Catalytic Pyrolysis of Oil Palm Frond Using Graphite Nanofiber (GNF) As Catalyst

By

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CERTIFICATION OF APPROVAL

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Approved by,

____

(Dr. Suzana bt Yusop)

UNIVERSITI TEKNOLOGI PETRONAS
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MAY 2014

CERTIFICATION OF ORIGINALITY

This to certify that I am responsible for the work submitted for 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.

(MUHAMAD SHAFIQ BIN SHAHIDAN)

ABSTRACT

Nowadays, people are focused in doing research to find the sustainable renewable energy as the future energy due to the depletion of fossil fuel as the current source of energy. Bio-fuel was observed as one of the alternative of the renewable energy as it is produced from biomass which can be easily obtained. It is proven as the sustainable future energy due to the high market demand which is 11.8 billion litres based on recent report data. Hence, utilizing the biomass by converting oil palm frond into bio-oil is seen as to be one of the possible way to produce bio-fuels by the process of pyrolysis. In this study, the catalytic pyrolysis process of oil palm frond will be carried out.

Catalytic pyrolysis is a process of decomposition of organic material through thermal degradation at high temperature with the absence oxygen and with the presence of catalyst while the product of the pyrolysis is called bio-oil. The objective of the study is to conduct the catalytic pyrolysis of oil palm frond using graphite nanofiber (GNF) as the catalyst support and to study the effectiveness of the catalyst in cracking the biomass. The experiment is conducted by varying the operating parameters ; temperature and catalyst loading. The effectiveness of the catalyst used in cracking the biomass is determined by the yield of the bio-oil product after the reaction.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

The project is all about converting the biomass which in this case oil palm frond into bio-fuel by using the technique of catalytic pyrolysis with the assistance of graphite nanofiber (GNF) as the catalyst support. Currently, the first generation of bio-fuels are mainly produced from the plant which is the source of food manufacturing. However, an issue was raised where people argued that the use of plant to produce bio-fuel would have a significant impact on the global food supply in developing countries. The suffering from hunger and starvation at some of the Earth's population have also strengthen this issue. Therefore, the "Second Generation" of producing bio-fuel were proposed by the experts which is from non-edible biomass feedstock. According to Hubber and Corma (2010), there are four categories of biomass-derived feedstock for the production of biofuel which are waste materials (agricultural, wood, crop residues), aquatic biomass (algae, water weed, water hyacinth), energy crops (corn, wheat, barley)and forest products (wood, logging residues, trees, shrubs).

The oil palm frond is the by-product of the cultivation of the oil palm trees. It is collected during pruning and replanting activities. Dahlan (2010) explained that the increasing output of fibrous wastes derived from the harvesting of oil palm fruit brunches is caused by the rapid development of the palm oil industry since 1990s, especially in South-East Asia (Malaysia, Indonesia). It was estimated that at an average of 54.43 million tonnes per year of oil palm fronds will be available during the replanting process in the years of 2007-2020 (Beijing Forestry and Parks Department of International Cooperaton, 2009). In the 1990s, the oil palm fronds were usually burned but due to the environmental issue, the practice was banned. For the current practice, oil palm fronds are left to rot on the ground and fertilize the soil (Lim et al., 2000, cited by Heuze et al., 2012). The utilization of this source of waste through energy recovery could have the potential to generate useful energy and solve the waste management problem due to their abundance.

1.2 PROBLEM STATEMENT

The rate of depletion of fossil fuels is vastly increased due to the burgeoning demand of its use nowadays. The continuous use of fossil fuel as the non-renewable source to produce fuel will only lead to its extinction and people will need to rely on other sources. The abundance of carbon dioxide generated from the consumption of fossil fuels have lead to the greenhouse effects and cause global warming. This environmental pollution is a concern because it will effects the life of mankind. Besides, the use of food sources such as corn, sugarcane, rapeseed, soybean and etc. in producing bio-fuel get a serious objection by the people. The continuous use of the food sources will disrupt the food supply for the people especially whom suffer from starvation.

Other than that, there are a very few studies regarding the usage of graphite nanofiber (GNF) as catalyst in pyrolysis. Hence, it is hope that this study can provide useful information on the new catalyst. Furthermore, the yield of the product is low in previous research. Hence, by using oil palm frond and GNF as catalyst it is estimated that the yield of liquid product will be higher.

1.3 OBJECTIVE

The objectives of this project that will be carry out are :

- To conduct properties characterization and catalytic pyrolysis of the oil palm frond using GNF as the catalyst
- To study the effectiveness of the catalyst in cracking the biomass

1.4 SCOPE OF STUDY

For this experiment, a semi batch reactor will be used to conduct the catalytic pyrolysis process at temperature from 300°C-500°C with the aid of GNF as the catalyst support. The reactor will be heated by an electric vertical furnace. The catalytic pyrolysis process will be conducted at different operating parameter which are :

1. Effect of catalyst loading
2. Effect of temperature

CHAPTER 2

LITERATURE REVIEW

2.1 CATALYTIC PYROLYSIS

Pyrolysis is a process in which thermochemical decomposition of biomass occur in the absence of oxygen at temperatures of 400°C-650°C (Dickerson and Soria, 2013). While according to Cheung et.al (2011), pyrolysis is defined as decomposition of organic material through thermal degradation process at high temperature with no oxygen involved. The product of the process called bio-oil, contains up to 70% of the energy of the biomass feed and it can produce high yield of the liquid product (Bridgewater and Peacocke, 2000). However, Czernik and Bridgewater (2004) reviewed that the application of the bio-oil is restricted due to its certain properties such as low heating value, incomplete volatility, acidity, instability and incompatibility with standard petroleum fuels.

The different classes of oxygenated organic compounds contained in the chemical composition of bio-oil caused the undesirable properties of pyrolysis oil (French and Czernik, 2009). Hence the bio-oil need to undergo catalytic cracking process to eliminate the oxygen content via deoxygenation. This is necessary so that the bio-oil produced would be broadly accepted and economically attractive due to its stability and quality. French and Czernik (2009) also reported that the deoxygenation can be accomplished through simulataneous dehydration, decarboxylation and decarbonylation reactions occurring in the presence of catalyst. According to Vi et al. (2013), decomposition reaction which reduces the oxygenate content to improve the calorific value of the bio-oil with better hydrocarbon distribution can be studied with the involvement of catalyst in the reaction. In the paper, the catalytic pyrolysis using the rice husk was investigated where maximum liquid yield was found to be optimum based on the catalyst type, catalyst loading, reaction temperature and nitrogen flow rate.

Studies of pyrolysis in the absence of a catalyst have been reported previously (Schwab et al., 1988; Fortes and Baugh, 1994; Idem et al., 1996 cited by Junming et al., 2010). The characterization of pyrolysis gas and liquid products were reported in those papers. In addition, the usage of catalyst such as HZSM-5 (Twaiq et al., 2003a,b cited by Junming et al., 2010) and MCM-41 (Sang et al., 2003,2004 cited by Junming et al., 2010) have been developed for the use in pyrolysis reaction. The usage of those catalyst have improvised the selectivity of products. However, Bhatia et al. (2009) revealed that rapid deactivation of the catalyst has become the major barrier in the catalytic cracking reaction. Bridgewater (1996) says that the chemicals from biomass pyrolysis can be recovered by physical and/or chemical processing from the crude liquid. He added, the natural catalyst in most biomass forms are enhanced to emphasize production of specific chemicals and hence, improve the product yield or derive higher value chemicals.

2.2 GNF AS CATALYST

Graphite nanofibers (GNF) are black spherical high surface area graphitic carbon. It consist of graphite platelets perfectly arranged in different orientations with respect to the fiber axis. The presence of large number of edges, which in turn constitute sites readily available for chemical or physical interaction especially adsorption have made it as the fabulous feature of the structure (Baker, 1998). Krijn and Geus (2007) wrote that the increasing interest in this graphitic materials originates from its potential for unique applications as well as their chemical similarity to fullerenes and carbon nanotubes.

The usage of GNF as the catalyst support in reaction was reported by Rodriguez et al. (1994). In their paper, catalytic hydrogenation activity of Fe-Cu was investigated by involving GNF as the catalyst support in attempting for exploitation of the GNF potential as a catalyst support. It was found that GNF give the effective reaction towards the performance of Fe-Cu. The conversion of the hydrocarbons was considerably higher compared to active carbon and γ-Alumina when they are used as catalyst support. Furthermore, GNF have been utilized in a number of catalysis. GNF was found to be ideal for lithium intercalation in Lithium-ion batteries (Yoon et al., 2004) while according to Ma et al. (2001), GNF can be used to enhance the reactivity and selectivity of supported metal catalyst. It was also explored to be used as cathode catalyst support for fuel cells.

CHAPTER 3

METHODOLOGY

3.1 PROJECT ACTIVITY

Literature Review

- For FYP II further understanding regarding the concept of catalytic pyrolysis is done.
- More literature have been reviewed to study more about GNF catalyst and its calcination process

Experiment

- Experiement is designed to conduct the catalytic pyrolysis process
- Equipments and materials required for the experiment are prepared. The experiment is currently ongoing.

Data Collection

- From the experiment conducted, the data is collected.
- Based on the results, the data is analyzed and calculated using the formula provided.

Conclusion

- The experiment is concluded based on the results obtained.
- Report for the project will be prepared

3.2 GANTT CHART AND KEY MILESTONE

No	Detail Work / 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- Sedex										🟥					
5	Submission of Draft Final Report											🟥				
6	Submission of Dissertation (soft bound)												🟥			
7	Submission of Technical Paper												🟥			
8	Viva													🟥		
9	Submission of Dissertation (hard bound)															🟥

🟩 Proces 🟥 Suggested Milestone

3.3 EXPERIMENT METHODOLOGY

3.3.1 Sample Preparation and Characterization

The sample which in this case is oil palm frond is prepared by reducing its size using the Cutting Mill and sieved to a particle size of 250 µm – 500 µm. The sieved oil palm frond are dried in the oven at 105°C before being used in the experiment and kept in a safe container.

For the graphite nano fiber (GNF) catalyst, it is obtained from the company provider which is Platinum Green Chemicals Sdn. Bhd. There are various batches of catalyst provided. Hence, the batches of the catalyst are differ in term of the internal composition, surface area and etc. The difference between the batches is determined by characterization process through various method such as field emission scanning electron microsope (FESEM), transmission electron microscopy (TEM) and Brunauer Emmett Teller (BET). Due to the limited quantity of the catalyst, the experiment is done by using only the first batch of the catalyst and thus, it will be one of the variable that being kept fixed. Before the catalyst is used, it has been calcined at 600°C and reduced in a 20% hydrogen-in-nitrogen flow (100ml/min) for 3 hours to ensure the catalyst is stay active (Tao et al., 2009)

3.3.2 Tools and Equipment

For the catalytic pyrolysis experiment, there are some equipment need to be used in order to complete the setup of the experiment. Among the equipment and tools that will be used in the experiment are summarised in the table below with their functions stated.

Equipment/Tool	Function
Borosilicate tube	To allow the catalytic pyrolysis to take place
Furnace	To heat the borosilicate tube contain with sample
Thermocouple	To measure the temperature inside the tube
Thermometer	To monitor the temperature inside the tube
Flow controller	To control the flow of nitrogen
Condenser	To condense the gas from the reaction. The product which is bio-oil is collected from condenser
Pressure gauge	To measure the pressure inside the tube
Ice box	To keep the ice so that the temperature of condenser is kept at 0°C
Spanner	To loose and tighten the screws and nuts

Table 3.1 : Equipments Used and Its Function

3.3.3 Experimental Setup

Figure 3.1 : Setup for Catalytic Pyrolysis

Figure 1 shows the setup of the catalytic pyrolysis experiment. The setup consist of a semi-batch reactor which was externally heated by the electric vertical furnace. Nitrogen gas flow is used in order to drive out all the oxygen in the reaction. In the reactor, there is a borosilicate tube contained sieved oil palm frond, glass wool and GNF catalyst as well as the thermocouple embedded in the tube. The tube is a place where pyrolysis occurred. A condenser which was placed in a box of ice is connected to the reactor to allow the collection of bio-oil. At the end of the process, bio-char is produced inside the tube as the by-product while the bio-oil is collected in the condenser.

3.3.4 Substance and Chemicals

The list of substance and chemicals that will be used in the experiment are :

1. Oil palm frond (sample)
2. Graphite nanofibers (catalyst)
3. Nitrogen gas
4. Ice cube
5. Methanol

3.3.5 Range of Variables

There will be some variables that will be manipulated and kept fixed in the experiment. The variables that have been identified are summarised in the table.

Manipulated variable :

Temperature (°C)	Catalyst Loading (%)
500 (A1)	1 (B1)
400 (A2)	5 (B2)
300 (A3)	12 (B3)

Table 3.2 : Manipulated Variables

Fixed variable :

Type of GNF	Batch 1
Nitrogen Flow Rate	60 ml/min
Heating Rate	20°C/min
Experiment Duration	30 minutes

Table 3.3 : Fixed Variables

Design of Experiment :

From the previous research (Vi et al., 2013), the temperature of the reaction and the amount of catalyst loading are varied. Hence, this experiment is designed to vary the same parameter as mentioned while the nitrogen flow rate and heating rate are kept fixed. A 60 ml/min of nitrogen flow rate which is the optimum based on Vi et al., (2013) and a 20°C/min heating rate are the fixed values used for every run of the experiment.

Run	Variables Test
1.	A1,B1
2.	A1,B2
3.	A1,B3
4.	A2,B1
5.	A2,B2
6.	A2,B3
7.	A3,B1
8.	A3,B2
9.	A3,B3

Table 3.4 : Variables Test

3.3.6 Experiment Procedure

The procedure for catalytic pyrolysis of oil palm frond using semi batch reactor are as follow :

1. The sample (the oil palm frond) is firstly grinded and sieved to a particle size of <500μm

2. The sample is dried in the oven at 105°C overnight before being used in the pyrolysis experiment to remove the moisture content.
3. The main switch (power supply) and fan are switched on.
4. The weight of the borosilicate tube, condenser and the pipe connecting condenser to the reactor are measured before the experiment.
5. An amount 15g of the sample is filled in the borosilicate tube. A glass wool as well as the thermocouple are inserted in the tube. A 1% (0.15g) of catalyst (GNF) is measured and put around the glass wool inside the tube. Then, the completed tube is put in the furnace.

Figure 3.2 : Sample

Figure 3.3 : Sample in Reactor

6. The tube is tighten using the screw and it is purged for 5 minutes with nitrogen gas flow of 500 ml/min to drive out all the oxygen.
7. For the first run, the desired temperature of 500°C is set. The furnace is heated at 20°C/min as the heating rate. The nitrogen flow rate is changed to 60 ml/min when the heating process started.
8. Gas from the reaction is carried out by nitrogen and passed through a condenser with the temperature maintained at 0°C where the condensable vapour is condensed to form bio-oil.
9. The reactor is left to cool to room temperature after the completion of experiment while nitrogen flow still continued.
10. The weight of the tube, condenser and pipe after the reaction are measured to identify the weight of the bio-char and bio-oil produced after the reactions.
11. The experiment are repeated by according to the parameter varied.

When the experiment is being carried out, there are some precaution steps :

1. After the reaction is done, do not open the furnace immediately because the will cracked when the temperature of reactor and environment are still not in equilibrium condition
2. The optimum quantity for the biomass and catalyst are 15-30 grams only. If the quantity is more than that, leaking will happen.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characterization Result

4.1.1 FESEM

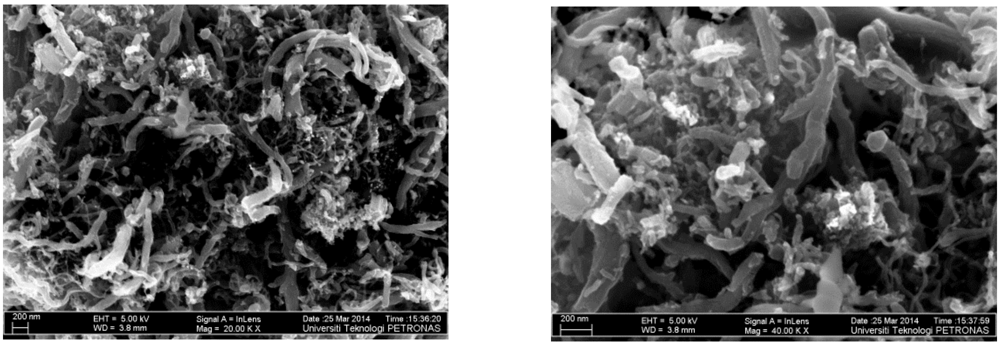

Figure 4.1 : FESEM Result of GNF

4.1.2 TEM

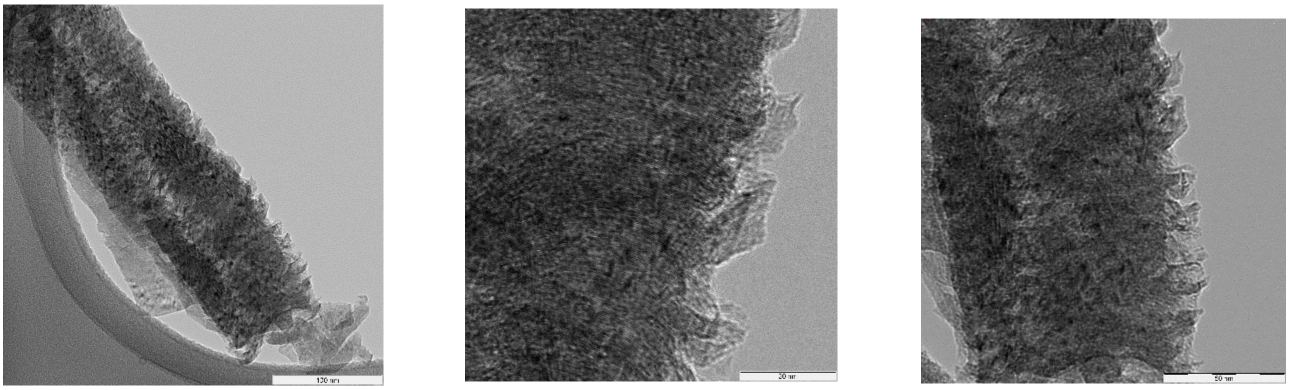

Figure 2.2 : TEM Result of GNF

4.2 Catalytic Pyrolysis Result

4.2.1 First run

Variables : A1, B1

Temperature : 500°C

Catalyst Loading : 1%

	Weight ,g (Before)	Weight ,g (After)
Condenser	518.48	524.34
Pipe	36.05	36.87
Char Container	95.63	99.64

4.2.2 Second run

Variables : A1, B2

Temperature : 500°C

Catalyst Loading : 5%

	Weight ,g (Before)	Weight ,g (After)
Condenser	518.25	524.15
Pipe	36.05	37.11
Char Container	88.50	92.75

4.2.3 Third run

Variables : A1, B3

Temperature : 500°C

Catalyst Loading : 12%

	Weight ,g (Before)	Weight ,g (After)
Condenser	518.16	524.56
Pipe	34.95	35.74
Char Container	84.77	89.14

4.2.4 Fourth run

Variables : A2, B1

Temperature : 400°C

Catalyst Loading : 1%

	Weight ,g (Before)	Weight ,g (After)
Condenser	517.70	523.87
Pipe	36.05	37.05
Char Container	88.50	89.52

4.2.5 Fifth run

Variables : A2, B2

Temperature : 400°C

Catalyst Loading : 5%

	Weight ,g (Before)	Weight ,g (After)
Condenser	517.75	523.97
Pipe	36.17	37.05
Char Container	85.91	89.95

4.2.6 Sixth run

Variables : A2, B3

Temperature : 400°C

Catalyst Loading : 12%

	Weight ,g (Before)	Weight ,g (After)
Condenser	517.90	524.35
Pipe	36.07	36.88
Char Container	84.77	88.99

4.2.7 Seventh run

Variables : A3, B1

Temperature : 300°C

Catalyst Loading : 1%

	Weight ,g (Before)	Weight ,g (After)
Condenser	518.26	523.71
Pipe	36.07	36.86
Char Container	84.74	89.65

4.2.8 Eight run

Variables : A3, B2

Temperature : 300°C

Catalyst Loading : 5%

	Weight ,g (Before)	Weight ,g (After)
Condenser	518.19	524.38
Pipe	36.06	36.16
Char Container	84.76	89.71

4.2.9 Ninth run

Variables : A3, B3

Temperature : 300°C

Catalyst Loading : 12%

	Weight ,g (Before)	Weight ,g (After)
Condenser	518.28	523.82
Pipe	36.07	36.86
Char Container	84.76	90.01

4.3 Overall Result

Run	W_{liquid}(g)	W_{char}(g)	$W_{gas+liquid}$(g)	Conversion (%)	Selectivity (%)	$Yield_{liquid}$ (%)	$Yield_{gas\ product}$ (%)
1	6.68	4.01	10.99	73.27	60.78	44.53	28.74
2	6.96	4.25	10.75	71.67	64.74	46.40	25.27
3	7.19	4.37	10.63	70.87	67.64	47.94	22.93
4	7.17	1.02	13.98	93.20	51.29	47.80	45.40
5	7.20	4.04	10.96	73.07	65.69	48.00	25.07
6	7.26	4.22	10.78	71.87	67.35	48.40	23.47
7	6.24	4.91	10.09	67.27	61.84	41.60	25.67
8	6.29	4.95	10.05	67.00	62.59	41.94	25.06
9	6.33	5.25	9.75	65.00	64.92	42.20	22.80

Table 4.1 : Overall results of the experiment

4.4 Calculation

The equations used to calculate the yield are as follow:

$$W_{liquid} = (W_{condenser-after} - W_{condenser-before}) + (W_{pipe-after} - W_{pipe-before}) \quad (1)$$

$$W_{char} = W_{char\ containter-after} - W_{char\ container-before} \quad (2)$$

$$W_{gas+liquid} = W_{sample} - W_{char} \quad (3)$$

$$Conversion = \frac{W_{gas+liquid\ product}}{W_{sample}} \times 100 \quad (4)$$

$$Selectivity_{liquid\ product} = \frac{W_{liquid}}{W_{gas+liquid\ product}} \times 100 \quad (5)$$

$$Yield_{liquid} = (Selectivity_{liquid\ product} \times Conversion)/100 \tag{6}$$

$$Yield_{gas\ product} = Conversion - Yield_{liquid\ product} \tag{7}$$

4.5 Discussion

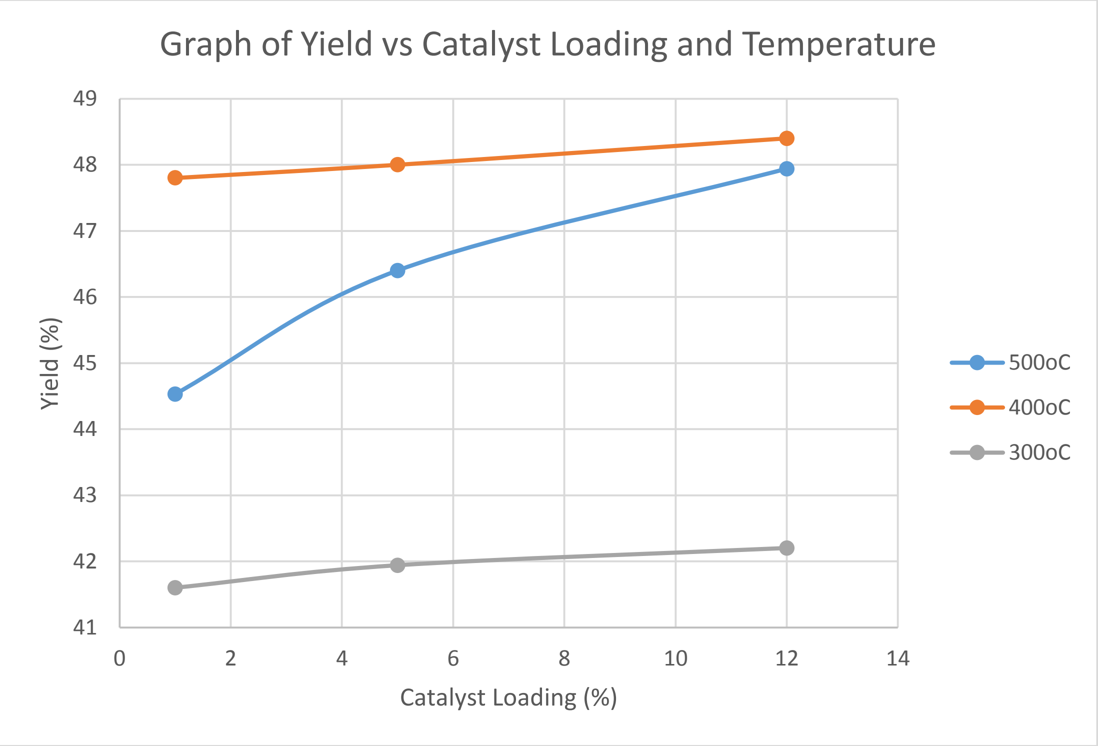

Figure 4.3: Yield of Bio-oil with Different Catalyst Loading and Temperature

Figure 4.3 shows the yield of bio-oil produced with different amount of catalyst loading and temperature. Based on the result of the nine runs, the yield of the liquid product increase as the amount of catalyst loading increase. This is because the GNF usage as the catalyst increase the rate of reaction of converting the sample into bio-oil. The higher the amount of catalyst used, the greater the yield of the liquid produced. However, the yield of the gas product decreased as the amount of catalyst loading increase. Even though the conversion of the sample using 1% of catalyst is higher compared to the sample using 12% of catalyst, the selectivity of the 12% catalyst used is still the highest among the others due to the higher presence of catalyst. The highest liquid product yield is 48.4% which was obtained at run number 6 by using 12% catalyst loading at 400°C. Whereas, the highest gas product yield is 45.4% which was obtained at run number 4 by using 1% catalyst loading at 400°C.

The temperature of the reaction does effect the liquid yield as well. From the graph, it can be seen that temperature at 400°C gives the highest liquid product yield while temperature at 300°C gives the lowest liquid product yield. The increment of temperature from 300°C to 400°C produce significant results in term of liquid product yield with 6.2% difference. For temperature between 500°C and 400°C at 1% catalyst loading, the liquid product yield also shows significant difference by 3.27%. However at 12% catalyst loading at the same temperature range, there is only small margin between the liquid product yield with only 0.46% margin.

Based on the research done by Vi etl al., (2013) which used zeolites as the catalyst, the range of the liquid product yield obtained are between 31%-38%. Compared to the results obtained in this experiment which used GNF as the catalyst, the liquid product yield are above 41% which is higher. This proves that graphite nanofiber (GNF) are the better catalyst than zeolites which is the common catalyst used in pyrolysis. Hence, for this experiment the best operating condition to run the catalytic pyrolysis is at 400°C with 1% catalyst loading. Despite the higher liquid yield obtained at 12% catalyst loading, 1% catalyst loading is chosen since there is not much different of liquid product with only 0.46% margin. In addition, the catalyst is quite expensive which suggest that we can used lesser amount of catalyst for the same liquid yield.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

As a conclusion, this project is important as it deals with the alternative way of producing bio-fuels which can be the successor of current fossil fuel usage as the main source through the process of catalytic pyrolysis. The usage of biomass (oil palm frond) is hopefully can solve the problem to manage the waste from palm oil crops. The involvement of graphite nanofiber (GNF) incorporated in the pyrolysis process is believed to be effective in favouring the high yield of liquid product (bio-oil).

The results shows that oil palm frond can be used to produce bio-oil and the incorporation of GNF catalyst in the reaction does give high and better yield of liquid product which is above 41%. It can be concluded that the best operating condition to run the catalytic pyrolysis using oil palm frond as the biomass is at the temperature of 400°C with 1% catalyst loading.

For the recommendation, it is suggested to upgrade the reactor used so that more sample can be run in one time. Other than that, a method to capture the gas product should be invented to measure the exact amount of the gas produced from the reaction. The experiment took quite a long time to be conducted for the next run because the reactor took a long time to cool down from the high temperature set. Besides, the cleaning process of the equipment such as cleaning the tube from the residual using methanol took a long time as well because the residual from the process are hard to be removed. Hence, it is recommended that there have to be more set of borosilicate tube ready to be used to save time to run the experiment.

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APPENDICES

APPENDIX 1 : Sample Calculation

The sample of calculation (1st run) are as follow :

$$W_{liquid} = (524.34 - 518.48) + (36.87 - 36.05) = 6.68g$$

$$W_{char(residue)} = 99.64 - 95.63 = 4.01g$$

$$W_{gas+liquid} = 15 - 4.01 = 10.99g$$

$$Conversion = \frac{W_{gas+liquid\ product}}{W_{sample}} \times 100 = \frac{10.99}{15} \times 100 = 73.27\%$$

$$Selectivity_{liquid\ product} = \frac{W_{liquid}}{W_{gas+liquid\ product}} \times 100 = \frac{6.68}{10.99} \times 100 = 60.78\%$$

$$Yield_{liquid} \frac{= (Selectivity_{liquid\ product} \times Conversion)}{100} = \frac{60.78 \times 73.27}{100}$$

$$= 44.53\%$$

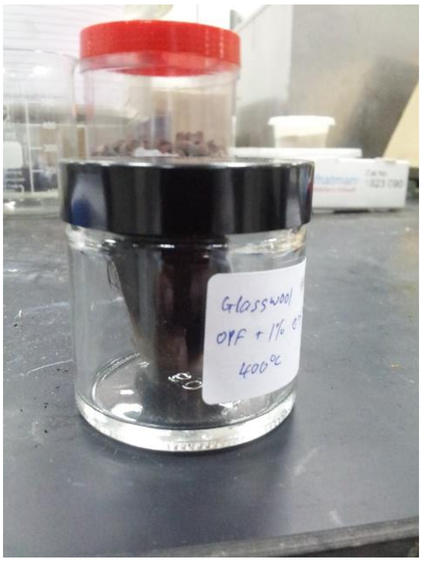
$$Yield_{gas\ product} = Conversion - Yield_{liquid\ product} = 73.27 - 44.53 = 28.74\%$$

APPENDIX 2 : Experiment Product

Oil palm frond sample weight

Bio-Oil

Bio-Char



Glass wool after reaction