Catalyst Screening for Conversion of Waste Polyethylentherphtalate Plastic in to Liquid Fuel

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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ABSTRACT

The world-wide use of PET has gradually increased from time to time. And most of the PET waste is being dumped into the environment instead of recycling. This creates serious environmental pollutions. In thermal degradation process, PET is being decomposed to liquid fuels. Conversion of recycled PET bottle into fuel through effective catalyst has been investigated as a strategy for wastes into useful energy. The main objective of this research was to screen out an effective catalyst for the conversion of PETE into liquid hydrocarbon fuel. In this study, Zinc oxide, ZnO/AC, Calcium Hydroxide (Ca (OH)$_2$) and Ferric oxide (Fe$_2$o$_3$) catalysts was used at different operation condition of temperature ranging from 400 to 500°C. The optimum liquid fuel productions from PET plastic bottle were obtained. And the significance result of the maximum temperature through the use of catalyst was enhancing to identify the most effective Catalyst with respect to oil yield. The oil yield increase when the temperature increases within the range of temperature (400°C-500°C). It was observed that gas/liquid fuel yield significantly affected by type of catalyst and temperature. Ca (OH)$_2$ catalyst result higher yield compared the two catalysts. Finally, during the characterization process it was categorized kerosene and crude oil; these oil were obtained by PET and PET with the combination of LDPE & HDPE respectively, and the analysis were under taken from the two types of oil samples and identified Benzene by the use of GC (Gas Chromatography).

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1. INTRODUCTION

Consumption of plastic similar to production shows an exponential increase with more plastics being produced in the first decade of the present century than in the entire preceding century [1].

Ethiopia has one of the fastest economic developing countries in Africa, following this economic development, industries producing more plastic waste due to use of more plastic packaging, shopping plastic bags, PET bottles and other goods which is use plastic as the major component. Several of the chemicals used to produce plastics are hazardous for human health and the environment [2]. Besides the sheer number of plastic bags and bottles produced each year, the energy required to manufacture and transport these bottles to market severely drains limited fossil fuels. For example, bottled water companies, due to their unregulated use of valuable resources and their production of billions of plastic bottles have presented a significant strain on the environment.

Some studies showed that plastic are causing several environmental pollutions to human and animal health in urban and rural areas of the country [3](Ramaswamy and Sharma). For example, it is common to find countless plastic materials washed ashore, which poses a serious problem to the natural environments. Plastic takes much longer to decompose than many other types of garbage that is typically thrown away. Waste plastics are one of the most promising materials for the production of fuel because it's high heat combustion and also the high amount of availability in local communities.

1.1 Definition

The term plastic refers to a family of materials which includes nylon, polyethylene and PTFE just as zinc, aluminum and steel fall within the family of metals.

1.1.1 Sources and properties of plastic wastes

Plastic wastes can be classified as industrial and municipal plastic wastes according to their origins; these groups have different qualities and properties and are subjected to different management strategies.

1) Industrial plastic wastes
2) Municipal plastic wastes[4]

1.1.2 Types of waste plastics and their recyclability

1.1.2.1 Thermoplastic materials

In a thermoplastic material the very long chain-like molecules are held together by relatively weak Vander Waals forces. A useful image of the structure is a mass of randomly distributed long strands of sticky wool. When the material is heated the intermolecular forces are weakened so that it becomes soft and flexible and eventually, at high temperatures, it is a viscous melt when the material is allowed to cool it solidifies again [5].

The presence of crystallinity in those plastics capable of crystallising is very dependent on their thermal history and hence on the processing conditions used to produce the moulded article. In turn, the mechanical properties of the moldings are very sensitive to whether or not the plastic possesses crystalline.

In general, plastics have a higher density when they crystallize due to the closer packing of the molecules. Qpical characteristics of crystalline and amorphous type.

1.1.2.2 Thermosetting plastics

A thermosetting plastic is produced by a chemical reaction which has two stages. The first stage results in the formation of long chain-like molecules similar to those present in thermoplastics, but still capable of further reaction. The second stage of the reaction (cross-linking of chains) takes place during molding, usually under the application of heat and pressure. The resultant molding will be rigid when cooled but a close network structure has been set up within the material [9].

During the second stage the long molecular chains have been interlinked by strong bonds so that the material cannot be softened again by the application of heat. If excess heat is applied to these materials they will char and degrade. This type of behavior is analogous to boiling an egg.

Once the egg has cooled and is hard, it cannot be softened again by the application of heat. Since the cross-linking of molecules is by strong chemical bonds, thermosetting materials are characteristically quite rigid materials and their mechanical properties are not heat sensitive. Examples of thermo sets are phenol formaldehyde, melamine formaldehyde, urea formaldehyde, epoxies and some polyester [5].

Every day PET plastics are being used all over the world and after use it is discarded in the landfill. PET is the thermoplastic type of plastic and the presence of the waste PET in the landfill causes a variety of environmental and economical problems because PET does not biodegrade in landfills. Waste PET goes through photo-degradation and turns into minuscule particles, which enter the food chain and cause harmful health hazard to habitants. In the incineration process PET forms a very hard solid complex sublimate substance which causes severe environmental problems. Furthermore, incineration encourages the emission of harmful green house gases. In a statement, pacific institute showed that 1 ton of PET produces approximately 3 tons of CO_2 and only water bottled created more than 2.5 million tons of CO2 in 2006 [6] (Moinuddin Sarker 11 February 2011). PET in particular presents a major growth area in the years to come. Indian PET consumption has grown at an annual rate of 30% and the current PET consumption is estimated to be around 45,000 tons per annum. India will probably see a rise in waste generation from less than 40,000 metric tons per year to over 125,000 metric tons by the year 2030. To overcome certain demerits of recycling and incineration, researchers across the world have focused on development of value added products from waste plastics such as activated carbon, liquid fuel and gaseous product and monomer recovery [7].

## 2. EXPERIMENTAL DETAILS

2.1 Chemicals

Analytical grade of calcium hydroxide (Ca (OH) $_2$) 96% , iron oxide (Fe_2O_3) 98%, Zinc oxide(ZnO) 96% was used as precursor catalysts Activated Carbon (AC) with fine particles size was use a support for impregnation with ZnO.

2.1.1 Raw material collection and preparation

Waste plastic were collected from the hotels trash, home rubbish and market. To remove the attached waste contaminators, the collected waste plastic was washed with high pressure water. The separation process of the PET, HDPE, LDPE undertake by using the international standard looking at the bottom of the bottles. The standard had been either number & symbols or one of them. Some plastic bottles have described PET or PET 1, HDPE and LDPE on the bottom of the bottles. After separated in this way, it was cut into flakes by a shredder machine. These waste samples were determined the melting temperature by differential scanning Calorimeter (DSC) to obtain the anticipated plastics. The decomposition pattern of plastic samples was carried out a silica crucible with temperature ranging from 350°C to 6000°C at a heating rate of 100°C/min.

LDPE melting point of the waste sample and HDPE melting point of the waste sample were 140°C and 200°C respectively. But PET melting point was 260°C. All these waste plastics were used for the pyrolysis experiments.
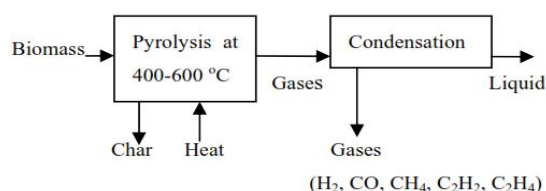
Fig. 1. A possible reaction pathway of pyrolysis of solid waste

Therefore, the conversion of plastic bottle in to liquid fuel from PET Plastic bottle used as raw materials to produce liquid fuel. It need to use pyrolyis process and also uses catalyst to obtain the liquid fuel, the temperature has also effect. PET combined with other plastic with HDPE, LDPE to get maximum yield of liquid fuel.

2.1.2 Catalyst preparation on ZnO/Ac (activated carbon)

Supported metal oxide catalysts were prepared according to the conventional incipient-wetness impregnation of aqueous solutions of the corresponding ZnO precursors on an activated carbon support (100 mesh particle size, Darco). Zinc oxide was under vigorous stirring. After four hours of reaction time, the catalyst was kept in oven for drying after that the impregnation process is ended and we can use this catalyst as ZnO/Ac [8].

Whereas, Fe_2O_3 and Ca (OH) $_2$ were laboratory standard grades, so they were used directly for the experiment purpose.

2.1.3 Experimental set up and description

The Pyrolysis Experiment was undertaking by putting the measured amount of plastic sample and catalysis in the round bottom flask, and then it was adjusted the temperature of the heater to melt the sample. After 30 minutes the melting sample changed into vapors, the vapor passed through the condenser. This condenser was attached with external cooling material chiller to condense the vaporized gas. This chiller or cooler circulated water in the outer diameter of the condenser, this could easily change the vapor into liquid fuel then it was collected by the beaker.

Fig. 2. Pyrolysis experimental set up

2.1.3.1 Effects of temperature

The 80 g of plastics samples (poly ethylene therphtalate) were loaded in each pyrolysis reaction.

For each, three different temperatures were selected that was 400°C, 450°C and 500°C. The condensable liquid products were collected through the condensers and weighed. The amount of the output was varied because of the temperature variation and the un-condensable gases such as Co, CO2, C2H4, and other less Hydrocarbons. The solid residue left out inside the reactors was weighed then the weight of gaseous product was calculated from the material balance. Reactions were carried out at different temperatures ranging from 400-500 °C the Temperature had a huge effect on the pyrolysis reaction.

2.1.3.2 Effect of PET concentration

PET on the aforementioned three Temperatures had different amount of output. But PET alone had less amount of output compared with the other types of plastics. To optimize the amount of liquid fuel which was found in the first pyrolysis process, another plastic type of HDPE, LDPE mixed with PET on different proportions were used. From the 100g of LDPE and HDPE plastics samples, the concentrations of PET were 30%, 50% and 75%.

2.1.3.3 Catalysis activity

Through the process of screen out of the catalysis among the three catalysis basically Ca (OH) $_2$, Fe_2O_3 and ZnO/AC were used. The Catalyst to plastics proportion (2:1) was subjected to pyrolysis in the reactor. The amount of metal contain of these catalysis determined the output either to increase or decrease.

2.1.3.4 Characterization of physicochemical property of liquid (diesel) fuel

The specified quality standard showed each physicochemical property of diesel fuels. Then, the characterizing processes of this physicochemical property of diesel fuels were done on the following procedures.

2.1.3.4.1 Physical properties

Specific Gravity (SG) and Density (ASTM D4052-91): The diesel fuel samples at the temperature of 25°C were filled into pyknometer (50ml) then measure the weight oil. The finding result of the specific Gravity of oil times with specific Gravity of water gave the density of oil.

Viscosity (Visco Star plus, V50001): the measurement of the viscosity by using *Visco Star plus* equipments of V50001 model, after selecting the appropriate shaft with the diameter of 0.4 mm insert into the beaker of the liquid fuel at the room Temperature (25°C) then measuring the μ dynamic viscosity (Pa.s).Kinematic viscosity ($mm2/s$) was calculated by using the following Equation (1):

$$\eta = \mu/\rho \qquad (1)$$

2.1.3.4.2 Chemical properties

Refractive index measure: To find the oil refractive index, the refractor meter equipment Mod RAM 340 at room temperature of 25°C was used. There was also a tool using to drop a small amount of droplet into the Refractive meter, after a while it was obtained Refractive index (nD).

Flash point: To measure the flash point it was used a small amount of droplet oil into distillation flask then the flask was on the heater. When it was vaporize, the gas was immediately ignited to measure the Temperature through the use of Thermometer.

Determination of high heating value (HHV): HHV was conducted using empirical equations available. The increasing iodine value (IV) [see in appendix A] (i.e. carbon double bond - C—C- content) the results in a decrease in the heat content of an oil. Therefore, for calculation of the HHV (M J/Kg) of vegetable oils Equation was suggested [9] (luisa FRios 2012).

$$HHV = 49.43 - [(0.041 \times SV) + 0.015 \times IV](2)$$

$$SV = Saponifcation\ value\ of\ oil \quad IV = Iodine\ value\ of\ oil$$

Determination of Cetane (CN): Cetane number determination was using empirical formula developed by kalayasir (luisa FRios 2012)the calculation was based on the results from the saponification value (SV) and Iodine value (IV).the CN was calculated with the help of equation

$$CN = 46.3 + 5458/SN - 0.225 \times IV \quad (3)$$

Percent yield of liquid (diesel) fuel: The percent yield of diesel fuel was based on the weight of the sample plastic, determine on the following equation:

$$\% \ \text{Yield} = \frac{Wsample - Wproduct - Wresidue}{Wsample} * 100 \quad (4)$$

Liquid fuel analysis by (Gas Chromatography): The composition of the liquid fuel was analyses by Gas chromatography (Clarus 500 Model)**.**The FID column temperature is set then run for 6-7 hour after that wash a syringe with acetone by filling the syringe completely and injecting the waste acetone onto a paper towel. Wash 2-3 times. Standard hydrocarbon from C_3-C_{62} Pull some of sample into the syringe. To remove air bubbles in the syringe by rapidly moving the plunger up and down while the needle is in the sample. Usually $1 - 2\ \mu L$ of sample is injected into the GC. The mobile phase or carrier gas was used the nitrogen gas. Then inject the sample of liquid fuel. Then the pick and retention time was displayed of liquid fuel.

2.1.3.5 Catalyst characterization

Catalyst stability was conducted with Differentia scanning colorimeter DSC (DSC 8000). After Prepared the three catalysts with each 0.1 gm powder took and set the DSC temperature range from 300-600 ^{o}c. Then it was putting in to DSC a after a while the result of catalyst stability found. The melting process results in an endothermic peak were in the DSC curve. The amount of metal in the catalyst was determined by ICP (optical Emission spectrometer) Model (Ultima 2). After prepared the standard of Zn Acetate, Iron Oxide and $CaCO_3$ then insert in ICP tube on the prepared sample of catalyst ZnO/AC, Fe_2O_3, and Ca (OH) $_2$ then measured the sample of each catalyst.
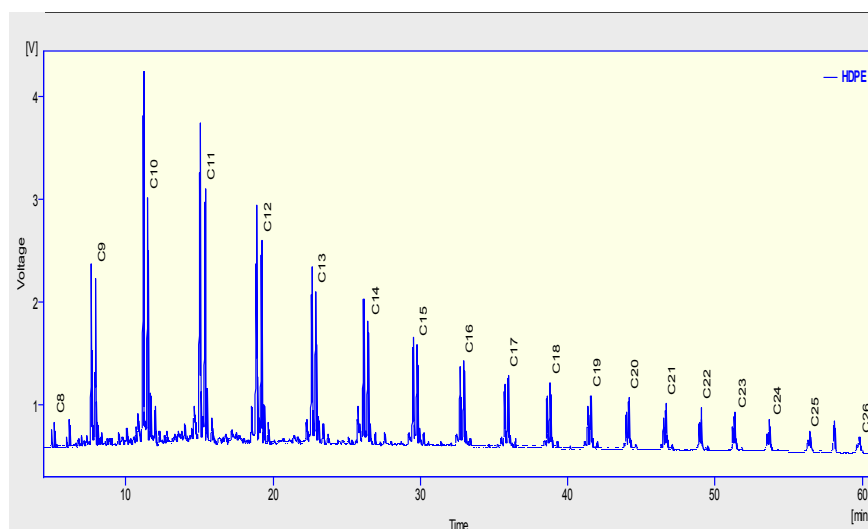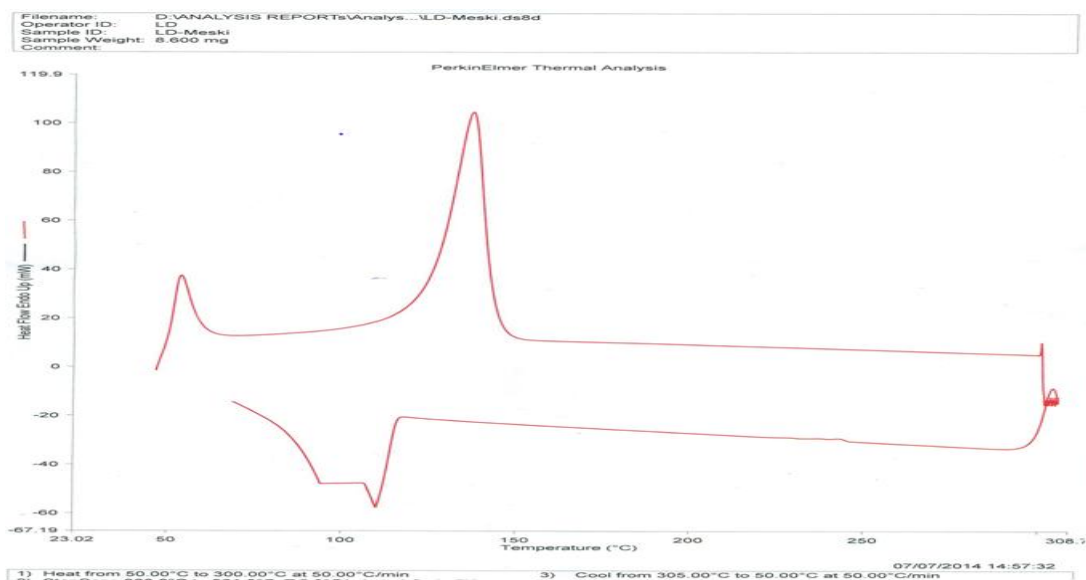

Fig. 3. The result of GC (Gas Chromatography)

Fig. 4. The result of DSC (Differential Scanning Colorimeter)

And finally, it was taking $0.5\ mg/100\ ml$ of ZnO/AC, $0.5\ mg/L$ of Fe_2O_3 and $0.1\ mg/100\ Ml$ of Ca (OH) ₂ by injecting these samples into the ICP.

3. RESULTS AND DISCUSSION

For this particular work, since the thesis is mainly focused on to screen out the effective catalyst type for the conversion of PET in to liquid fuel and to characterize the obtained standard quality of liquid (Diesel) fuel.

3.1 Catalyst Screening

The effect of catalysis type on conversion of PET waste into liquid fuel was investigated for a wide range of Temperature.

3.2 Effects of Catalyst Type and Temperature on PET Conversion into Liquid Fuel

The result obtained present in Fig. 5 As it can be seen in the figure, conversion of PET (yield) increases with Temperature for all types of catalysis. But, on the PET production, ZnO/Ac catalyst was using at 400ºC temperatures and the final oil output was 4%. The use of Fe_2O_3 catalyst was also better than ZnO/AC catalyst, and the Maximum expected PET production was 6.7% at the given range of temperature.

Whereas, the use of Ca (OH) ₂ catalyst at the three different range of temperature was better in output than the previous two catalysts especially this catalyst (Ca (OH) ₂) at 500°c temperature was the effective catalysis to obtain the highest amount of yields.

This indicates that the ZnO/Ac and Fe_2O_3 catalyst at 400°C and 450°C temperatures respectively were not that much effective to obtain the intended high amount of oil yield. Furthermore, the increasing temperature on Fe_2O_3 catalyst didn't change on the output of the oil yield, even though there was some point of difference in the production with ZnO/Ac catalyst compared with Fe_2O_3. The ZnO/Ac catalyst output was almost in between 0 and 1% when the degree of temperature was increasing on, so the difference in production was not that much significant for use of ZnO/Ac and Fe_2O_3 catalysts.

However this, as a bench mark to show how much these three different catalysts had an effect on the conversion of plastic into liquid fuel but it was impossible to get the diesel oil from the PET without additional catalysts. Thus, the increasing degree of temperature on these three different catalysts had different amount of products from 4% up to 10%, but Ca (OH)₂ catalysts at 500°C was an effective catalyst to get the best amount of oil yield.

The PET gas removal was measures by using material balance. The result obtained present in

Fig. 6. As it can be seen in the figure, on the use of ZnO/AC catalyst, the escaped gas level was rising from 52.5% to 56% when the temperature increasing from 400°C up to 500°C. To support this argument see the previous graph, with the use of ZnO/AC catalyst for the production of oil was decrease in amount because of the removal gas increased. These removal gases were CO, CH_4, C_2H_2 & C_2H_4 can be easily mixed into the environment. Whereas, on the use of Ca (OH)$_2$ catalyst, the escaped gas amount was decrease from 50% to 30% due to the increasing temperature from 400°C to 500°C. However this, reverse condition was occurred on the use of the two catalysts of ZnO/AC and Ca (OH)$_2$. When the temperature was increased; the escaped amount of gas was also decreased on Ca (OH)$_2$ and increase on ZnO/AC catalysts. But on the use of Fe_2O_3 catalyst the amount of escaped gas at the temperature of 400°C up to 450°C was become declining from 50% to 40%, at the same time, the amount of gas escaped percentage was rising from 40% to 52% when the degree of temperature increase from 450°C to 500°C.

3.3 Effect of PET Ratio

The Effect of addition of waste plastic on conversion of PET waste in to liquid fuel.

As it can be seen in Fig. 7, it was used 70% of HDPE with a composition of 30% of PET on these three different catalysts of Ca (OH)$_2$, Fe_2O_3 and ZnO/AC at different of temperature.

The amount of ZnO/AC catalyst was 13% at 400°C temperature and the oil yield was growing when the degree of temperature was increasing at 450°C and 500°C from 20% to 22% of oil yield respectively. But, when It was used Fe_2O_3, the amount of oil yield was better than ZnO/AC even at the same of temperature 400°C degree the output was 24% and at 450°C temperature 34% was acquired. Whereas, at 500°C it was slightly better than from the preceding temperature and the output of the oil yield was close to 40%.

Besides the above fact, Ca (OH)$_2$ catalyst was more effective than the above two types of catalysts. At the degree of 400°C the output was 34% and at 450°C it was also 50%, at the same time the degree of temperature increase at 500°C the product was gone beyond 50%. Thus, it can be said that the composition of 70% of HDPE with 30% of PET with the use of Ca (OH)$_2$ catalysts was obtained the maximum percentage of oil yield close to 60%. So, Ca (OH)$_2$ was the effective catalyst between the remains regarding to get the amount of the final oil yield and it was up to 60%.

When the result present in Fig. 5 (PET alone) compared with PET with the composition of HDPE had big difference regarding to the output. Both PET and PET with HDPE were used the same catalyst of Ca (OH)$_2$ but the output which was used PET alone could not go beyond 10% though; PET with HDPE was close to 60%.
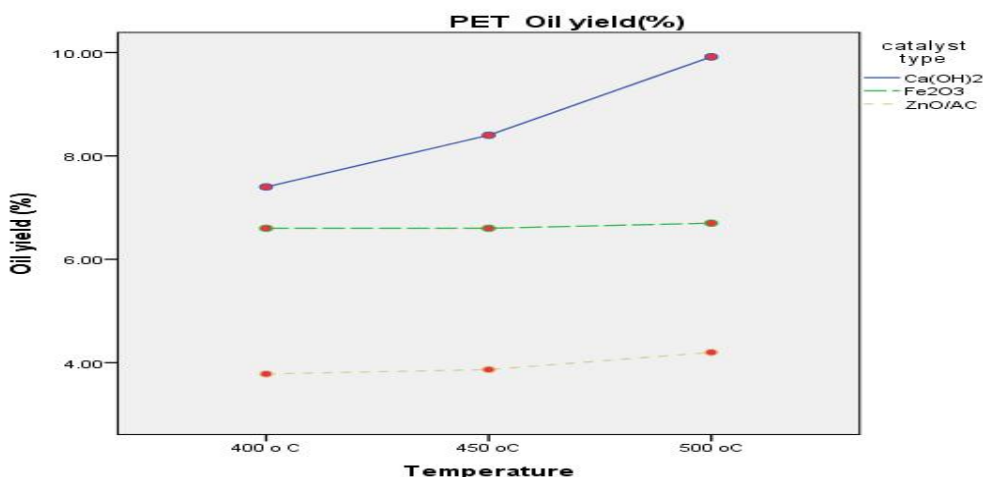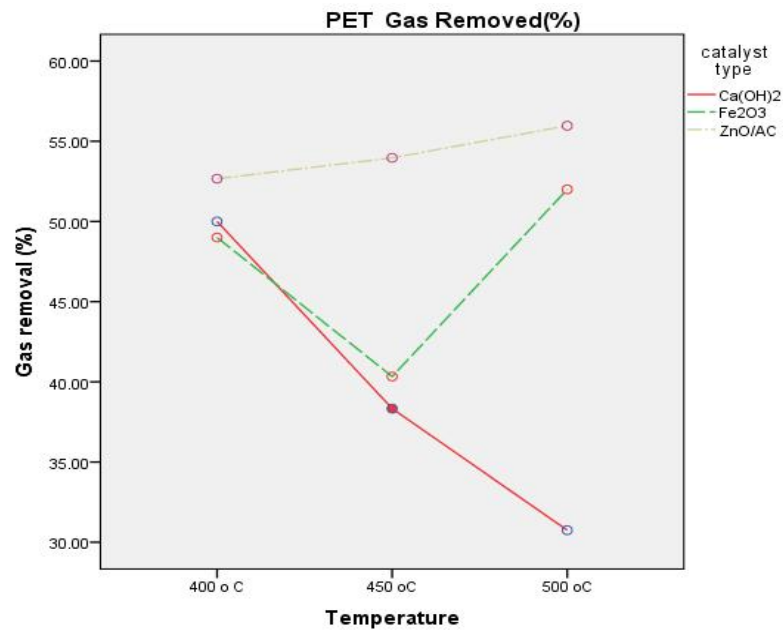

Fig. 5. Oil yield on PET
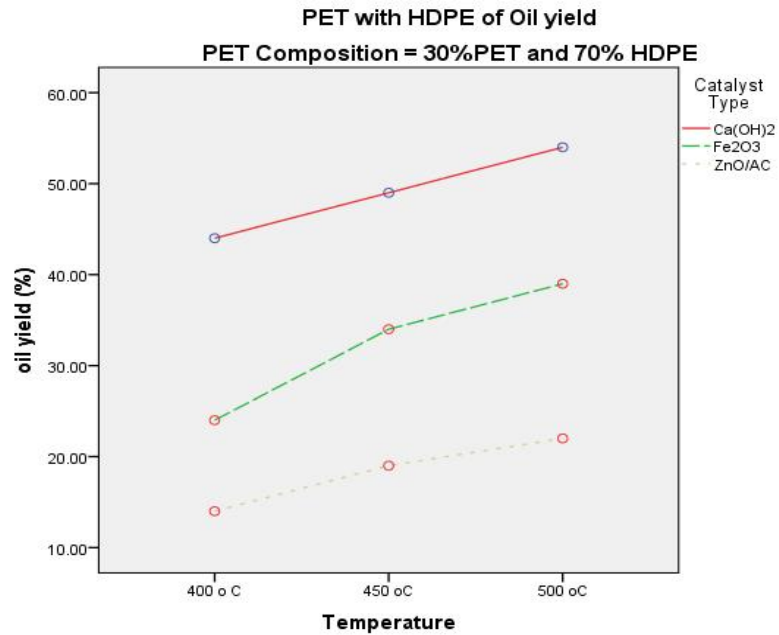
Fig. 6. The removal gas of PET

Fig. 7. PET with HDPE

There was a composition of 30% PET and 70% LDPE, to obtain the maximum oil yield and the ZnO/AC catalyst result was within the range of 25% up to 27.5% at the temperature of 400 °C to 500°C; on the use of Fe_2O_3 catalyst the product was growing up from 30% to 32%, when the temperature was increase from 400°C to 450°C but, the result was reached up to 34% at the degree of 500°C. Due to the use Ca (OH) $_2$ with the composition of 30% PET and 70% LDPE the output yield had 35% at the temperature of 400 °c and the oil yield were become growing from 40% to 45% at the temperatures of 450°C & 500ºC degree respectively. Therefore, with these

kinds of compositions of 30% PET and 70% LDPE, when the temperature rising, the amount of output was also increase. So, with the above composition on the use of Ca (OH)$_2$ at the degree of 500°C temperature, the maximum amount of oil yield was 45%.

With the use of 50% PET with the composition of 25% of HDPE and LDPE was used with the ZnO/AC catalysts at the temperature of 400°C, it was almost 23% oil yield obtained. The oil yield was rising 25% at 450°C and 26% at 500°C. And the use of Fe$_2$O$_3$ catalyst at 400°C the acquired oil yield was 26% and at the temperature of 450°C and 500°C it was obtained 27% and 30% oil yield respectively but Fe$_2$O$_3$ catalyst had a better output than ZnO/AC catalyst. Whereas, Ca (OH) 2 catalyst of 50% PET with 25% of both HDPE and LDPE composition was more effective catalyst than the other two catalysts of ZnO/AC & Fe$_2$O$_3.$ Because at the temperature of 400°C the acquired oil yield was 26%, at the 450°C it was also 29%; but at 500°C the obtained oil yield output was the maximum amount of above 32%.

The composition of 30% of PET with 70% of HDPE and LDPE was used with the ZnO/AC catalysts at the temperature of 400°C, the obtained output was 37% and the amount of oil yield was also slightly increase when the temperature becomes raising up at the temperature of 450°C the result was 38% and at 500°C it was also 40%. While, on the use of Fe$_2$O$_3$ catalyst at the temperature of 400°C it was

obtained 34% of oil yield; moreover this, at 450°C and 500°C temperature the result was 40% and 48% respectively. But, Ca (OH) 2 with the composition of 30% of PET with 70% of HDPE and DPE was used and 44% obtained at the temperature of 400°C, as well as the degree of temperature became increasing the amount of oil yield output were dramatically increasing at 450°C it was 53% and at degree of 500°C the product was 84%. Hence, the composition of 30% of PET with 70% of HDPE and DPE with among the three different catalysts at different range of temperature from 400°C to 500°C were used and the result which was obtained as the output were different.

3.4 Physical and Chemical Properties of Characterization of PET Liquid Fuel

The importance of characterization is helping to identify easily which types of diesel oil were obtained on the experiment and through the use of physical and chemical parameter based on Table 1 can compare the experiment result with the standard and for identification of benzene for this diesel was analysis by Gas chromatograph.

Based on the Table 1, on the use of PET without any other composition, the intended oil was strongly similar with kerosene and PET with the composition of HDPE and LDPE obtained oil was crude oil. These crude oil and kerosene can help the nation by providing as an additional source of diesel oil.
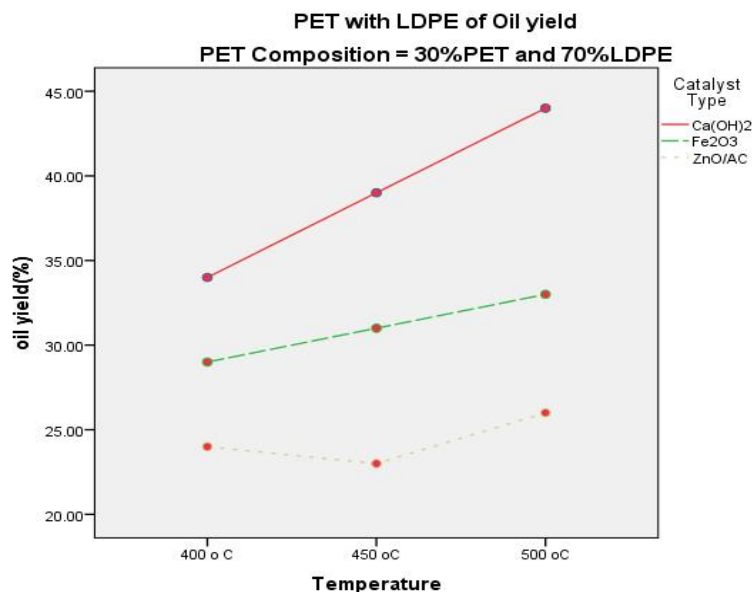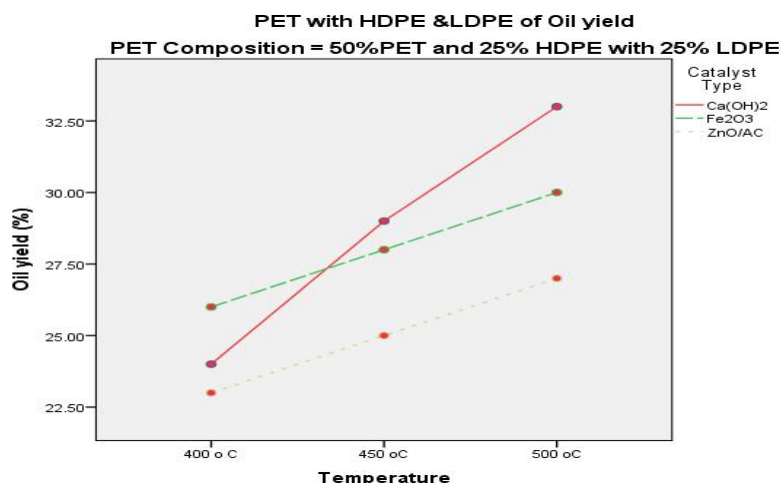

Fig. 8. PET with LDPE
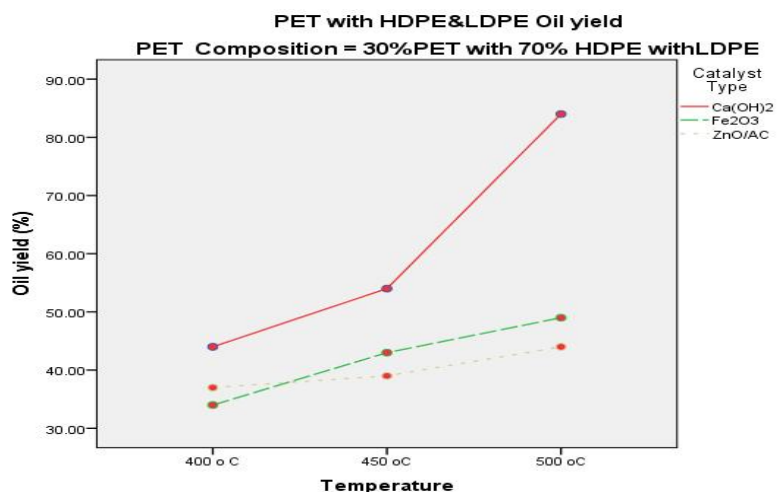
Fig. 9. PET with HDPE & LDPE

Fig. 10. PET with a combination of HDPE & LDPE

Table 1. Standard parameter of diesel oil

Parameters	Standard value		Experimental result	
	Crude oil value	Kerosene value	PET	PET with LDPE and HDPE
Density kg/m3	746-856	780- 810	780	732
Viscosity centipoises	0.25-0.413	0.2- 0.4	0.4	0.3
Flash point	62-96	37- 65 °C	49°C	80 ^{O}C
Cetane number	38-60	38-60	58	58
Heating value MJ/kg	42-46	43.1- 46	45.6	45.6
Referactive index	----	----	1.44	1.44

4. CONCLUSION

The conversion of PET in to liquid fuel with catalysts and the intended oil output at 500°C of temperature had obtained the amount of 10% oil yield as a maximum amount. But, to optimize the product of oil yield, PET combined with others plastics like HDPE and LDPE at different ratios is more effective. In my research, the combination of PET with others had their own great contribution to raise the intended amount of diesel oil with 500°C Temperature and the result was reached more than eight times from the maximum percentages of PET oil yield (84%). It

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can reach on the conclusion that the most effective catalyst obtained through this experiment is Ca $(OH)_2$ at 500°C.

Temperature has played a great role for the conversion of plastics in to liquid fuel, when the temperature rise the thermal catalytic reaction is also increase. At the temperature of 400°C and 450°C the amount of oil yield vary from 40% to 60% whereas, at 500°C the obtained oil yield is more than even 60%.

To minimize the residue amount the temperature should be increasing on both Ca $(OH)_2$ and Fe_2O_3 catalysts. But the best least remains had on the use of Ca $(OH)_2$ at the maximum degree of 500°C. GC (Gaschromatography) was used to identify the Benzene from the obtained crude oil and it helps to identfy the hydrocarbon categories. The final output of the oil yield under this equipment shows there is Benzene. Thus, catalyst screening of Ca $(OH)_2$ and identifying effective Temperature of 500°C could help to get Benzene.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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APPENDIX A

Experimental Producer

Determination of Saponification Number (SN): Indicator method was used as specified by ISO 3657 (1988). The SN determination was conducted by dissolving the oil in an ethanol KOH solution. 2 g of the sample was weighed into a conical flask, and then 25 ml of 0.1 N ethanol potassium hydroxide solution was added. The content was constantly stirred, and allowed to boil gently for 60min. A reflux condenser was placed on the flask containing the mixture. Few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5 M HCl (volume Va was recorded) to the endpoint until the pink color of the indicator just disappeared. Then a blank determination was carried out upon the same quantity of potassium hydroxide solution at the same time and under the same conditions and (volume Vb was recorded).

$$SN = 56.5 * N (Vb-Va)/W$$

Where W= weight of oil taken in gram, N= normality of HCL solution, Va= volume of HCL solution used in test in ml, Vb= volume of HCL solution used in blank in ml.

Determination of Iodine Value (IV): The method specified by ISO 3961 (1989) was used. 0.4 g of the sample was weighed into a conical flask and 20ml of carbon tetra chloride was added to dissolve the oil. Then 25ml of Dam's (Iodine monochloride) reagent was added to the flask using a safety pipette in fume chamber. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark place for 2.5 hours. At the end of this period, 20 ml of 10% aqueous potassium iodide and 125 ml of water were added using a measuring cylinder. The content was titrated with 0.1 N Sodium-thiosuphate solutions until the yellow color almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding sodium thiosuphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples. The iodine value (IV) is given by the expression

$$IV = (12.69 * N (Vb-Va))/W$$

Where,

N = normality of sodium thiosuphate, V1 = Volume of sodium thiosuphate, V2 = Volume of sodium thiosuphate used for blank, M = Mass of the sample.