

## Catalytic Pyrolysis of High-density Polyethylene (HDPE) by using Multiple Stage Reactor

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**Abstract:** Plastics have rapidly gained favor with the general public due to their high durability, low production cost, and lightweight nature. But it has a major problem during disposal treatment which leads to negative impacts on health and the environment. This pyrolysis process converts the plastic into energy (liquid black oil) and value-added products (char). There is a growing interest in the pyrolysis of waste plastics, where the plastic is thermally degraded at moderate temperature (~450 °C) in the absence of oxygen to produce shorter molecular chains and low molecular weight molecules. This project is conducted to investigate the correlation of multiple stage reactors with the effect of temperature on the yield and quality of produced liquid oil. Thus, to synthesize and characterize the yield, Fourier Transform Infrared Spectroscopy (FTIR), latent heat, density, and viscosity are used to find the result. This project used a multistage reactor and nickel catalyst on the reaction for this project and the method may be a shift methodology with multiple heating steps to realize bound technical advantages. shift is mostly thought of to be endothermic though it includes exothermic and endothermic reactions. With only nine samples of yield, the output yields then go through the analysis with latent heat of blank nickel catalyst at 150 °C the absorption process of heat happened and with three nickel catalyst showed that the result is better (29.288kJ/kg) with three nickel catalyst compared with other numbers of nickel catalyst. In FTIR, one nickel catalyst, the FTIR result shows all the peaks in the spectrum have a wide range compared with other numbers of nickel catalyst. So, the yield is produced much better according to the spectrum graph for the comparison of the yield. The viscosity shows the result shows that with one nickel catalyst is suitable and perform for the viscosity of HDPE oil because the structure not much solid because at temperature of 250 °C, the maximum of centipoise value at 61.7 cP with the temperature drop into 26.6 °C. The result of density, blank nickel catalyst, at 150 °C was the better density with 0.9512g/cm<sup>3</sup> of yield. This density was suitable and easy for the oil to react with the combustion process because the HDPE oil had different types of structure when it used a different number of nickel catalysts. Back to one nickel catalyst, it also at 150 °C was better density with 0.9588 g/cm<sup>3</sup> compared with other temperatures. It is not

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too much different with blank nickel catalyst with the process of pyrolysis happen and the lastly on three nickel catalyst, at 150 °C is the good type of density and this show that all the process at 150 °C is consume the best density for HDPE oil to use in the industry.

**Keywords:** Temperature, Yield, Mass, Oil, Project, Pyrolysis, Time, Yields

## 1. Introduction

Plastics is easy to find and also need some effort to recycle of use it back for beneficial for our daily usage. Plastics are a regular cluster of artificial or natural materials, made from high molecular chains comprises carbon as sole or major element. Plastics are extremely durable, strong, elastic and fewer high-ticket to produce, that build them ideal selection for packaging and storage applications. the world production of plastics keeps on increasing, so will the waste generation. The period of time span of plastics product varies from weeks for packaging to many years for building appliances [1]. Like any other technological trend, engineering professions play an important role in plastic waste treatment. Discarded plastic products and packaging materials account for an increasing proportion of urban waste. Plastic waste in households is mainly composed of low-density polyethylene (LDPE), high-density polyethylene (HDPE), polyethylene terephthalate (PET), polypropylene (PP), polystyrene (PS) and polystyrene composed of vinyl chloride (PVC) [2]. In this study, the type of plastic that we used were from polyethylene plastics.

However, catalysts can play a critical role in the thermochemical processing of waste plastics in terms of promoting targeted reactions, reducing reaction temperature and improving process system efficiency. Thus, the most commonly investigated catalysts are solid acid catalysts, for example, zeolite ZSM-5, Y-zeolite and MCM-41. However, this study uses a multistage reactor comprising a reactor inside cylindrical housing given body of water and outlet openings for current flow of liquid reactants and product pyrolysis process including nickel catalyst for selective chemical change of poly-unsaturated compounds fashioned throughout steam cracking. The produced liquid oil was analyzed for various parameters. The dynamic viscosity of liquid oil was found to be in the range of 1.66–3.02 mPa. s from all studied conditions. The value of 1.77 mPa.s was observed for the liquid oil produced at optimum conditions of 450°C for 75 min. According to [3], liquid oil produced from the pyrolysis of plastic has a lower kinematic viscosity than diesel fuel due to the presence of a high fraction of gasoline and a low fraction of heavy oil. This study is to emphasize using nickel catalyst for at optimum temperature and reaction time to produce pyrolysis oil.

In this study, a catalyst was employed to study its effect on the pyrolysis of HDPE plastics. The catalysts used is nickel catalysts. Hence, the catalyst was investigated with High Density Polyethylene (HDPE). This plastic type was chosen as it is a very common plastic in Malaysian streams. From previous studies, the acceptable temperature to obtain liquid fuel from plastic pyrolysis is 500°C, however different plastic types have different decomposition temperatures. Therefore, in this study, pyrolysis was conducted at three different temperatures between 150°C, 200°C and 250°C to investigate the effect of temperature on pyrolysis of high-density polyethylene (HDPE). The objective of this research is about to investigate the correlation between reaction temperature and retention time for multistage reactors in catalytic pyrolysis and liquid oil yield. The characterize the physical and chemical properties of pyrolysis oil and also to establish a design of experiment (DOE) of producing pyrolysis oil and the synthesise and characterize the yield by sing Fourier Transform Infrared Spectroscopy (FTIR), latent heat, density, and viscosity.

## 2. Materials and Methods

The materials and methods contain the methodology that will be used for this project to find out the result and analysis according to parameters that will be discussed.

## 2.1 Material of this study

This study examined the process of pyrolysis with Nitrogen ( $N_2$ ) and nickel catalyst to reform a type of biodiesel oil by using the high-density polyethylene (HDPE) plastics. The material has density  $0.965 \text{ g/cm}^3$  ( $23 \text{ }^\circ\text{C}$ ) and a soft flow index  $7.5 \text{ g/10 min}$ . The data contains ultraviolet light stabilizations and recommends its process temperature to be  $220 - 270 \text{ }^\circ\text{C}$  [4].

Pyrolysis of plastics is mostly administered at a quick heating rate to a final temperature up to  $700 \text{ }^\circ\text{C}$  or at moderate temperature ( $\sim 500 \text{ }^\circ\text{C}$ ) in presence of a catalyst. Below are the physical properties of HDPE and PP material. [1].

**Table 1: Physical properties of HDPE and PP material [1]**

Plastics	Density ( $\text{kg/cm}^3$ )	Melting point ( $^\circ\text{C}$ )
HDPE	950	$132 \pm 1$
PP	900	$168 \pm 1$



**Figure 1: High-density polyethylene (HDPE) plastic**

## 2.2 Nickel catalyst

This method produces char, gas, and liquid products. The liquid product (bio-oil) will be upgraded to transportation fuel through varied processes together with chemical process cracking, hydrodeoxygenation (HDO), emulsification, and esterification [5]. Based on Figure 2, this one nickel catalyst is just 8 gram of weight and this is the reason why this catalyst was chosen compared to other catalysts like previously wanting to use a catalytic converter.

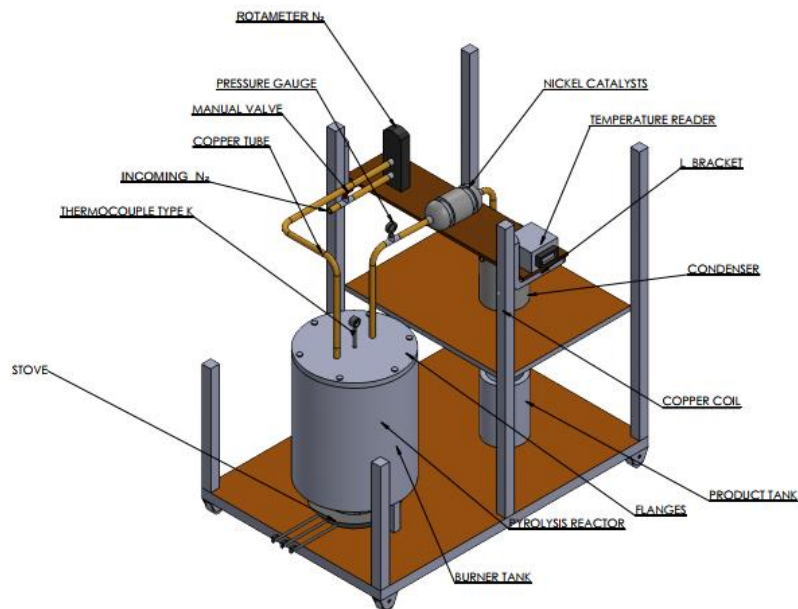


**Figure 2: Mass of nickel catalyst**

## 2.3 Method of multistage reactor test rig bench

The multistage reactor test rig bench working starting from the main body burner tank and reactor where the feedstock of high-density polyethylene (HDPE) is put in. Then, Nitrogen gas purged into the

reactor. The reaction of plastic is then to burn out and then flow to nickel catalyst by increasing the conversion to CO<sub>2</sub>, char and etc. Also, the condenser reaction to recover oil from vapor when the process of pyrolysis oil flow. After the overall process is complete, next for collecting the sample for testing on FTIR, latent heat, density, and viscosity.



**Figure 3: Isometric view of multistage reactor test rig bench**

#### 2.4 Instrumentation analysis

There are many types of characterizations that can be used for analyzing the result based on this method specially to measure density, viscosity, latent heat, and Fourier transform infrared spectroscopy (FTIR) for analysis where the product will refer to criteria that should be happening according to the parameter. To collect the data with specific method, design of experiments (DOE) will be applied in this project to pick the number of experimental parameters and the result.

### 3. Results and Discussion

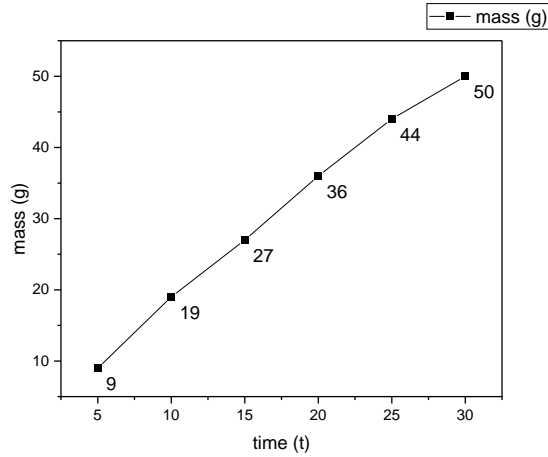
The high-density polyethylene (HDPE) was preferred to use in this pyrolysis oil process because the melting point from this plastic reaction occurs at 125 °C and it is easy to react at this point. The reaction from nitrogen gas and firing process outside the reactor starting from 150 °C, 200 °C, and 250 °C were sufficient to release the pyrolysis oil with the nickel catalyst reaction. The parameters in this process used a blank nickel catalyst, one nickel catalyst (8 gram) and three nickel catalysts (24 gram). All of the processes then come out with retention time of yields and other analysis from density, viscosity, latent heat and Fourier-transform infrared spectroscopy (FTIR).

#### 3.1 Retention time of yield

Retention time is the time that a substance spends in a very column or it is often outlined because the time spent within the stationary and mobile phases. The longer retention time depends on the interaction of the analyte with the stationary phase. The stronger the interaction, the additional will be the interaction time [6]. All the yield sample then was recorded into a graph to see mass and time taken of the pyrolysis oil yields according to specific time (30 minutes).

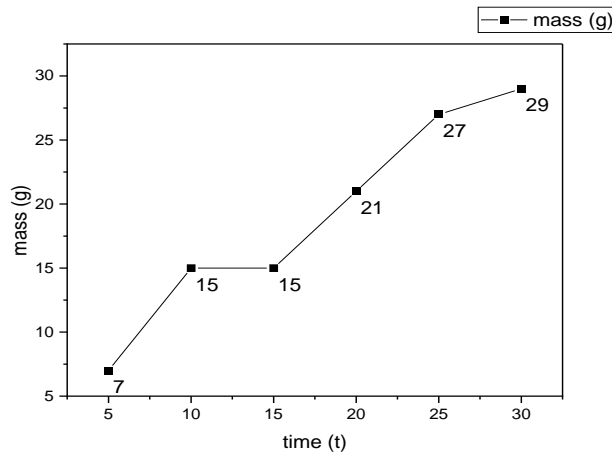
#### 3.2 Blank nickel catalyst effect on mass and time yields

Based on Figure 4, there are retention times where pyrolysis yields occur on certain temperatures. Starting with a temperature of 150 °C, pyrolysis yields mass reaching 9 gram of oil in a range of 5 minutes of time. The pyrolysis process yields then increases by 19 gram of oil and it shows in 10 minutes of time, the flow still occurs and the process with the nitrogen purge in the reactor chamber to eliminate oxygen when the pyrolysis is running.



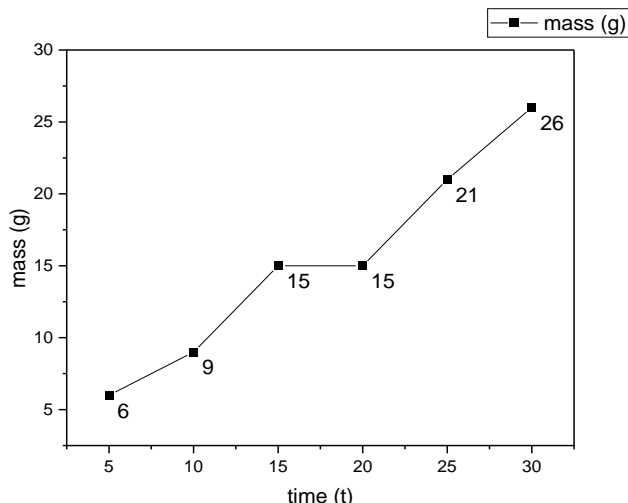
**Figure 4: Retention time of 150°C with blank nickel catalyst of yields mass**

Figure 5, the retention time of 200 °C temperature shows the trend mass of yields also increases and when it reaches 5 to 10 minutes, the mass of yield on static value is 15 g of mass and it may be because of flow rate inside the copper tube affecting the oil process. Then, the flow continues to increase until the last 30 minutes of range.

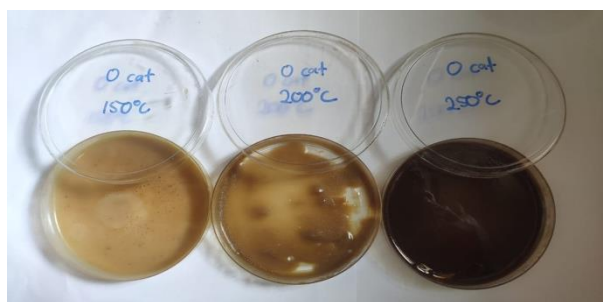


**Figure 5: Retention time of 200 °C with blank nickel catalyst of yields mass**

From Figure 6, the oil yields appear with 6 gram of oil in 5 minutes of time. At a high maximum temperature of 250 °C, the mass of yield slowly drops into 26 grams when it reaches 30 minutes of time. This process occurs because the HDPE plastic has maximum burn and the quantity of the plastic does not generate more oil.



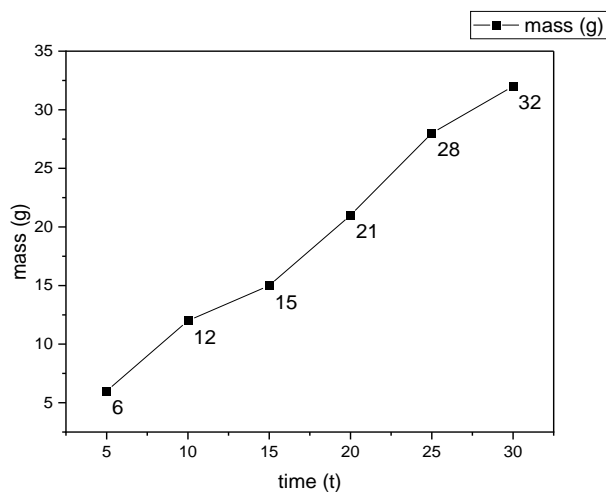
**Figure 6: Retention time of 250 °C with blank nickel catalyst of yields mass**



**Figure 7: HDPE oil with 0 nickel catalyst**

### 3.3 One nickel catalyst effect on mass and time yields

Based on Figure 8, the yield come out with 6 gram of oil in duration of 5 minutes. The graph shows the increasing of yields on 12 gram and the trend of the graph more increase with the suitable temperature of 150 °C. The range of mass just around 15 to 32 grams because the starting point of burning need reactor chamber and Nitrogen to push the liquid flow. The mass of HDPE plastics around  $\pm 300$  g for burning process to produce pyrolysis oil.



**Figure 8: Retention time of 150°C with one nickel catalyst of yields mass**

By refer to Figure 9, 200 °C on the left graph, the mass of 200 °C starting with 6 grams of yield. This flow of oil inside the copper tube no much faster compared with temperature previously. The increasing also slow and the final mass of the pyrolysis yield on 30 grams. This result of the pyrolysis process almost slow to produce more yield. Same with 250 °C on the right graph, the trend of the graph shows the mass of yield just produce 5 gram in 5 minutes of time and the final 30 minutes of time show the yield only take 28 grams.

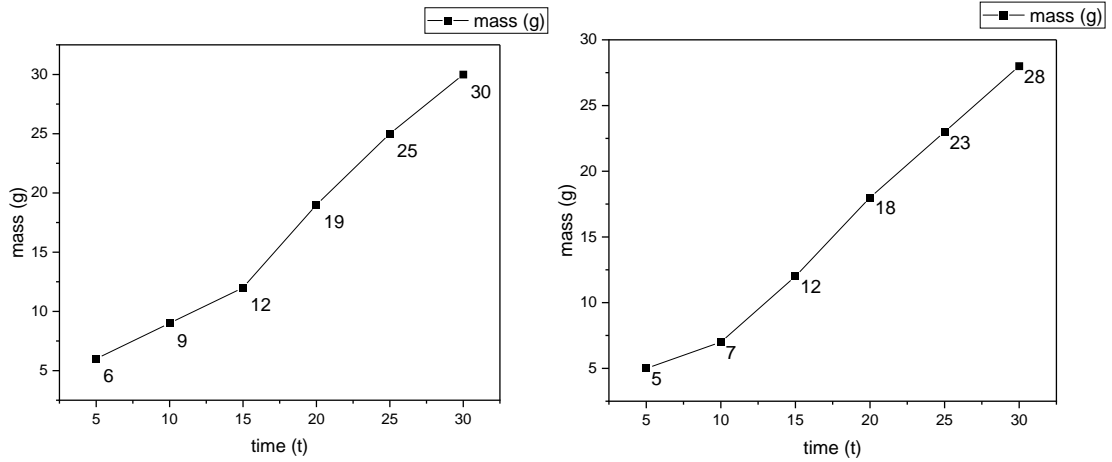


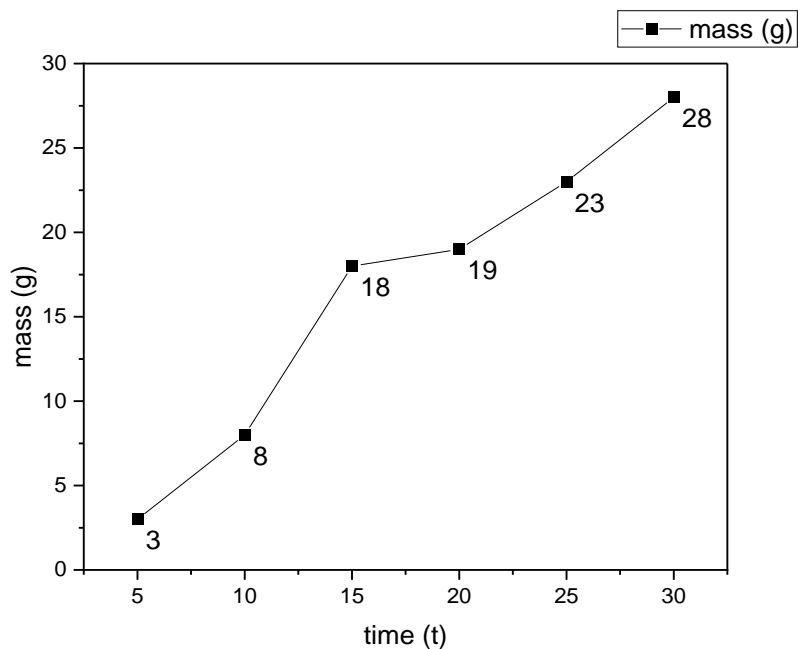
Figure 9: Retention time of 200°C (left) and 250°C (right) with one nickel catalyst of yields mass



Figure 10: 1 nickel catalyst yield with different temperature

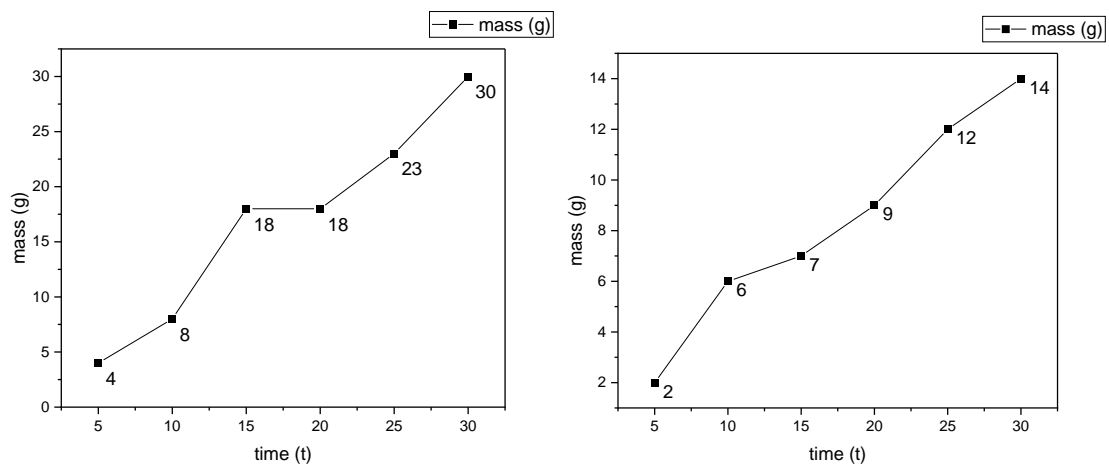
### 3.4 Three nickel catalyst effect on mass and time yields

Based on Figure 11, the yields come out with 3 gram of oil in duration of 5 minutes. The graph shows the increasing of yields on 8 gram and the trend of the graph more increase with the suitable temperature of 150 °C. The range of mass just around 18 to 28 gram and Nitrogen to push the liquid flow starting slow. The mass of HDPE plastics around ± 300 g for burning process to produce pyrolysis oil.



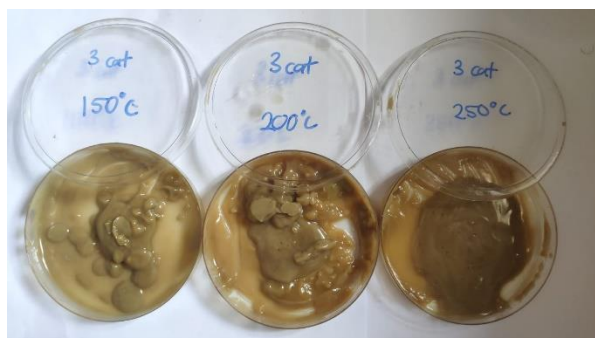
**Figure 11: Retention time of 150 °C with three nickel catalyst of yields mass**

When it reaches 200 °C based on figure below, the mass starting with 4 grams of yield. This flow of oil inside the copper tube no much faster compared with temperature previously. The increasing also slow and the final mass of the pyrolysis yield on 30 grams. This result of the pyrolysis process almost slow to produce more yield. Same with 250 °C on the right figure, the trend of the graph shows the mass of yield just produce 2 gram in 5 minutes of time and the final 30 minutes of time show the yield only take 14 grams.



**Figure 12: Retention time of 200 °C (left) and 250 °C (right) with three nickel catalyst of yields mass**





**Figure 13: 3 nickel catalyst yield with different temperature**

### 3.5 Design of experiment of nickel catalyst on temperature for reforming yields

Based on the table below, there are analysis that combine in the table to identify the characteristics of HDPE pyrolysis oil which is testing under analysis of the lab. Each data that lists on the table is the summary of the analysis after identify the result of each analysis.

**Table 2: Blank nickel catalyst analysis**

Temperature	150°C	200°C	250°C
Density	0.9512 g/cm <sup>3</sup>	0.9520 g/cm <sup>3</sup>	0.9532 g/cm <sup>3</sup>
Viscosity	Medium	Medium	Medium
FTIR	2924.45cm <sup>-1</sup> , 68.37 %T	2925.04 cm <sup>-1</sup> , 71.76 %T	2921.13 cm <sup>-1</sup> , 58.81 %T
Latent heat	30.334 kJ/kg	30.334 kJ/kg	30.334 kJ/kg

**Table 3: One nickel catalyst analysis**

Temperature	150°C	200°C	250°C
Density	0.9588 g/cm <sup>3</sup>	0.966 g/cm <sup>3</sup>	0.9664 g/cm <sup>3</sup>
Viscosity	Medium	Medium	High
FTIR	2926.83 cm <sup>-1</sup> , 70.59 %T	2922.66 cm <sup>-1</sup> , 64.92 %T	2921.75 cm <sup>-1</sup> , 62.27 %T
Latent heat	30.334 kJ/kg	30.334 kJ/kg	29.288 kJ/kg

**Table 4: Three nickel catalyst analysis**

Temperature	150°C	200°C	250°C
Density	0.9716 g/cm <sup>3</sup>	0.9724 g/cm <sup>3</sup>	0.9728 g/cm <sup>3</sup>
Viscosity	Medium	High	High
FTIR	2921.84 cm <sup>-1</sup> , 68.36 %T	2920.09 cm <sup>-1</sup> , 53.33 %T	2920.37 cm <sup>-1</sup> , 59.51 %T
Latent heat	29.288 kJ/kg	29.288 kJ/kg	28.242 kJ/kg

### 3.6 Density of the yields

Pyrolysis of high-density polyethylene (HDPE) by using different number of nickel catalyst show that the density on each yield is different. Each pyrolysis yields then will test under density to find the mass of sample and the volume of pycnometer (25 ml) and the temperature about 20 °C. Below is the formula and calculation of density to find HDPE oil with different temperature and number of nickel catalyst.

$$\rho = \frac{m}{v} \tag{Eq.1}$$



**Figure 14: Mass of pycnometer and oil sample**

Temperature of oil: 20°C

Pycnometer mass: 9.83 g

Based on blank nickel catalyst, at 150 °C was the better density with 0.9512g/cm<sup>3</sup> of yield. This density was suitable and easy for the oil to react with the combustion process because the HDPE oil had different types of structure when it used a different number of nickel catalysts. Back to one nickel catalyst, it also at 150 °C was better density with 0.9588g/cm<sup>3</sup> compared with other temperatures. It is not too much different with blank nickel catalyst with the process of pyrolysis happen and the lastly on three nickel catalyst, at 150 °C is the good type of density and this show that all the process at 150 °C is consume the best density for HDPE oil to use in the industry.

**Table 5: Calculation of density with different temperature and number of nickel catalyst**

Temperature	Number of catalysts		
	0 catalyst	1 catalyst	3 catalysts
150°C	$\rho = \frac{m}{v}$	$\rho = \frac{m}{v}$	$\rho = \frac{m}{v}$
	$= \frac{23.78 \text{ g}}{25 \text{ ml}}$	$= \frac{23.97 \text{ g}}{25 \text{ ml}}$	$= \frac{24.29 \text{ g}}{25 \text{ ml}}$
	$= 0.9512 \text{ g/cm}^3$	$= 0.9588 \text{ g/cm}^3$	$= 0.9716 \text{ g/cm}^3$
200°C	$\rho = \frac{m}{v}$	$\rho = \frac{m}{v}$	$\rho = \frac{m}{v}$
	$= \frac{23.80 \text{ g}}{25 \text{ ml}}$	$= \frac{24.15 \text{ g}}{25 \text{ ml}}$	$= \frac{24.31 \text{ g}}{25 \text{ ml}}$
	$= 0.952 \text{ g/cm}^3$	$= 0.9660 \text{ g/cm}^3$	$= 0.9724 \text{ g/cm}^3$
250°C	$\rho = \frac{m}{v}$	$\rho = \frac{m}{v}$	$\rho = \frac{m}{v}$
	$= \frac{23.81 \text{ g}}{25 \text{ ml}}$	$= \frac{24.16 \text{ g}}{25 \text{ ml}}$	$= \frac{24.32 \text{ g}}{25 \text{ ml}}$
	$= 0.9524 \text{ g/cm}^3$	$= 0.9664 \text{ g/cm}^3$	$= 0.9728 \text{ g/cm}^3$

### 3.7 Viscosity measurements

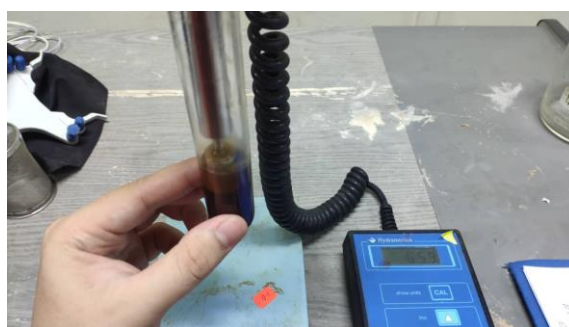
Below is the data analysis viscosity of HDPE oil where it measures temperature, temperature-corrected viscosity, and live viscosity.

**Table 6: Blank nickel catalyst**

Temperature	Viscosity		
	Temperature	Live viscosity	Temperature-corrected viscosity
150°C	27.7°C	23.1 cP	23.8 Pa.s
200°C	27.4°C	23.5 cP	31.0 Pa.s
250°C	26.7°C	65.9 cP	95.7 Pa.s

**Table 7: One nickel catalyst**

Temperature	Viscosity		
	Temperature	Live viscosity	Temperature-corrected viscosity
150°C	27.5°C	24.5 cP	23.5 Pa.s
200°C	27.3°C	25.2cP	27.4 Pa.s
250°C	26.6°C	61.7cP	97.9 Pa.s

**Figure 15: Viscosity test on HDPE pyrolysis oil**

Viscosity test on HDPE oil to analyze the viscosity of oil and the suitable number of nickel catalyst and the temperature. When the viscosity is lower viscosity, the friction of oil is reduced and easy for the combustion process. The result shows that with one nickel catalyst is suitable and perform for the viscosity of HDPE oil because the structure not much solid because at temperature of 250 °C, the maximum of centipoise value at 61.7 cP with the temperature drop into 26.6 °C. so, it clearly the optimum specification of viscosity to use for the combustion of HDPE oil

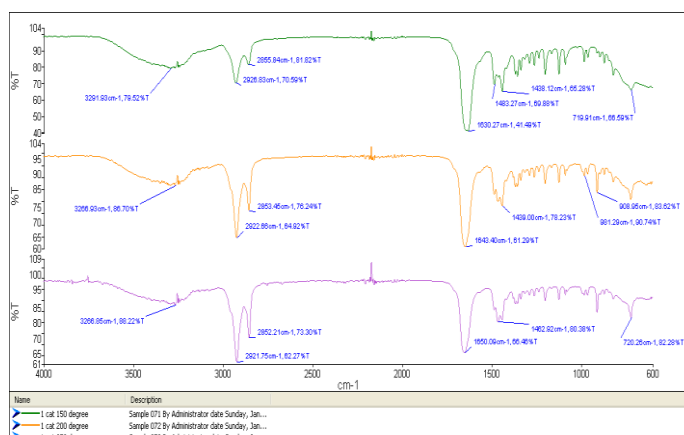
**Table 8: Three nickel catalyst**

Temperature	Viscosity		
	Temperature	Live viscosity	Temperature-corrected viscosity
150°C	27.5°C	27. 5cP	31.0 Pa.s
200°C	27.3°C	28.1 cP	65.9 Pa.s
250°C	26.5°C	67.3 cP	112.6 Pa.s

### 3.8 Fourier-transform infrared spectroscopy (FTIR) test of the yields

Figure 16 show the combination of three different type of temperature of blank catalyst with different colour of line graph to compare the wavenumber ( $\text{cm}^{-1}$ ) value and transmittance. On 1500 to 4000  $\text{cm}^{-1}$ , the region calls functional group region and all three types of temperature shows three peaks

each on them. By starting with 150°C line on red colour, the absorbance peaks value shows the value is 2924.45 cm<sup>-1</sup>.



**Figure 16: Graph of spectrometer with 1 nickel catalyst**

With only one nickel catalyst, the FTIR result shows all the peaks in the spectrum have a wide range compared with other numbers of nickel catalyst. So, the yield is produced much better according to the spectrum graph for the comparison of the yield.

### 3.9 Latent heat reaction on the yields

Latent heat test was conducted by using thermometer, glass bottle, oil, and distillation water. Before start the experiment, prepare 1 gram of oil to heat on 80 +/- 5 °C. This heat process used water bath concept because oil sample cannot be on direct heat. By refer to equation below, H is that the heat free by combustion, m is the mass in grams of the water used as a heat collector, C is the heat energy of water (4.184 J/g·°C), and ΔT is the activity in °C.

$$H = m \cdot C \cdot \Delta T \tag{Eq.2}$$

**Table 9: Latent heat of HDPE pyrolysis oil calculation**

Temperature	Number of catalysts		
	0 catalyst	1 catalyst	3 catalyst
150°C	$H = m \cdot C \cdot \Delta T$	$H = m \cdot C \cdot \Delta T$	$H = m \cdot C \cdot \Delta T$
	$= 250 \text{ g} \times 4.184 \text{ J/g}^\circ\text{C} \times 29^\circ\text{C}$	$= 250 \text{ g} \times 4.184 \text{ J/g}^\circ\text{C} \times 29^\circ\text{C}$	$= 250 \text{ g} \times 4.184 \text{ J/g}^\circ\text{C} \times 28^\circ\text{C}$
	$= 30334 \text{ J/g}$ (30.334kJ/kg)	$= 30334 \text{ J/g}$ (30.334kJ/kg)	$= 29288 \text{ J/g}$ (29.288kJ/kg)
200°C	$H = m \cdot C \cdot \Delta T$	$H = m \cdot C \cdot \Delta T$	$H = m \cdot C \cdot \Delta T$
	$= 250 \text{ g} \times 4.184 \text{ J/g}^\circ\text{C} \times 29^\circ\text{C}$	$= 250 \text{ g} \times 4.184 \text{ J/g}^\circ\text{C} \times 28^\circ\text{C}$	$= 250 \text{ g} \times 4.184 \text{ J/g}^\circ\text{C} \times 27^\circ\text{C}$
	$= 30334 \text{ J/g}$ (30.334kJ/kg)	$= 29288 \text{ J/g}$ (29.288kJ/kg)	$= 28242 \text{ J/g}$ (28.242 kJ/kg)
	$H = m \cdot C \cdot \Delta T$	$H = m \cdot C \cdot \Delta T$	$H = m \cdot C \cdot \Delta T$

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250°C	= 250 g × 4.184 J/g°C × 29°C	= 250 g × 4.184 J/g°C × 29°C	= 250 g × 4.184 J/g°C × 28°C
	= 30334 J/g (30.334kJ/kg)	= 30334 J/g (30.334kJ/kg)	= 29288 J/g (29.288kJ/kg)

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**Figure 19: Latent heat test on pyrolysis oil**

When the temperature at 150 °C the absorption process of heat happened and with three nickel catalyst showed that the result is better (29.288 kJ/kg) with three nickel catalyst compared with other numbers of nickel catalyst. When the result of latent heat is low, it means the absorption process of the yield is changed from liquid into solid and the water temperature also drops when the shaking of the bottle occurs. End up the temperature was around 29 °C on the last reading.

#### 4. Conclusion

The present study is conducted by running the pyrolysis of HDPE oil with the combination of Nitrogen and nickel catalyst. The result shows all the experiments to analyze the yields need some point to achieve with the same other pyrolysis process. According to data that are achieved, the results show the pyrolysis of HDPE plastic oil can produce more yield if the optimum temperature can be achieved. Other types of thermoplastics also can be used as feedstock for the pyrolysis process because it has a specific melting point which is sure to produce for other processes in between separation types of plastic or combine.

#### Acknowledgement

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