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THE OPTIMIZATION OF NATURAL CATALYSTS IN OIL PRODUCTION FROM PLASTIC WASTE PYROLYSIS

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ABSTRAK

Penelitian ini dilakukan untuk mengetahui pengaruh jumlah katalis terhadap hasil (yield) dari pirolisis sampah plastik jenis Polypropylene (PP) serta mengetahui karakteristik minyak pirolisis sampah plastic yang meliputi sifat fisis dan kimia minyak pirolisis. Sifat fisis yang diuji meliputi viskositas, fire point, flash point, pour point, density dan water content sedangkan sifat kimia yaitu senyawa yang terkandung didalam minyak pirolisis dengan menggunakan Gas Chromathography Mass Spectrometry (GCMS) serta mengetahui nilai kalornya. Penelitian dilakukan pada jenis plastik High Density Polyethylene (HDPE) yaitu Polypropylene (PP) yang dipirolisis pada reaktor batch dengan menggunakan incenerator dimana pemanasan berasal dari luar dengan menggunakan serbuk kayu bekas dan LPG. Perbandingan bahan baku yang digunakan sebanyak 1 kg plastik dengan dengan variasi katalis zeolit 0,25; 0,5; 0,75; 1 dalam kg.

Hasil penelitian menunjukkan bahwa semakin banyak katalis yang ditambahkan maka minyak, pirolisis yang dihasilkan semakin banyak. Hasil minyak pengukuran di LEMIGAS kinematic viskositas pada suhu 40°C adalah 1.556 cST , fire point 42°C, flash point PMCC 26°C, pour point -42°C, density pada suhu 15°C 0.7857 g/cm³, dan water content adalah 507.54 ppm. Sedangkan unsur senyawa hidrokarbon yang dihasilkan berupa pentene, heptane, dan cyclohexane mempunyai sifat flammable. Nilai kalor yang dihasilkan dari minyak hasil pirolisis ini adalah sebesar 40,947 Mj/Kg. Jumlah volume minyak plastik yang maksimal didapatkan dengan menggunakan sebanyak 1 kg bahan baku dengan 1kg katalis zeolit sebanyak 955 ml.

Kata Kunci: *Pirolisis, Sifat Fisik dan Kimia, Plastik Polypropylene (PP) , Plastik HDPE katalis, zeolit.*

ABSTRACT

This study was conducted to determine the effect of the amount of catalyst on yield from the pyrolysis of Polypropylene (PP) type plastic waste and to determine the characteristics of plastic waste pyrolysis oil which includes the physical and chemical properties of pyrolysis oil. The physical properties tested include viscosity, fire point, flash point, pour point, density, and water content while the chemical properties are compounds contained in pyrolysis oil using Gas ChromatoChromatography -Massmetry (GCMS) and knowing its calorific value. The research was conducted on the type of High-Density Polyethylene (HDPE), namely Polypropylene (PP) which was hydrolyzed in a batch reactor using an incinerator where heating originated from outside using wood powder and LPG. The ratio of raw materials used is as much as 1 kg of plastic with a variation of zeolite catalyst 0.25; 0.5; 0.75; 1 in kg. The results showed that the more catalysts added, the more pyrolysis oil was produced. The results of measuring oil at LEMIGAS kinematic viscosity at 40oC are 1.556 cST, 42 oC fire point, PMCC 26 °C flash point, pour point -42 °C, density at 15 °C 0.7857 g / cm3, and water content is 507.54 ppm . While the hydrocarbon compounds produced are pentene, heptane, and cyclohexane which have flammable properties. The calorific value produced from this pyrolysis oil is 40.947 Mj / Kg. The maximum amount of plastic oil volume is obtained by using 1 kg of raw material with 1 kg of 955 ml zeolite catalyst.

Keywords: Pyrolysis, Physical and Chemical Properties, Polypropylene (PP) Plastics, HDPE catalyst plastics, zeolites **.**

1. PRELIMINARY

The waste problem is a classic problem that has always been the subject of discussion, but its handlers have not been able to solve this problem. It has been recorded that in the last 2 years Indonesia has produced 67.8 million tons of waste which according to the Ministry of Environment and Forestry, if it is presented based on the type of waste, plastic waste is in second position, namely 17% of all waste produced. On the other hand, the Central Statistics Agency (BPS) says that plastic waste in Indonesia reaches 64 million tons per year of the total waste, said the amount of plastic waste is 3.2 million tons in the sea (Sasoko DM, 2022). It is a classic problem if we discuss the waste problem. It can be said that waste is a problem whose impact is like a time bomb which is increasing day by day because plastic waste is very slow in the process of decomposition (decomposition) (Azzaki et al., 2022) . So far, the methods used, such as combustion, are still ineffective because they trigger pollutants from gas excretion emissions consisting of CO_2, CO, NO_x, and SO_x and particulates which can change the composition of the excrement content and the surrounding environment. Plastic has a high enough heat that is parallel to fossil fuels (gasoline and diesel). (Wicaksono & Arijanto, 2017) . Based on this statement, plastic waste processing can be carried out using the priolysis method to produce a new fuel source (Musta et al., 2021) . The processing of waste into a source of fuel oil uses a *batch* type *incinerator machine* whose design can be seen in Figure 1.

Converting plastic waste into fuel oil includes tertiary recycling. Converting plastic waste into fuel oil can be done by the *cracking process* . *Cracking* is the process of breaking polymer chains into lower molecular weight compounds. The results of this plastic cracking process can be used as chemicals or fuel (Panda, 2011) .

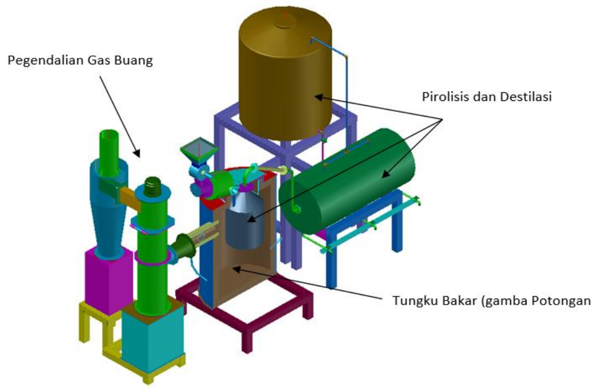

Figure 1. *Batch type incinerator* machine design

The process of converting long chain hydrocarbon, polyaromatic or polymer fractions required is a cracking catalyst which is a heterogeneous solid type catalyst. The use of pure metal catalysts has disadvantages including not having high thermal stability, relatively small surface area and clumping can occur during the catalytic process. (Wega Trisunaryati, 2016). The type of catalyst for this process is a *metal supported catalyst* which consists of a metal contained in a solid carrier pad such as silica alumina, alumina and zeolite. Natural zeolite is a material that has been widely used as a carrier. Zeolite is a mineral composed of a silica alumina tetrahedral framework in three dimensions (Panda, 2011) .

2. TOOLS AND MATERIALS

2.1 Tools

The reactor with a diameter of 600 mm and a height of 900 mm with 2 lids is used, functions to accommodate plastic raw materials which are heated to become fuel oil, see figure 2. The condenser here functions to convert gas/steam resulting from pyrolysis into liquid (fuel oil).), has 2 cooling systems that use air media around the environment , each of which has a

JBP Vol. 25, No. 2, Desember 2023- Denni Afriadi , Fazrul M Yasin
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Jurnal Biosains Pascasarjana Vol. _ (2023) pp

diameter of 100 mm and uses stainless steel steel plate material.

(A) **(B)**

Figure 2. (A) Reactor, (B) Condenser Design

In the second condenser, there is a pipe with a diameter of 25 mm and a pipe length of 700 mm with a total of 104 pipes inside the condenser, see. *The water* pump function is to pump water into the second condenser tube, the purpose is to cool the gas from burning into oil, see picture 3.

Figure 3. *Water Pump*

The fire source/ *Burner* is a supplier of heat energy to heat the pyrolysis machine with temperatures reaching 400°C - 600°C so that the plastic turns into liquid (liquid) and becomes steam, then the steam will be cooled by the condenser so that it can produce fuel oil, see picture 4.

Figure 4. *Burners*

holding tube in the form of Erlenmeyer glass is used as a product container so that the amount of plastic oil resulting from pyrolysis can be immediately known, see figure 5.

Figure 5. Tubes containing pyrolysis results

2.2 Materials

The material used is LPG (*Liquified Petroleum Gas*), waste from the woodworking industry, a type of *polypropylene* (PP) plastic waste which will be converted from plastic waste to plastic oil.

3. HOW IT WORKS

3.1 Preparation and treatment of the catalyst

HDPE (*High Density Polyethylene) plastic* type *Polypropylene* . This HDPE plastic waste is taken from a type of used plastic left over from a glass of mineral water that is thin and clear. Before being used, the plastic is first washed with soapy water and rinsed and washed with water to remove dirt attached to the plastic and then dried in the sun to remove the water. After that it is cut into small pieces to make it easier to put it in the reactor and melt the plastic in the pyrolysis process. For the preparation of the catalyst, the natural zeolite samples after being weighed are washed with distilled water to remove impurities (impurities) in them and followed by drying. The dried zeolite was ground and then sieved through a 100 mesh sieve, then soaked in 1% HF solution with a ratio of 1:2 (v/v) for 10 minutes and filtered using filter paper and washed with distilled water. Soaking in 6 N hydrochloric acid with a ratio of 1:2 (v/v) for 12 hours and then refluxing for 30 minutes at 90°C, then filtered and washed with distilled water until the pH is equal to 6. After that, the zeolite is dried using an oven at 110°C for 1 hour to remove the water

JBP Vol. 25, No. 2, Desember 2023- Denni Afriadi , Fazrul M Yasin
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content in it and followed by immersion in a solution of 0.1 M NH_4Cl with a ratio of 1: 2 (v/v) at 90°C for 3 hours per day up to one week. This zeolite was again dried in the oven at 110°C and followed by calcination (giving steam) using N2 gas for $_4$ hours at 500°C. The zeolite is ready to be characterized and used as a catalyst .

3.2 Pyrolysis Process

The tool used in this pyrolysis process is an incinerator machine that is ready-made and ready to use only in the use of fuel assisted by wood waste in the form of sawdust left over by wood craftsmen to increase heat in the combustion chamber. This is because the incinerator engine has undergone changes to include raw materials and the addition of catalysts is done manually by opening the reactor lid and closing it again in an airtight condition. After the plastic raw material used is 1 kg of plastic raw material while the catalyst is used with a variation of 0.25 kg; 0.5 kg; 0.75 kg; 1 kg. The temperature and pressure conditions are maintained to produce liquid vapor in the form of oil after passing through the distillation stage which is condensed by circulating water. The temperature is recorded starting from the ignition of the combustion chamber until the last drop that falls on the plastic oil container. Prepare raw materials that have been weighed as much as 1 kg, then put into the reactor without adding a catalyst and then adding the catalyst. The change in time per 10 minutes is recorded to determine the maximum temperature and the volume of results obtained.

The next step is to prepare a stove *(burner)* with fuel from LPG gas and position the *burner* at the bottom of the pyrolysis reactor tube. As additional heat from the combustion chamber, wood waste in the form of powder is used to raise the temperature in the reactor.

Prepare a stopwatch to measure the time needed in the pyrolysis process and record the temperature installed in the reactor according to the specified time and an infrared laser thermometer to measure / find out the temperature in each section when the test is running.

The steam from the pyrolysis flows into the condenser circuit which is then cooled by cooling water, then the condensed liquid is collected in a plastic bottle and measured using an Erlenmeyer flask.

The distillation process where the steam flowing from the pyrolysis process will undergo a steam distillation process where the condenser (coolant) contents of the condenser tube are prepared with water as a cooling medium and prepare a water pump in the reservoir to circulate the water so that the water temperature in the cooling condenser remains stable. Connect the condenser steam flow pipe with the reactor tube to cool the results of the pyrolysis process in the form of oil which then results will come out through the hole that has been prepared for the reservoir .

3.3 Test the chemical properties and compounds resulting from pyrolysis

This test uses the *Gas Chromatography Mass Specroscopy* (GCMS) instrument. A sample in the form of pyrolysis oil is taken as much as 0.2 µl and immediately injected into the sample into the GC column via a heated injection port. The volatile nature of the solution will be read by the Mass Spectrometry (MS) detector to determine the type of hydrocarbon compound present in the the solution .

3.4 Test the Physical Properties of Pyrolysis Oil

3.4.1 Density Test

Using ASTM D 5002. The unit for density is gr/ml or kg/m ^{3} . A number of samples are inserted into the oscillating sample tube and changes in the oscillation frequency caused by changes in the mass of the tube are correlated with calibration data to determine the density of the sample .

JBP Vol. 25, No. 2, Desember 2023- Denni Afriadi , Fazrul M Yasin
DOI 10.20473/jbp.v25i2.2023.108-116

3.4.2 Flash Point Test

ASTM D 92, approximately 70 mL of the test sample was filled into the test bowl. The temperature of the test sample was then raised rapidly first then slowed down at a constant speed as it approached the flash point. At specified intervals a test flame is passed over the surface of the test bowl. The flash point is the lowest sample temperature at which application of the test flame causes the sample vapor to ignite. To determine the point of fire continue the test by increasing the temperature until combustion occurs for at least 5 seconds .

3.4.3 Pour Point Test

Using ASTM D 97 , by preheating, the sample is cooled slowly at a certain speed and its flow properties are checked every 3 ^{o} C. The lowest temperature at which the sample cannot flow is recorded as the pour point .

3.4.4 Kinematic Viscosity

Using ASTM D 445 by pouring the sample lubricating oil into the Glass Vial and placing it on the Steel Adapter Vial provided on the Sample Tray according to the estimated viscosity. Tray 1 for viscosities 3 – 300 (diluted sample) and Tray 2 for viscosity 10 – 1000 (thick sample), then position the Pilot Tray until the red arrows meet and allow the Bath temperature to stabilize before testing the sample.

4. RESULTS AND DISCUSSION

4.1 Test Results

4.1.1 Testing the Chemical Properties of Pyrolysis Oil

The components of the chemical compounds in the pyrolyzed oil tested using the GCMS instrument detected hydrocarbon compounds which are components of oil compounds that are identical to those of petroleum. These results can be seen in Figure 6 and Table 1, which displays the results of the analysis using the instrument.

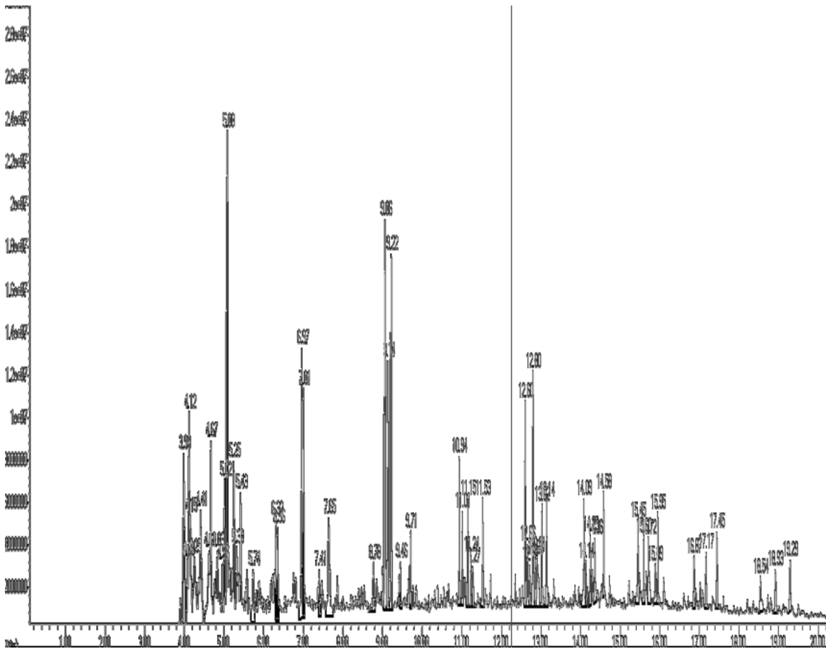

Figure 6. Chromatogram analysis of oil compounds resulting from pyrolysis using GCMS.

Table 1. Data on liquid fuel compounds resulting from pyrolysis of plastic waste

No	Peak	% Area	Compound Name	Formula
1	5	12,713	Pentane	C_5H_{11}
2	15	7,491	1-pentane,2-methyl	C_6H_{14}
3	17	6,573	2-pentene,2-methyl-	$C_6H_{12}O$
4	1	4,496	2-hexene,3-methyl-	C_7H_{14}
5	12	4,472	3-heptene,2-methyl	$C_6H_{12}O$
6	16	4,205	Heptane, 4-methyl	$C_6H_{13}O$
7	2	4,162	Cyclohexane,propyl-	C_6H_{12}
8	6	4.155	2,4-dimethyl-1-heptane	$C_9H_{20}0$
9	24	3,850	3-heptane,4-methyl-	C_8H_{16}
10	4	3,677	Nonane,2,6-dimethyl-	$C_9H_{20}O$
11	13	3,524	2,4-dimethy-1-heptane	C9H20 _ _ _

4.1.2 The results of the pyrolysis process using an incinerator machine

The results of the testing process which was carried out using the UP incinerator machine in the pyrolysis process took approximately 2 hours more and obtained the results of liquid fuel with varying weight of zeolid. As much as 1 kg of plastic without a zeolite catalyst, 811 ml of oil was obtained. The raw material is 1 kg of plastic with 0.25 kg of zeolite catalyst with a ratio of 1:0.25 to get 815 ml of oil. The raw material is 1 kg of plastic with 0.5 kg of zeolite catalyst with a ratio of 1:0.5 to get 825 ml of oil. The raw material is 1 kg of plastic with 0.75 kg of zeolite catalyst with a ratio of 1:1 to get 855 ml of oil. The raw material is 1 kg of plastic with zeolite catalyst. 1 kg with a ratio of 1:1 obtained as much as 955 ml of oil. These results can be seen in Figure 7 and Figure 8 which displays the results of pyrolysis using a variety of catalysts.

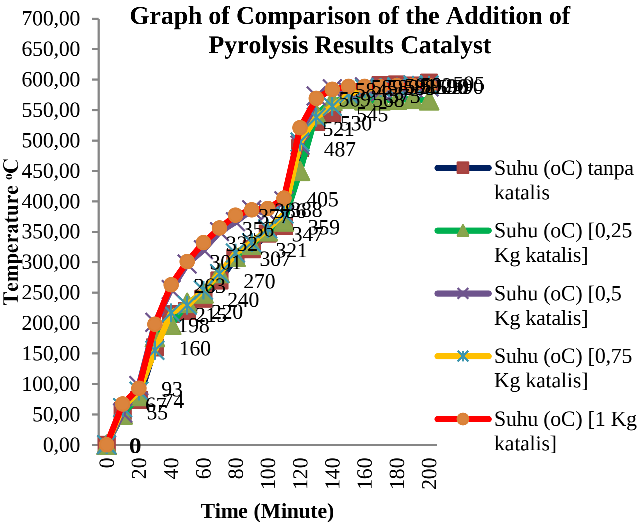

Figure 7. Graph of pyrolysis results with a comparison of variations of zeolid catalysts

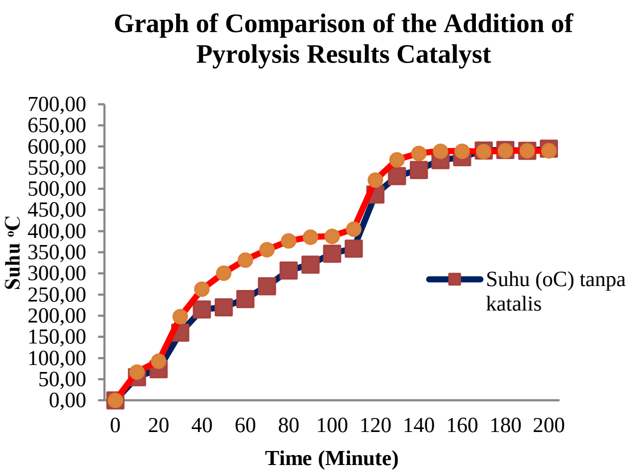

Figure 8. Graph of pyrolysis results with a comparison of variations of zeolid catalysts

The results of the priolysis of the sample which is already in the form of a yellow liquid, can be seen in Figure 9.

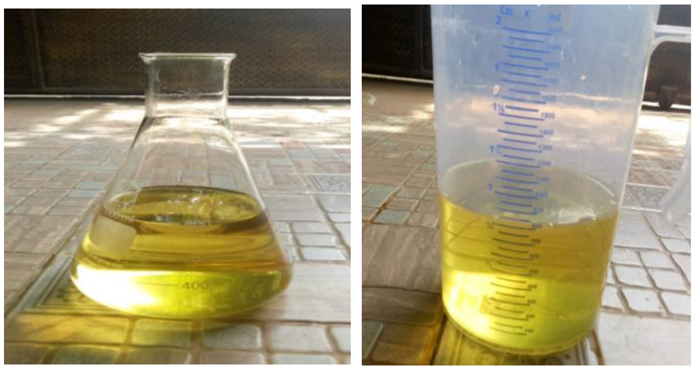

Figure 9. Oil products from pyrolysis of plastic waste samples

The results of the pyrolysis oil measurement tests at the Lemigas Laboratory can be seen in Table 2 and Table 3 which display the data.

4.1.3 Condenser analysis on the type of "*Cross Flow Condencor*"

The condenser used is adapted to the pyrolysis reactor laboratory at the Faculty of Engineering , Pancasila University . In the condenser there are two levels, in each level of the tube there are 52 steam pipes, so the total is 104 steam pipes .

Table 2. Parameter test results for pyrolysis oil at Lemigas

No	Test Method	Characteristics	units	Test Results
1.	ASTM D 240	Calorific Value	Mj/Kg	40.947 Mj/Kg
2	ASTM D 445-12	Kinematic Viscosity at 40 °C	cST	1,556
3	ASTM D 93	Fire Point	°C	42
4	ASTM D 93-13	PMCC flash point	°C	26
5	ASTM D 97-12	Pour Point	°C	-42
6	ASTM D 4052-11	Density at 15 °C	g/cm^3	0.7857
7	ASTM D 6304-07 $^{\epsilon 1}$	Water Content	Ppm	507.54

The results of free convection in the condenser after the calculations are carried out, the results are obtained successively, namely, the value for the heat transfer in the fluid in the condenser is 969,569,70 W; Heat rate in water fluid 963, 270 W; The calculation results for the Reynolds number

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(laminar flow) obtained a Re $_{tube\ value}$ of 7.176.

The *pressure drop* calculation is calculated based on several parameters including *the friction factor* which is calculated using the calculated *Reynolds number, the friction tactor* value is 0.0677, the tube cross-sectional area is 0.000314 m^2, the length of the Lc tube is 0.3 m, ρ_{gas} is 959.50 kg/m^3, while the *pressure drop* (ΔP) is 0.18357 N/m^2.

carbon chain atoms. straight, starting from the carbon atoms C_5 to C_9 (Pentane - Nonana) (Dhamayanthie et al., 2018). Based on the test results data that has been included, it can be said that time and temperature greatly affect the results of the pyrolysis itself. However, with the addition of a catalyst in the form of zeolid The graph produced in Figure 7 shows the difference in temperature in the addition of the catalyst and also affects the volume of pyrolysis results. With no catalyst, the volume produced was 811 ml, with the addition of 0.25 kg of catalyst a volume of 815 ml was produced, with the addition of 0.5 kg of catalyst a volume of 825 ml was produced, with the addition of 0.75 kg of catalyst a volume of 855 ml was produced, with the addition of 1 kg of catalyst produced a volume of 897 ml. The maximum temperature of the pyrolysis results is at 590 ^{o}C at 180 to 200 minutes. Figure 8 is a graph that presents data on the temperature difference of the pyrolysis results without a catalyst at 20 to 160 minutes, there is a significant increase and affects the volume of pyrolysis results. Where without a catalyst it produces a volume of 811 ml and with a catalyst of 1 kg it produces a volume of 955 ml, this shows that the effect of adding catalyst has an effect on temperature and the resulting volume. This proves that during the

Table 3. Characteristic specifications of fuel oil, Director General of Oil and Gas, Ministry of Energy and Mineral Resources

No	Characteristics	Unit	Limitation				Test Method
			IFO-1		IFO-2		
			Min.	max	Min.	max.	ASTM
1	Calorific Value	MJ/kg	41.87		41.87		D 240
2	Density at 15 ^{o}C	kg/ ^{m3}	-	911	-	991	D 1298
3	Kinematic viscosity at 50 ^{o}C	mm2 / sec	-	180	-	380	D 445
4	Sulfur content	% m/m	-	3.6	-	4.0	D1552/2622
5	pour point	^{o}C	-	30	-	40	D 97
6	Flash point	^{o}C	60	-	60	-	D 93
7	Carbon residue	% m/m	-	16		20	D 189
8	Ash content	% m/m	-	0.10	-	0.15	D 482
9	Total sediment	% m/m	-	0.10	-	0.10	D 473
10	water content	%v/v	-	0.75	-	1.0	D 95
11	Vanadium	mg/kg	-	200	-	-	AAS
12	Aluminum + silicon	mg/kg	-	80	-	-	D 5184/AAS

4.2 DISCUSSION

The GC-MS (*Gas Chromatography-Mass Spectrometry*) test was carried out to determine the percentage of plastic oil content composition which was carried out at the Puslabfor Bareskrim Polri. From table 1 it can be seen that the percent (%) of the peak area indicates the occurrence of a cracking process. Cracking that occurs in plastic is the breaking of the carbon chain bonds that make up the plastic. Plastic is composed of *hydrocarbon molecules* derived from petroleum fractions. From the results of the analysis using GC-MS, it is said that after the plastic waste sample has gone through the pyrolysis process, the results obtained are in the form of a component of a hydrocarbon compound which has components such as the petroleum fraction of gasoline, in which it is said that gasoline fuel is composed of simple hydrocarbons having the form of priolysis process, there are several aspects including time; temperature; Particle size and mass greatly affect the pyrolysis process itself (Ramadhan & Ali, 2012) . In this case the intended particles are the sample and the catalyst .

Tables 2 and 3 which present comparative data between the pyrolysis test results at LEMIGAS and the BBM test results from the Directorate General of Oil & Gas show that the difference is not too great in the resulting calorific value, this shows that the results of pyrolysis oil at Lemigas produce a calorific value of 40.947 Mj/Kg while the standard for the Directorate General of Oil & Gas is 41.87 Mj/Kg. Meanwhile, the Fire Point Test (flash point) at Lemigas is 42 ^oC, while the standard for the Director General for Oil & Gas is 60 ^oC. The Pour Point Test (point cast) in Lemigas is -42 ^oC, while the standard for the Director General of Oil & Gas is 30 ^oC, in the Density test at 15 ^oC (density at 15 ^oC) is 0.7857 g/cm ^{3} = 785.7 kg/m^3 , while the standard for the Director General of Oil & Gas is 991 kg/m ^{3} , in the Water Content test it is 507.54 ppm, the standard for the Director General for Oil & Gas is 991 kg/m^3, 0.75 % v/v = 750 ppm.

Determination of the *Reynolds number* is carried out with the aim of knowing the magnitude of the value of the fluid flow itself. This number is classified into 3 numbers, namely laminar flow where the *Re value* is <2000, turbulent flow is *Re* > 2300, and transitional flow is *Re* = 2000 – 2300. The Re value obtained based on experimental results is that it has a Re value = 7,176 laminar flow . It is said that there is a relationship between the *Reynolds number* and the velocity of the fluid flow, if the higher the value of Re, the greater the coefficient of friction produced (Subagyo et al., 2016) .

5. CONCLUSION

From the results of this study the effect of the amount of catalyst on the yield/yield of plastic oil from the plastic pyrolysis process and testing the characteristics of pyrolysis oil, and the chemical test results showed that there were 12.713% pentane compounds, this indicated the presence of flammable hydrocarbon compounds

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