



Characterization of the Liquid Fuel Produced from Catalytic Depolymerization of Polymeric Waste Using Batch Reactor

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ABSTRACT

The high rate of generation of plastic waste in the country and the fact that all other means of Municipal Plastic Waste (MPW) management techniques had failed leading to the requirement of efficient and alternative disposal technique-depolymerization. The technique involves heating the polymeric waste at an elevated temperature in an inert environment to produce condensable, non-condensable, hydrocarbon and biochar. The plastic waste was collected at the Ilokun dumpsite in Ado-Ekiti, southwest Nigeria. Each component of the waste samples was depolymerized in a batch reactor without the use of a catalyst and with the addition of 10 g of activated carbon (AC) and calcium oxide (CaO) as catalysts. The liquid fuels which were produced between the temperature range of 219 and 232 were blended with standard fuel. Fuel samples with conventional diesel and depolymerized plastic diesel were characterized based on ASTM standards. The results of the proximate and ultimate analysis indicated that percentage moisture content ranges from 0.00-0.18%, volatile matter ranges between 96.66-99.75% and percentage ash content ranges from 0.13-3.03%. Fixed carbon ranges from 0.004-0.31% while the Gross Heating Value (GHV) ranges from 42.66-45.87 MJ/kg. The CHONS analyzer indicated the percentage of carbon, hydrogen, oxygen, nitrogen, and sulfur content range 81.64-85.51%, 12-31-18.04%, 0.00-1.51%, 0.00-0.73%, and 0.10- 0.97% respectively. The results of the physiochemical properties of the samples show that the density, API gravity, Kinematic viscosity and Flash point vary from 0.76-0.83 (g/cm³), 38.98-54.68, 17-2.80 (cm²/s) and 50.0-70.0 (°C) respectively while Cloud point, Pour point, Fire point and Cetane index range from -20-15.0 (°C), -23-7 (°C), 61.0-79.0 (°C) and 38.50-47.0. The pH values of the liquid fuel samples vary from 6.60-3.30. The overall results of the characterization indicated the fuel samples have proximity to the properties of the conventional diesel following the ASTM D975, ASTM D4737, ASTM D1298, ASTM D445, ASTM D2709, and ASTM D482 standards. The depolymerized polymeric waste is sustainable, with a low cost of production. Hence a good substitute as an alternative fuel and means of wealth creation from waste.

INTRODUCTION

There is a gradual phasing out of ceramics and cans in the utilization cycle due to their nature and chemical composition. This has led to the replacement of polymeric materials and consequently the generation of municipal plastic waste in the metropolis. The rise in the usage of non-biodegradable plastic products has had negative repercussions, such as clogging drains, smothering some animals who mistakenly eat them, making ground surfaces impermeable to water, and posing a number of other risks. The challenge then becomes figuring out a secure way to transform these

plastic waste products into other beneficial and safe goods.

Plastic can be understood as polymeric, synthetic, or semi-synthetic materials that are made of big, organic molecules known as monomers. Polymers are the big molecules created during the polymerization process, according to Hazzan (2003). Polymers that are thermosetting and thermoplastic. Thermoplastics are a type of plastic that can be reshaped even after solidification since heat exposure does not cause chemical changes in its makeup. Thermosetting, on the other hand, refers to non-recycled polymers that, when heated, go

through an irreversible chemical transformation; they melt and adopt a shape only once before being immobile. It is impossible to overstate the importance of plastics in human life, which includes their use as furniture, toys, vehicle parts, household appliances, packaging materials, drinkable water and beverage containers, cooking utensils, and kitchenware (Abota 2012).

The reason plastic is used so frequently is because it is typically lightweight, affordable, and durable, which accounts for its favor over other materials (Hopewell et al. 2009). Every nook and cranny of the Nigerian terrain is littered with empty polythene plastic containers. Plastic is a non-biodegradable material that, when it gets into the soil, stops water from reaching the roots of plants and limits how far their roots may spread. Some of it prevents plants and animals from exchanging gases necessary for breathing (Rominiyi et al. 2017).

Unplanned garbage disposal by the side of the road, which creates an eyesore and hinders the free flow of run-off and cars, frequently leads to an accident during the busiest times of the day (Temitope et al. 2015). Some of the trash that is left on the streets makes its way into trenches, canals, and drains, where it clogs them and prevents water from flowing freely, which causes flooding. Municipal Plastic Wastes (MPW) have a severe influence on the environment, which is being exacerbated by the growing population and rising predilection for packaged goods (Kalilu 2013).

During the height of the rainy season, plastic waste is seen to accumulate on river banks after it has rained, seriously impeding the river's flow and ultimately causing the river bank to overflow (Kalilu 2013). As a result, a serious disaster occurs that results in the loss of many lives and extensive property damage. Recovering discarded or wasted plastics and processing them into useable items-sometimes products that are radically different from what they were in the first place is known as recycling plastic. Numerous academics have conducted various studies on the recycling of plastic garbage in the past. Trevor (2019), took into account the historical facets of plastics and the overdependence on polymeric materials for plastic waste. He emphasized the negative effects this material's use and disposal have on the ecosystem. A summary of the types and amounts of plastics in the trash stream is given by Vanessa (2007). Primary, secondary, tertiary, and quaternary recycling were the four categories of recycling outlined by Kalilu (2013). While a large portion of industrial wastes are not biodegradable, horticulture wastes are, and as a result, represent a hazard to health, drainage, and urban planning.

Temperature, reactor type, pressure, residence duration, fluidized bed type, and flow rate are a few variables that

affect how well plastic is pyrolyzed. Temperature is the most important of these variables because depolymerization involves the breaking or cracking of the chemical connection between the molecules. High temperatures facilitate the breakdown of chemical bonds (Sharaddin et al. 2018). Each type of plastic material has a different optimum temperature for pyrolysis. Low-Density Polyethylene (LDPE) ranges from 360 to 550 °C, while Polyethylene Terephthalate (PET) has an ideal temperature range of 350 to 520 °C. High-density polyethylene (HDPE) operates within a somewhat close range, operating between 378 and 539 °C (Anuar Sharaddin et al. 2016).

Due to the extremely slow rates of plastic waste decomposition, landfilling is not a viable alternative. The use of incinerators produces some air pollutants that have a negative impact on the environment. Therefore, techniques for recycling and recovering garbage have been utilized to lessen their negative effects on the environment and to lessen the harm that plastic waste causes. One of the most promising techniques for recovering used plastics is chemical recycling through the pyrolysis process, which involves the thermo-chemical breakdown of organic and synthetic materials at high temperatures without oxygen to produce fuels (Aguado et al. 2007) The procedure is typically carried out at temperatures ranging from 400°C to 800°C.

Aboulkas et al. (2010) looked into the behavior of polymers as they degraded thermally. For non-isothermal kinetic outcomes, the activation energy and the reaction model of the pyrolysis of polyethylene (PE) and polypropylene (PP) have been determined. The "contracting sphere" model can be used to explain the pyrolysis reactions of polyethylene, whereas the "contracting cylinder" model can be used to describe the reactions of polypropylene. The design of the cracking reactor has significant difficulties due to the high viscosity and limited thermal conductivity of plastics (Aboulkas et al. 2010).

PET boosted the production of new chlorinated hydrocarbons in liquid products and dramatically decreased the production of inorganic chlorine content when it was present in model mixed plastics and MPW. When MPW was converted, the contaminants were hazardous for acidic catalysts and caused easy catalyst deactivation (Wang & Wang 2011).

In 2009, Lee looked at upgrading pyrolytic oil made from municipal plastic waste (MPW) using FCC Fluid Cracking Catalyst. Due to greater cracking residue, the addition of FCC catalyst to the degrading process resulted in improved liquid and gas yields as well as a high fraction of heavy hydrocarbons in the oil output. Waste PE, PP, and PS have also been used to study non-catalytic pyrolysis. As

a result, waste PS created more liquid while waste PE and PP produced more gaseous products, according to the results (Demirbas 2004). While the gaseous product can be utilized as a heating source for the reactors or as a cooking gas stove application, the oil produced can be used in a pressured cooking stove. For co-firing with coal and biomass, which can be used as fuel for various purposes, the solid products will be utilized (Demirbas 2004).

Experimentally, HDPE, PP, and LDPE have calorific values that are all greater than 40 MJ/kg and are therefore regarded as having high energy usage. Due to the presence of an aromatic ring in its chemical structure, which has less combustion energy than an aliphatic hydrocarbon, PS typically has a lower calorific value than polyolefin plastic (PO) (Onwudili 2009). Due to the presence of benzoic acid in PET and chlorine compound in PVC, which reduced the fuel quality, these two materials had the lowest calorific values overall, falling below 30 MJ/kg. The low calorific value of PET was explained by the aromatic ring component of benzoic acid (Onwudili 2009). Both Cullis et al. (1981) and Panda et al. (2010) suggested a thorough investigation of the process of polymer heat degradation. The characterization and comparative analysis of conventional diesel and depolymerized plastic waste liquid fuel was done to suggest whether the latter can be used as an alternative to conventional diesel.

MATERIALS AND METHODS

Materials used for this characterization including but not limited to the following: Raw HDPE liquid fuel sample; Activated carbon catalyzed HDPE liquid fuel sample; Calcium oxide catalyzed HDPE liquid fuel sample; Raw LDPE liquid fuel sample; Activated carbon catalyzed LDPE liquid fuel sample; Calcium oxide catalyzed LDPE liquid fuel sample; Raw HDPE/LDPE mixture 1:1; liquid fuel sample; HDPE/LDPE Activated carbon catalyzed mixture 1:1 liquid fuel sample; HDPE/LDPE Calcium oxide catalyzed mixture 1:1 liquid fuel sample; HDPE/ Conventional Diesel Blended 1:1 liquid fuel sample; HDPE/Conventional Diesel Blended 1:2 liquid fuel sample; HDPE/Conventional Diesel Blended 2:1 liquid fuel sample; LDPE/ Conventional Diesel Blended 1:1 liquid fuel sample; LDPE/ Conventional Diesel Blended 1:2 liquid fuel sample; LDPE/ Conventional Diesel Blended 2:1 liquid fuel sample; Conventional diesel; Kerosine; Petrol/ Gasoline; Silica crucible; Muffle furnace; Beaker; Two mouthed conical flask; Thermometer; Desicator; Bomb calorimeter; CHNS/O Elemental Analyzer; Pycnometer; Programmable Rheometer; Oakion ion-700 pH Meter.

The proximate analysis was conducted to ascertain the fuel sample's moisture content, volatile matter, ash content,

and calorific value. The mass of the silica crucible was determined by employing a digital weighing balance and denoted as W1 (g). A mass of 1.00 g of the samples was introduced into the crucible. The contents contained within the silica crucible were quantified and documented as W2 (g). The sample was thereafter subjected to thermal treatment within an oven, whereby it was exposed to a temperature of 105°C for one hour. The crucible is carefully removed from the experimental setup, allowed to cool in a desiccator to reach room temperature, and afterward weighed to determine its mass. The procedure of subjecting the sample to several cycles of heating, cooling, and weighing was iterated until a consistent mass of the anhydrous sample, denoted as W3 (g), was achieved. Equation (1) was employed to ascertain the percentage moisture content of the specimen.

$$\% \text{ Moisture content} = \frac{W_2 - W_3}{W_2 - W_1} \times \frac{100}{1} \quad \dots(1)$$

The determination of the volatile matter content was conducted using the method of igniting the sample at a temperature of 950°C. The weight of the moisture-free sample in the silica crucible was measured using a digital weighing balance and recorded as W3(g). The provided sample was subjected to additional heating within a crucible that was equipped with a cover. This heating process took place in a muffle furnace, and the temperature was set to 950°C. The duration of this heating process was precisely 7 minutes, adhering to the guidelines outlined in ISO 1974/562. The sample was subjected to cooling within a desiccator and afterward measured using a digital weighing balance, yielding a weight of W4 (g). The calculation of the volatile matter percentage in the sample was conducted using equation (2).

$$\% \text{ Volatile Matter} = \frac{W_3 - W_4}{W_3 - W_1} \times \frac{100}{1} \quad \dots(2)$$

Where:

$W_3 - W_4$ is the loss in weight of the moisture sample

$W_3 - W_1$ is the initial weight of the moisture-free sample

The crucible was measured using a computerized weighing balance and the recorded value was denoted as W1(g). A spatula was employed to transfer a mass of 1.00 g of the sample into the silica crucible. The weight was subsequently measured and documented as W6(g). The specimen contained within the exposed crucible was subsequently incinerated (in the presence of oxygen) at a temperature of 750 °C within a muffle furnace until a consistent mass was attained. The residual ash was quantified and documented as W7 (g). Each of the combustible components of the fuel samples underwent three repetitions of the technique. The equation denoted as (3) was employed

to ascertain the proportion of ash content present in the given sample.

$$\% \text{ Ash content} = \frac{W_7 - W_1}{W_6 - W_1} \times \frac{100}{1} \quad \dots(3)$$

Where:

$W_7 - W_1$ is the weight of residual ash formed.

$W_6 - W_1$ is the weight of liquid fuel initially taken

The methods used in the determination of other parameters, such as ash content; calorific value; fixed carbon. Fixed carbon can be found in the work of Rominiyi (2017). The results are presented in Table 1.

The CHNS/O content in the sample was determined through the application of ultimate analysis utilizing a CHNS/O Elemental Analyzer. This analysis concurrently displays the weight percentages of carbon, hydrogen, oxygen, nitrogen, and sulfur in the sample, with the weight percentage of oxygen being obtained using a difference calculation.

The physical properties of liquid fuel, such as density, kinematic viscosity, specific gravity, pH value, pour point, flash point, cloud point, and cetane number of the prepared liquid fuel and the conventional diesel were analyzed with the standard equipment meant for that purpose and in accordance with ASTM standard and can be found in the work of Rominiyi (2017). The density was determined at 40. An empty density bottle (pycnometer) was weighed and recorded as W_1 . Afterwards, the density bottle was filled with water and the weight was recorded as W_2 . Furthermore, the bottle was emptied and properly cleaned the fuel sample was poured into the bottle and the weight was recorded as W_3 . The density of the fuel was determined using equation (4). This was repeated for other samples, and their specific gravity and API gravity were calculated.

$$\text{Specific gravity } 60/60 \text{ } ^\circ\text{C} = \frac{W_o}{W_w} = \frac{W_3 - W_1}{W_2 - W_1} \quad \dots(4)$$

$$\text{Density of fuel at } 60 \text{ } ^\circ\text{C} = \text{Specific gravity} \times 1 \text{ g/cm}^3 \quad \dots(5)$$

The process of fractional distillation was quantified utilizing the ASTM D86 standard. The initial boiling point (IBP) refers to the temperature recorded by a thermometer when the first droplet of condensed vapor is observed emerging from the lower portion of the condenser tube. A lower initial boiling point (IBP) indicates the existence of lower hydrocarbon compounds within the sample. A continuous distillation method was employed to distill the pyrolyzed liquid fuel derived from polymeric waste into diesel-range fractions. The device has a notable degree of separation efficiency when it comes to the fractionation of gasoline and diesel cuts derived from pyrolysis hydrocarbon mixtures. The experimental setup comprises a 35 L round-bottomed flask made of stainless steel, which is affixed to

an aluminum support frame. The process of filling occurs through a 100 mm neck, which includes a viewing glass while draining is facilitated by a drain valve located at the bottom. The distillation column DN80, composed of borosilicate glass, is outfitted with a wire mesh packing to achieve a significant distillation rate accompanied by a notable separation efficiency. The fractions are systematically allocated to six individual receivers, each with a capacity of 20 liters, by an automated fraction collector. The software connected to the processor control unit DCD4001 regulates many distillation parameters, including temperature, vacuum, reflux ratio, and fraction collector.

The cetane index serves as a metric for assessing the igniting characteristics of diesel fuel within an internal combustion engine. This index can be derived by the analysis of density and distillation data. An alternative approach is the utilization of ASTM D976, in which the calculation of the cetane index (CI) is based on the outcome derived from the density measurement at a temperature of 15°C and the mid-boiling temperature of the specimen. ASTM D4737 might also be employed for its acquisition. The cetane index (CI) was determined using the “2” point method and density measurement in accordance with the ASTM-D976 specification at a temperature corresponding to 50% recovery. The cetane index (CI) was determined using equation (6) in accordance with the work of Joshua et al. (2009) and Dooley et al. (2012),

$$CI = 45.2 + 0.0892T_{10N} + (0.131 + 0.901B)T_{50N} + (0.0523 - 0.42B)T_{90N} + 0.00049(T_{10N}^2 - T_{90N}^2) + 107B + 60B^2 \quad \dots(6)$$

$$\text{where } N_{10H} = N_{10} - 215 \quad \dots(7)$$

$$N_{50H} = N_{50} - 260 \quad \dots(8)$$

$$N_{90} = N_{90} - 310 \quad \dots(9)$$

T_{10} , T_{50} , and T_{90} are the distillation recovery temperatures at in degrees Celsius; at 10 %, 50%, and 90 % (WV) respectively.

$$B = \left(\text{Exp}^{-0.0035D_N} \right) - 1 \quad \dots(10)$$

$$D_H = D - 850 \quad \dots(11)$$

D is the density at 15

Proximate and Ultimate Analysis

The proximate and ultimate analyses of different fuel samples and conventional diesel shown in Table 4.1 indicated that all the samples had negligible moisture content and varied between 0.00% and 0.18% with minimum for conventional diesel and maximum for HDPE/Diesel blended 2:1 sample. The percentage volatile matter (VM) of the samples tested

Table 1: Proximate, ultimate, and physical properties analysis of liquid fuel samples and conventional diesel.

Parameters	Standard (Conventional Diesel)	HDPE/ CaO	HDPE/ AC	HDPE (Raw)	LDPE/ CaO	LDPE/ AC	LDPE (Raw)	HDPE/Standard Diesel Blended		
								2:1	1:2	1:1
Proximate Analysis (wt%)										
Moisture Content (MC) (%)	0.00	0.12	0.11	0.08	0.14	0.14	0.14	0.18	0.14	0.16
Volatile matter (VM) (%)	96.66	99.73	99.75	99.61	99.70	99.68	99.63	96.68	98.15	97.94
Ash Content (AS) (%)	3.03	0.15	0.13	0.32	0.15	0.15	0.18	2.41	1.18	1.15
Fixed carbon (FC) (%)	0.31	0.01	0.01	0.004	0.04	0.03	0.05	0.09	0.02	0.01
Gross Heating Value GHV MJ kg ⁻¹	45.38	45.27	44.94	43.29	45.06	44.63	42.66	42.85	45.01	44.42
Ultimate Analysis (wt%)										
Carbon (%)	85.51,	82.25	82.49	81.64	84.37	84.62	84.62	82.81	82.82	82.61
Hydrogen (%)	12.34	17.44	17.2	18.05	15.45	15.16	15.16	13.97	13.44	14.29
Oxygen (%)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.48	1.51	1.48
Nitrogen (%)	0.08	0.00	0.03	0.00	0.00	0.03	0.03	0.66	0.73	0.61
Sulfur (%)	0.40	0.3	0.31	0.3	0.18	0.15	0.15	0.10	0.14	0.97
Physical Properties										
Density (g/cm ³)	0.83	0.78	0.76	0.79	0.79	0.78	0.81	0.77	0.76	0.77
API Gravity	38.98	49.91	54.68	47.61	47.61	40.91	43.91	52.27	54.68	52.27
Kinematic Viscosity (cm ² /s)	2.54	2.69	2.70	2.68	2.77	2.80	2.75	2.22	2.17	2.20
Flash Point (°C)	68.0	55.0	53.50	52.0	68.0	70.0	65.0	53.0	55.0	50.0
Cloud Point (°C)	-20	8.0	8.0	6.0	13.0	10.0	15.0	10.0	10.0	11.0
Pour Point (°C)	-21	-8.0	-11.0	-8.0	-10	-14	7.0	-16.0	-20.0	-23.0
Fire Point (°C)	78.0	65.0	62.5	61.0	78.0	79.0	74.5	64.0	64.5	61.5
Cetane Number	47.0	40.0	38.50	40.0	42.0	42.0	45.0	47.0	46.0	44.0

ranged from 96.66% (for conventional diesel) to 99.75% in HDPE/AC. All the samples have the best rate of VM greater than 80% (Ndecky et al. 2022). The ash content of the sample tested ranges from 0.13% minimum for HDPE/AC and 3.03% maximum for conventional diesel. This suggested that all the samples tested should be considered as an alternative fuel since high ash content can result in an increment in combustion remnant resulting in a reduction in the heating effect of the fuel. Fixed carbon contents vary between samples with HDPE (Raw) having the minimum value and conventional diesel having the maximum value of 0.31%. All the samples tested had fixed carbon lower than 40%. The GHV MJ kg⁻¹ obtained ranged from 14.94 MJ kg⁻¹ minimum (for HDPE/AC) and 45.6 MJ kg⁻¹ maximum (for LDPE/CaO). The three (HDPE/CaO, HDPE/AC, HDPE (Raw)) have the lowest GHV (14.94 – 17.29 MJ kg⁻¹) while the three (LDPE/ CaO, LDPE/AC, LDPE (Raw)) along with conventional diesel have higher GHV (42.66 – 45.38 MJ kg⁻¹). LDPE and conventional diesel. All the samples indicated a proximity to the calorific value of conventional diesel. The ultimate analysis of the samples are results are summarized in Table 4.1.

Physical Properties of the Liquid Fuel Samples

In terms of physical properties, the density of the samples

Table 2: The pH values of the liquid fuel samples.

S/N	Liquid Fuel Samples	pH Values
1.	HDPE Raw	3.62
2.	HDPE / AC	3.50
3.	HDPE/ CaO	6.60
4.	LDPE Raw	3.75
5.	LDPE/AC	3.60
6.	LDPE / CaO	3.80
7.	LDPE/ Diesel Blended 1:1	3.58
8.	LDPE/ Diesel Blended 1:2	3.52
9.	LDPE/ Diesel Blended 2:1	3.30
10.	HDPE/ Diesel Blended 1:1	3.90
11.	HDPE/ Diesel Blended 1:2	4.32
12.	HDPE/ Diesel Blended 2:1	4.10
13.	Diesel	4.70
14.	Petrol	5.06
15.	Kerosine	3.60

tested is less than 1 g/cm^3 and varies between 0.76 g/cm^3 minimum (for HDPE/AC and HDPE/Conventional Diesel blended) and 0.83 g/cm^3 maximum (for conventional diesel). LDPE (Raw) of 0.81 g/cm^3 has a close range to the conventional diesel density. The API gravity determined range from 38.98 – 54.68 for conventional diesel HDPE/AC and HDPE/Diesel blended 1:2. The Kinematic Viscosity of the samples varies from 2.17-2.80 with minimum value for HDPE/conventional diesel blended 1:2 and maximum value for LDPE/AC. The flash point of all other samples apart from LDPE/AC and LDPE/CaO is relatively lower than the flash point of conventional diesel. The cetane number of the sample product ranged from 3.50-47.0 with a minimum for HDPE/AC and a maximum for HDPE/conventional diesel blended 2:1 which is comparable to the cetane number of conventional diesel. The higher the cetane number of the fuel the higher the level of auto ignition in the internal combustion engine.

Table 4.2 shows that the pH values of all the liquid fuel samples tested indicated that they are all acidic with acidity strength range from 6.60-3.30 (HDPE/ CaO-LDPE/ Diesel Blended 2:1). This shows that HDPE/CaO is better when used in an engine because it has the least tendency of corroding the fuel tank since the acidity strength is less than that of the conventional diesel of 4.70 pH value.

CONCLUSION AND RECOMMENDATIONS

The negligible moisture content, sulfur, nitrogen, ash, and high fixed carbon, cetane number volatile matter, and hydrogen content in all the samples is a good reason to justify that the liquid fuel produced through depolymerization of polymeric waste has the tendency to act as a good substitute to conventional diesel hence an alternative energy resource. The range of results obtained as physiochemical properties also vindicated that depolymerized liquid fuel samples have a good quality of a potential energy resource. pH test conducted revealed that all the liquid fuel samples are acidic and the lower the acidity level the better the fuel. The higher the degree of acidity level of the fuel the higher the tendency to cause rusting and corrosion in an internal combustion engine. That justifies the least acidity among the samples (HDPE/ CaO) as the best alternative to run the engine. The liquid fuel obtained through the depolymerization process can further be distilled to obtain purer samples that can be used directly to run the engine and its quality can be improved by blending.

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