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The Effect of Temperature Variation on the Liquefaction of High-Density Polyethylene Plastic Waste

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Abstrak

Penelitian ini bertujuan untuk mengetahui perbandingan *yield* minyak yang diperoleh dari pengolahan limbah plastik HDPE dengan membandingkan secara kuantitatif dan kualitatif bahan bakar minyak yang dihasilkan. Pengolahan sampah plastik menjadi minyak dengan proses pirolisis dilakukan pada variasi suhu (400; 500; 600; 700; 800)°C selama 120 menit. Dalam satu kali percobaan, diperlukan 500 gram sampah plastik, 250 gram katalis dolomit, dan 250 ml pelarut H_2SO_4. Penelitian ini dilakukan di Laboratorium Jurusan Teknik SKL Universitas Jambi dengan parameter yang diuji meliputi analisis volume, massa, %-*yield*, densitas, dan GC-MS. Hasil penelitian menunjukkan bahwa secara kuantitatif volume, massa, dan %-*yield* terendah ada pada variasi suhu 400°C, dan tertinggi pada variasi suhu 500°C. Pada variasi suhu pirolisis 500°C, densitas yang dihasilkan adalah 0,744 gram/ml, dan hasil analisis GC-MS menghasilkan rantai karbon C_5-C_{12} 97,6081% (bensin), C_{13}> 2,3919% (minyak tanah), dan senyawa aromatik yang terkandung di dalamnya berjumlah menjadi 14,3786%.

Kata Kunci: pirolisis, bensin, energi, densitas, kromatografi gas, spektometri massa

Abstract

This study aims to determine the comparison of oil yields obtained from processing HDPE plastic waste by comparing quantitatively and qualitatively the fuel oil produced. The processing of plastic waste into oil by pyrolysis process was carried out at a temperature variation of (400; 500; 600; 700; 800)°C for 120 minutes. In one experiment, 500 grams of plastic waste, 250 grams of dolomite catalyst, and 250 ml of H_2SO_4 solvent were required. This research was conducted at the Laboratory of SKL Engineering Department, Jambi University with the parameters tested including volume, mass, %-yield, density, and GC-MS analysis. The results showed that quantitatively the lowest volume, mass, and %-yield were at the 400°C temperature variation, and the highest at 500°C. At 500°C pyrolysis temperature variation, the resulting density was 0.744 grams/ml, and the results of GC-MS analysis produced carbon chains C_5-C_{12} 97.6081% (gasoline), C_{13}> 2.3919% (kerosene), and aromatic compounds contained in it amounted to 14.3786%.

***Key words** : pyrolysis, gasoline, energy, density, gas chromatography-mass spectrometry*

INTRODUCTION

Indonesia is one of the most populous countries in the world, which is accompanied by the rapid need for the use of plastics. The use of plastic is driven by its advantages of being lightweight, strong, rustproof, flexible, easy to shape, unbreakable, and easy to color (Wisnujati & Yudhanto, 2020). Excessive use and not all plastic products are suitable for repeated use, impacting the amount of waste generated. Based on data from the National Waste Management Information System (SIPSN) by the Ministry of Environment and Forestry (KLHK) in 2022, the total volume of national waste is 19.58 million tons, and the amount of plastic waste generated is 18.2% or 3.56 million tons.

According to (Febrianta & Yuwono, 2022), plastic waste causes environmental damage because it cannot be decomposed by microorganisms so it is difficult to destroy by itself or is non-biodegredable. Its decomposition is estimated to take 100 to 500 years because its components are composed of petroleum hydrocarbons as the main ingredient, as well as the synthesis of other materials such as natural gas and coal (Alfernando et al., 2020). High-Density Polyethylene (HDPE) plastic which is classified as a long-chain polymer of ethylene monomer is one of the plastic compositions that contribute 46% of its waste to the environment (Praputri et al., 2016).

HDPE plastic is widely used such as in milk bottles, medicine bottles, gallon lids, lubricant jerry cans, plastic bags, and plastic cups. However, (Masyruroh & Rahmawati, 2021), recommend the use of HDPE plastic in single use because repeated use will increase the release of Antimony Trioxide (SbO_3) compounds which are carcinogenic to human health. As an illustration, to produce 1 kg of HDPE plastic requires 1.75 kg of petroleum hydrocarbons and one of the constituent components is naphtha. From this statement, HDPE plastic waste can be returned to its original form through the pyrolysis process (Nugraheni & Maulana, 2019).

The pyrolysis process is carried out by heating HDPE plastic so that the polymer is degraded with low volatility and produces liquid hydrocarbons in the form of fuels that have the potential to be used as an alternative energy source. The pyrolysis process will use dolomite catalyst which refers to the results of HDPE plastic pyrolysis research by (Sonawane et al., 2017), where dolomite catalyst plays a role in breaking the length of HDPE plastic polymer chains into short chains and producing 82% yield of liquid fuel in the gasoline fraction with carbon chains (C_{10}-C_{25}). Before use, dolomite catalyst will be activated in the furnace so that the pores are uniform so as to facilitate separation and accelerate the reaction by reducing the activation energy (Ardisa et al., 2017)

Not only catalysts, this research also used a solvent of Sulfuric Acid (H_2SO_4) which refers to the results of sulfuric acid in cellulose pyrolysis by (Hu et al., 2018), where sulfuric acid composed of two Hydrogen ions ($2H^+$) as proton donors. The use of sulfuric acid resulted in depolymerization by breaking C_1-C_4 bonds up to 72%. On that research, the sulfuric acid used was able to produce the highest yield of 20.5%. On the research I will do, this solvent is thought to have a positive value on protonation activity to depolymerize and stretch the polymer chains that compose it. Then the presence of two Hydrogen ions ($2H^+$) as proton donors has the potential to assist hydrogenation activity so as to reduce the content of olefin, aromatic and nonhydrocarbon compounds contained in the fuel because the impurities produced during the pyrolysis process will be bound by sulfuric acid. Furthermore, the vessel used to pyrolyze HDPE plastic is a batch reactor. Before the pyrolysis process is carried out, the raw material is put into the reactor bucket and after that it will be closed until the reaction time ends so that there is no addition to the incoming or outgoing mass (Cuoci, 2019).

Based on the description above, it is important to conduct research on the liquefaction of HDPE type plastic waste into liquid fuel. In the research to be carried out, the variation of temperature becomes the main role to be studied. It will be seen how the effect of several temperature variations on the HDPE plastic waste liquefaction process. Based on the background that has been described, the problem formulation in this study is how the effect of temperature variations on the quantity and quality of liquid from pyrolysis of HDPE type plastic waste into liquid fuel.

The scope of this research is limited to temperature variations during the pyrolysis process, namely (400;500;600;700;800) °C, the ratio of HDPE plastic and sulfuric acid solvent is 1:1, dolomite catalyst as much as 250 grams, and pyrolysis time for 2 hours. Furthermore, the pyrolysis liquid will be analyzed using a Gas Chromatography Mass Spectrometry (GC-MS) tool to determine the content and characteristics of the compounds in liquid fuel after passing through a distillation process that uses a simple distillation circuit at 150°C for 1 hour. The results of this study are expected to provide several benefits including getting an alternative to the use of HDPE plastic waste to be pyrolyzed into liquid fuel, providing solutions to plastic waste problems, and can be used as a reference for further research.

RESEARCH METHOD

Locations

This research was held at the Laboratory of the Department of Civil, Chemical, and Environmental Engineering, Faculty of Science and Technology, Jambi University.

Equipment and Materials

The equipment used in this study include: 200 mesh sieve, sample bottle, glass funnel, erlenmeyer, furnace, gas, measuring cup, scissors, heating jacket, condenser, three neck flask, analytical balance, oven, pump, pycnometer, batch reactor, stative and clamp, Gas Chromatography Mass Spectrometry. The materials used in this study include: 2×2 cm HDPE plastic waste as much as 500 grams, 250 grams of activated dolomite catalyst, and 500 ml sulfuric acid solvent.

Procedurs

HDPE plastic waste is certainly not clean and has a lot of dirt attached, so before pyrolyzing the raw material will be washed thoroughly and then dried under the heat of the sun. Then the raw material will be reduced in size by cutting into 2×2 cm pieces. This research used dolomite catalyst, it would be screened using a 200 mesh sieve. The sieved catalyst will be weighed as much as 250 grams, then placed in a closed iron tube and heated in a furnace. Heating is done at 400ºC for 2 hours. After heating, the activated dolomite catalyst will be obtained. Therefore, this activation process is classified as physical, because it does not involve the use of chemical solutions. The clean HDPE plastic measuring 2×2 cm was dissolved together with sulfuric acid in a ratio of 1:1, in a bucket. Pouring sulfuric acid into the bucket is done slowly and carefully. The next process is mixing by stirring for 15 minutes. A total of 250 grams of activated dolomite catalyst was put into a bucket that had been mixed with solvents and raw materials. Then the bucket is put into the reactor to be heated so that the raw material can be pyrolyzed. The pyrolysis process will last for 2 hours, with the temperature variations used are (400; 500; 600; 700; 800) ºC, so there are 5 trials. The results of pyrolysis in the gas phase will pass through the condenser coil to change its phase to the liquid phase, this is what is called the pyrolysis liquid.

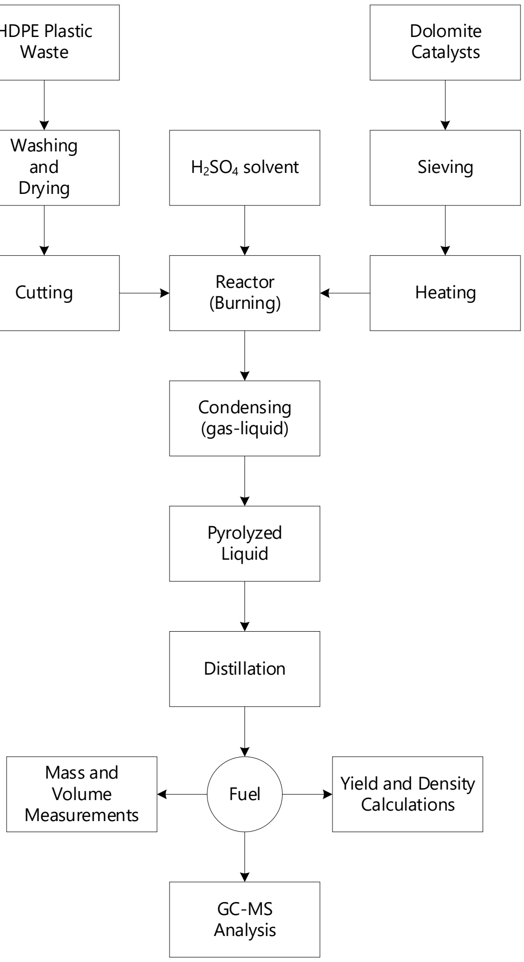

Figure 1. Block Diagram of Research Method and Result Analysis

The pyrolysis liquid from each variation of temperature produced will be distilled to separate and purify the oil produced from a mixture of solvents and impurities. This process is carried out for 1 hour with a temperature of 150ºC. The refined oil is referred to as distillate. The analysis in this research is volume and mass measurements, %-yield and density calculations, and GC-MS analysis in the laboratory of FMIPA UGM.

RESULT AND DISCUSSION

Particle size affects the yield and composition of pyrolysis products because smaller particle sizes produce more pyrolysis products. However, a large particle size makes the surface area per unit weight small so that the pyrolysis process will experience a slowdown and produce less CHP. So before the pyrolysis process begins, the author makes size adjustments to HDPE plastic which is cut into 2×2 cm and according to the size of the previous research conducted (Lubi et al., 2017). The importance of determining the time in the pyrolysis process, so the author uses a pyrolysis time of 2 hours. The determination is based on research conducted by (Chiwara et al., 2018), who used HDPE plastic to be pyrolyzed with a time variation of (20;40;60;80;100;120;140;160;180) minutes. The results obtained at the 120th minute pyrolysis time were 70% of the raw material converted into CHP products with an oil yield of 95%. From the research that I have conducted, the following results were obtained:

Table 1. Results of High-Density Polyethylene Plastic Pyrolysis

No	Pyrolysis Time (hr)	Ratio of HDPE and Solvent	Raw HDPE (gr)	Pyrolysis Temperature (°C)	Pyrolysis Liquid Volume (ml)	Mass of Pyrolysis Liquid (gr)	Yield (%)
1				400	170,0000	140,0800	9,8996
2				500	199,0000	163,9760	11,5884
3	2	1:1	500	600	228,0000	186,0480	13,1483
4				700	270,0000	222,4800	15,7230
5				800	345,0000	300,8400	21,2608

Based on Table 1, it can be seen that temperature variations greatly affect the volume and mass of the pyrolyzed liquid. Furthermore, based on these temperature variations, the author can determine the minimum temperature and optimum temperature in the HDPE plastic pyrolysis process. Based on table 1, the lowest volume and mass of pyrolyzed liquid are at 400°C with a volume of 170.0000 ml and a mass of 140.0800 grams. While the volume and mass of the highest pyrolyzed liquid were at 800°C with a volume of 345 ml and a mass of 300.8400 grams.

According to (Ramadhan & Ali, 2019), temperature is an operating condition that will affect the amount of product. In accordance with the Arhenius equation, an increase in temperature makes the value of the thermal decomposition constant increase and then makes the pyrolysis rate and yield increase. At higher temperatures, gas yields will increase, and oil yields will increase. It can be seen that temperature variations will affect the %-yield obtained. This statement is supported by (Firman et al., 2019), where the higher the pyrolysis temperature, the higher the %-yield produced. From the research that has been done, the lowest %-yield is 9.8996% with a pyrolysis temperature of 400°C. Furthermore, the highest %-yield was 21.2608% with a pyrolysis temperature of 800°C. Then, for the liquid density analysis results obtained as follows:

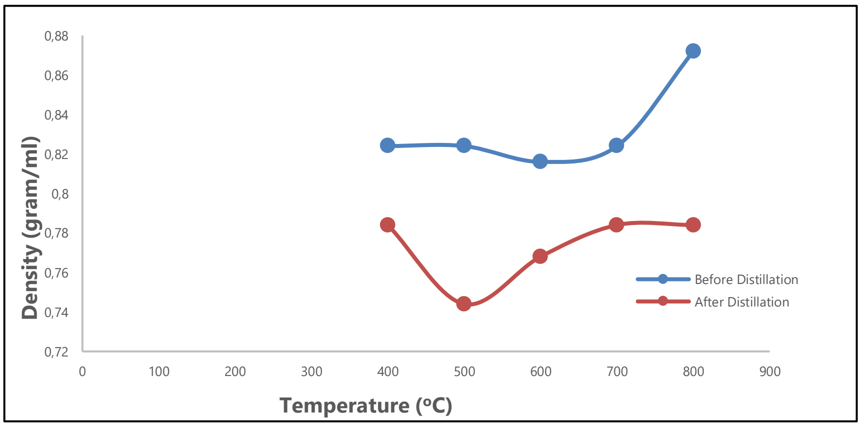

Figure 2. Before and After Distillation Density of Product Graph

Table 2. t-Test Paired Two Sample for Means

Parameters	Before	After
Mean	0,8320	0,7728
Variance	0,0005	0,0003
Observations	5,0000	5,0000
Pearson Correlation	0,3631	
Hypothesized Mean Difference	0,0000	
df	4,0000	
t Stat	5,7435	
P(T<=t) one-tail	0,0023	
t Critical one-tail	2,1318	
P(T<=t) two-tail	0,0046	
t Critical two-tail	2,7764	

By using the paired t-test testing method, the author was able to find out the difference between before and after the density of product. The alpha (α) value used in this test is 0,05. Note that if the alpha (α) value is less than 0,05 then there is a significant difference between the density before and after. Conversely, if the alpha (α) value exceeds 0,05 then there is no significant difference between the density before and after. From the results of the statistical tests carried out, it can be seen in the table that the alpha value is below 0,05, namely 0,0046. Thus, the density data before and after show different values.

Then the density of the sample before distillation is in the high value range. The high-density value exceeds the standard value of SNI 3506-2017 regarding the density of RON 88 gasoline fuel with a value range of 0.7150 grams/ml-0.7700 grams/ml. The high-density value of the sample before distillation is in accordance with the standard Kep. DJM No.14499K/14/DJM/2008 regarding the density of CN 48 diesel fuel with a range of 0.8150 grams/ml- 0.8700 grams/ml (Lubis et al., 2022). The following is a figure of the pyrolysis liquid before distillation:

Figure 3. Pyrolysis Liquid Before Distilling

Based on the literature, the difference in density values before and after distillation occurs because the pyrolysis liquid before distillation has impurities and moisture content in it. Of the five samples obtained, it can be seen that the samples at 500°C and 600°C that have been distilled are in accordance with the SNI 3506-2017 standard, namely the density of RON 88 gasoline fuel, with a density of 0.7440 grams/ml and 0.7680 grams/ml, respectively. For GC-MS analysis, the samples used were samples with temperature variations of 500°C. The selection is based on the color produced is very clear with a yellowish color compared to other samples. This sample is in accordance with SNI 3506-2017 standards, namely the density of RON 88 gasoline fuel, where the density of this sample is 0.7440 grams/ml (Figure 4).

Based on the GC-MS results that have been carried out and shown in figure 4, the HDPE plastic pyrolysis oil has the highest chromatogram peak at the 15th peak with a retention time of 3.6600 minutes, with a %-area of 6.37%. At that peak, the carbon chain produced is C_7 with the dominant compound that appears is Cycloheptane or C_7H_{14}. According to (Dhaniswara & Fahriani, 2021), the retention time and area detected in the GC-MS analysis results will definitely vary, this is due to the influence of the physical and chemical properties of the chemical compounds present, as well as the molecular weight contained in the product.

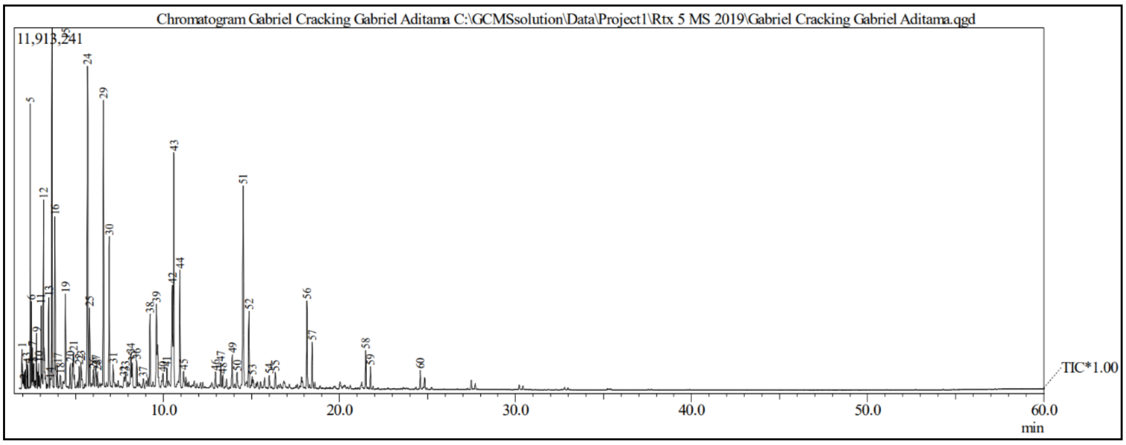

Figure 4. Chromatogram of Fuel from Pyrolysis of HDPE Temperature 500°C

There were 60 chromatogram peaks with the fastest retention time of 1.9580 minutes and the longest of 24.5830 minutes. The results also showed diverse SI values, %-area, and carbon fractions. This indicates that the resulting fuel sample does not come from a single compound, but consists of various compounds characterized by the smallest constituent chain is C_7 and the largest is C_{14}. The analysis resulted in a C_5-C_{12} carbon chain of 97.6081%, and C_{13}> 2.3919%. Furthermore, the compounds contained in the resulting carbon range are:

Table 3. Dominant Compound of GC-MS Analysis Result

Carbon Chain	**Area (%)**	**Dominant Compound**
C_5-C_{12}	97,6081	C_5H_8 0,3466%-area, C_5H_{10} 0,9219%-area, $C_5H_{10}O_2S$ 0,3435%-area, C_6H_6 4,3382%-area, C_6H_8 0,6158%-area, C_6H_{10} 4,8681%-area, C_6H_{12} 5,1102%-area, C_6H_{14} 1,1957%-area, C_7H_8 9,3122%-area, C_7H_{12} 5,2553%-area, C_7H_{14} 9,6922%-area, C_7H_{10} 0,7176%-area, C_7H_{16} 3,7596%-area, C_8H_{10} 9,4134%-area, C_8H_{14} 1,2147%-area, C_8H_{16} 8,7415%-area, C_8H_{18} 3,9537%-area, C_9H_8 0,4950%-area, C_9H_{10} 0,4026%-area, C_9H_{12} 2,4688%-area, C_9H_{16} 0,3434%-area, C_9H_{17} 0,4482%-area, C_9H_{18} 7,7420%-area, $C_9H_{18}O$ 0,4934%-area, C_9H_{20} 2,8250%-area, $C_{10}H_{18}$ 0,4382%-area, $C_{10}H_{20}$ 1,0737%-area, $C_{10}H_{22}$ 1,9500%-area, $C_{11}H_{22}$ 8,1401%-area, dan $C_{12}H_{24}$ 0,9874%-area
C_{13}>	2,3919	$C_{13}H_{28}$ 1,8947%-area, dan $C_{14}H_{28}$ 0,4972%-area

Based on table 3, the most dominant compound appearing is C_7H_{14} at 9.6922%-area. This fact is supported from the chromatogram results as shown in figure 4, where the highest chromatogram is on the 15^{th} peak with a retention time of 3.6600 minutes. The C_5-C_{12} hydrocarbon fraction has the largest percentage with a value of 97.6081%, and the long-chain hydrocarbon fraction C_{13}> 2.3919%. According to Fanani et al (2021), the C_5-C_{12} hydrocarbon fraction is a gasoline-type liquid fuel, and C_{13}> is a type of kerosine. When referring to the theoretical basis, the research that the author has done produces gasoline fuel with a percentage of 97.6081% and by-products in the form of 2.3919% kerosene. Then the aromatic compounds contained in the products of this research can be seen in the following table:

Table 4. Content of Aromatic Compounds Result of GC-MS Analysis

Aromatic Compounds (%)	**Dominant Compound**
14,3786	hexahydrobenzene, benzene, ethylbenzene, p-dimethylbenzene, 1,4-dimethylbenzene, 1,4-dimethyl-benzene, m-dimethylbenzene, 1,3-dimethylbenzene, 1,3-dimethyl-benzene, 1,2-dimethylbenzene, 1,4-dimethylbenzene, 1,4-dimethyl-benzene, n-propylbenzene, propylbenzene, 1-propylbenzene, benzeneacetaldehyde, benzeneethanamine, isopropylbenzene, o-ethylmethylbenzene, o-methylethylbenzene, 1-ethyl-2-methylbenzene, 1-methyl-2-ethylbenzene, p-ethylmethylbenzene, p-methylethylbenzene, 1-ethyl-4-methylbenzene, 1-methyl-4-ethylbenzene, 1-propenylbenzene, 1-propynylbenzene

To more clearly know the content of liquid fuel expected in this study, it can be seen from the content of aromatic compounds contained in the resulting product. Based on table 4, it can be seen that the content of aromatic compounds in the resulting product is 14.3786%. When viewed from the content of aromatic compounds in the product from the results of this study, it further proves the presence of liquid hydrocarbons that lead to gasoline-type fuels. This is proven because the content of aromatic compounds in the product of this study is in accordance with the terms and conditions of the Decree of the Director General of Oil and Gas No. 3674K/24/DJM/2006 dated March 17, 2006, namely the specification of RN 88 gasoline, with a maximum of 40% aromatic compounds (Nofendri, 2018). Sinaga et al (2023) explained that too high a percentage of aromatics can pollute the environment and be harmful to health. Aromatic compounds are high octane components in gasoline that can produce benzene vapor which is very dangerous to health (carcinogen) and can increase CO exhaust emissions in the air.

Figure 5. Pyrolysis Fuel from All Temperature Variations

It is proven that the dolomite catalyst used in the study is able to break the long polymer chains of HDPE plastic. This is because dolomite catalyst is a natural mineral form of calcium and magnesium double carbonate. Its chemical formula is ($CaMg(CO_3)_2$), which is predominantly composed of 41% $MgCO_2$ and 58% $CaCO_2$. The suitability of using dolomite catalyst is based on the results of HDPE plastic pyrolysis research into fuel conducted by (Hanif et al., 2016), where it produced a gasoline fraction of 60.9% with carbon chains (C_5-C_9) and by (Sonawane et al., 2017) producing carbon chains (C_{10}-C_{25}) which leads to the gasoline fraction. When the pyrolysis process takes place, dolomite will break the long polymer carbon chains in HDPE plastic to produce carbon compounds with short chains. Then dolomite suppresses the high use of temperature and is able to cut the pyrolysis reaction time for 30 minutes. In addition to accelerating the reaction rate by lowering its activation energy, dolomite catalyst also aims to direct the reactants to form pyrolysis products.

In accordance with the statement (Hu et al., 2018), in the early stages of plastic pyrolysis, a protonation process occurs. The protonation process can occur due to the acidity contained in Sulfuric Acid (H_2SO_4). This compound is classified as a strong lewis acid because it has two Hydrogen ions ($2H^+$) which act as proton donors on secondary carbon bonds so called secondary carbocations. Secondary carboxyation will act as a resonance contributor to depolymerize and stretch the polymer chain in HDPE plastic.

Secondary carboxyation is an unstable species because there is no hyperconjugation effect produced by its alkyl group. Furthermore, with two Hydrogen ions ($2H^+$) as donors, it will be positive in relation to the hydrogenation process for HDPE plastic melting because it can reduce the presence of olefin compounds and non-hetero atoms such as Nitrogen (N) and Sulfur (S) in the form of volatile compounds. Therefore, the study used 98% H_2SO_4 which helps to reduce the content of olefin compounds, aromatics and nonhydrocarbon compounds contained in the liquid fuel. This research was conducted using a cylindrical batch reactor. Batch reactors are often referred to as closed reactors because there is no flow of mass in or out during the reaction (Cuoci, 2019). The cleaned HDPE plastic will be dried and then reduced in size because the surface area of the raw material per unit weight affects the speed or slowness of the pyrolysis process, then after that the raw material is put into the reactor. The batch reactor is equipped with a bucket to place and react between raw materials, solvents, and catalysts.

CONCLUSION

The variation of pyrolysis temperature greatly affects the amount of pyrolysis liquid output produced. The higher the pyrolysis temperature, the greater the volume of pyrolysis liquid obtained as well as the %-yield. The results of the CHP product sample test at a temperature variation of 500°C using a GC-MS instrument lead to gasoline-type fuel with a C_5-C_{12} carbon chain of 97.6081% and aromatic compounds contained in it as much as 14.3786%. The fuel produced is in accordance with the SNI 3506-2017 standard with the type of gasoline RN 88 which has a density of 0.7440 grams/ml.

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