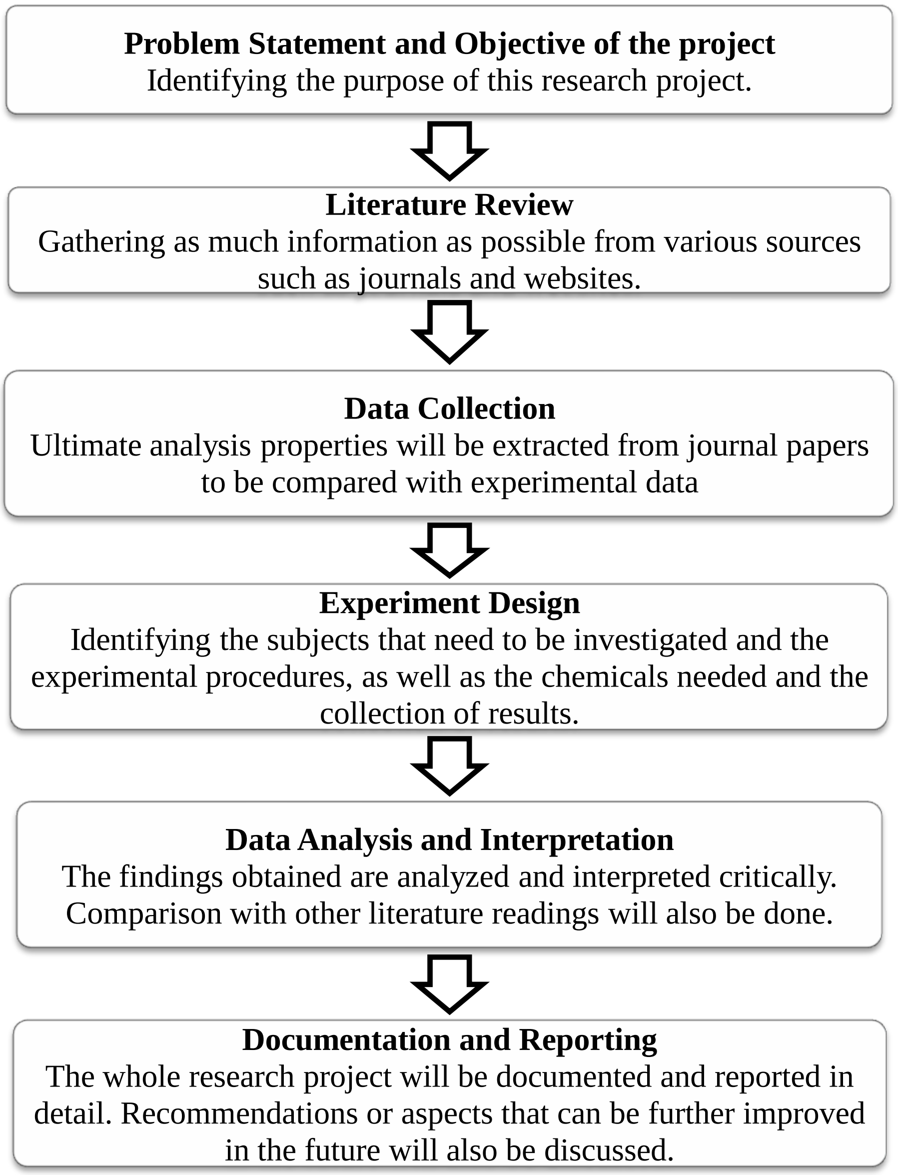
APPENDICES

Appendix 1: Project Activities



Problem Statement and Objective of the project
Identifying the purpose of this research project.

Literature Review
Gathering as much information as possible from various sources such as journals and websites.

Data Collection
Ultimate analysis properties will be extracted from journal papers to be compared with experimental data

Experiment Design
Identifying the subjects that need to be investigated and the experimental procedures, as well as the chemicals needed and the collection of results.

Data Analysis and Interpretation
The findings obtained are analyzed and interpreted critically. Comparison with other literature readings will also be done.

Documentation and Reporting
The whole research project will be documented and reported in detail. Recommendations or aspects that can be further improved in the future will also be discussed.

Figure I: The Sequence of Project Activities

Appendix 2: Key Milestones

The key milestones are important agendas that need to be accomplished in time. Note that ● represents the important milestones that need to be fulfilled on time.

Table I: Key Milestones for Final Year Project (FYP I and II)

No	DETAIL WEEK	1,2	3,4	5,6	7,8	9,10	11,12	13,14	15,16	17,18	19,20	21,22	23,24	25,26	27,28
1	Confirmation of FYP topic	🟦													
2	Literature Review		🟦	🟦											
3	Extended Proposal				●										
4	Rough planning of experiments				🟦	🟦									
5	Proposal Defence				●	●									
6	Detailed literature review				🟦	🟦									
7	Detailed planning of experiments				🟦	🟦	🟦								
8	Documentation on interim report						●								
10	Obtaining the raw materials and chemicals needed								🟦						
11	Run experiments and obtaining results								🟦	🟦	🟦	🟦	🟦		
12	Analyze and interpret the results obtained									🟦	🟦	🟦	🟦		
13	Documentation of the whole project and presentation												🟦	🟦	●

Appendix 3: Gantt Chart

The Gantt Charts for FYP I and II are outlined in Table II and Table III respectively.

Table II: Gantt Chart for FYP I

NO	DETAIL / WEEK	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues	🟪	🟪	🟪	🟪	🟪	🟪	🟪									
2	Submission of Progress Report									●							
3	Project Work Continues									🟪	🟪	🟪	🟪	🟪			
4	Pre-EDX Presentation												●				
5	Submission of Draft Report													●			
6	Submission of Dissertation (Soft Bound)														●		
7	Submission of Technical Paper														●		
8	Oral Presentation															●	
9	Submission of Project Dissertation (Hard Bound)																●

● Suggested milestone

🟪 Process

Table III: Gantt Chart for FYP II

No	Week Task Name	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues	🟦	🟦	🟦	🟦	🟦	🟦	🟦									
2	Submission of Progress Report									●							
3	Project Work Continues									🟦	🟦	🟦	🟦	🟦			
4	Preparation for Pre-SEDEX											🟦	🟦				
5	Pre-SEDEX Presentation												●				
6	Preparation of Draft Report and Draft Technical Report											🟦	🟦	🟦			
6	Submission of Draft Report													●			
7	Submission of Dissertation (Soft Bound)														●		
8	Submission of Technical Paper														●		
9	Oral Presentation															●	
10	Submission of Project Dissertation (Hard Bound)																●

● Suggested milestone

🟦 Process