

Non-catalytic and catalytic pyrolysis of low-density polyethylene (LDPE) plastic waste into fuel-ranged hydrocarbons using Nigerian local clay composites

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Abstract:

Plastic utilisation and plastic waste accumulation is rising in recent times mostly in developing countries. The utilization of modified and unmodified Nigerian bentonite clay as catalyst for preparation of fuel-ranged hydrocarbons from low-density polyethylene (LDPE) sachet wastes by pyrolysis was investigated. Pyrolysis of LDPE was done using locally fabricated semi-batch reactor condenser column. The products were characterized using Fourier transform infra-red spectroscopy (FTIR) and gas chromatograph with a flame ionisation detector (GC-FID). Physicochemical properties of the products were determined using standard analytical methods. The products from non-catalysed contained mostly saturated hydrocarbons compared to that of the catalytic process which contained more branched-chain hydrocarbons and mono-substituted aromatics. Physicochemical properties of the product from non-catalytic process showed resemblance to those of kerosene and diesel fuel while the properties of products from catalytic pyrolysis closely approximates those of commercial gasoline. This implies that the catalytic process gave products that were more suitable as fuels for auto engine which require higher octane number based on their naphthenic and aromatic content.

Keywords: Plastic waste; pyrolysis; fuel-ranged hydrocarbons; mesoporous clay composites

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1. Introduction

Plastics are high molecular weight organic compounds or polymers that have a wide range of applications in recent years. Global advances in science and technology have led to changes in pattern of material consumption, energy demand and utilization, hence plastics is vast replacing the use of tradition materials such as metals and steel. In the last 100 years, plastic consumption and utilization has increased from 10 million tons to over 50 billion tons per annum in 2013 (Raja & Murali 2011). More than 70 % of these huge plastics are single use or single application and thereafter passes into waste stream as plastic wastes. In Japan for instance, 15 million tons of plastic is produced annually which gave over 10 million tons waste plastic annually (Thahir et al. 2019). Depletion of oil resources and increase in energy demand have driven researchers to seek ways to convert these waste plastics into high quality oils that could replace fossil fuels (Kalargaris et al. 2017).

Waste plastics are one of the most promising resources for fuel production because of its high heat of combustion and increasing availability in local communities (Syamsiro & Mufrodi 2020). Unlike paper and wood, plastics do not absorb much moisture and the water content of plastics is far lower than the water content of biomass such as crops and kitchen wastes (Miandad et al. 2019). The conversion methods of waste plastics into fuel depend on plastics types. Additionally the effective conversion requires appropriate technologies to be selected according to local economy, environmental, social and technical conditions. In general, the conversion of waste plastic into fuel requires feedstock which is non-hazardous and combustible (Erdogan 2020).

The use of low-density polyethylene for packaging treated water is on the increase in Nigeria and other West African countries. Producers of treated water prefer it, as well as the consumers because it is relatively cheaper (Quesada et al. 2020). Polyethylene films (commonly called pure water sachets in Nigeria) are therefore seen littering the cities, schools, stadium, wedding reception venues etc. During packaging of the water, lots of waste polyethylene is generated (Miandad et al. 2019). The producers of water sachets often burn most of the waste sachets in a pit behind their companies. These polyethylene films are non-biodegradable and can remain on the ground for years. Since the duration of useful life of some plastic products is relatively small, there is a vast plastic waste stream that reaches the final recipients each year, thus, creating serious environmental problem. Again, because disposal of post-consumer plastics is increasingly being constrained by legislation and escalating costs, there is considerable demand for reuse of plastic wastes (Chung et al. 2014; Scarlat et al. 2015).

Nigeria is a developing country with poor technology for waste management leading to indiscriminate disposal of solid waste, mostly plastic waste, most of it being pure water sachet (Adekomaya & Ojo 2016). Very few work have been reported on waste plastic pyrolysis in Nigeria with emphases on commercial catalyst and hence the need to explore local and sustainable technology for waste plastic pyrolysis. Thus, the recovery of useful chemicals from this readily available huge plastic waste, mostly LDPE sachet wastes will ensure environmental safety and serve as a possible source of renewable energy for economic prosperity.

2. Materials and Methods

2.1 Sample collection

The LDPE used as water sachets were collected by hand-picking from waste dumpsites within the University of Uyo, Nwaniba, Uyo, Nigeria. The samples were washed with acetone to de-ink and dried at ambient condition for 12 hours before shredding. The shredded sample was stored in glass beakers. Also, bentonite clay was obtained from clay deposits in Itu Local Government Area of Akwa Ibom State, Nigeria.

2.2 Fabrication of pyrolysis reactor with distillation column and heat exchange

A semi-batch reactor fitted with metallic condenser was fabricated. The reactor body was made of stainless steel with lagging materials made of galvanized steel pan with local insulating materials; example, sisal hemp and fiber glass. The column and condenser were made of galvanized steel pipe. Temperature gauge of 650 °C was fitted to the reactor to measure the pyrolysis temperature and the exit temperature of the liquid products from the condenser column. Figure 1 shows a flow diagram for the batch reactor used in the pyrolysis experiment. The flow diagram of the batch pyrolysis system is presented in Figure 1.

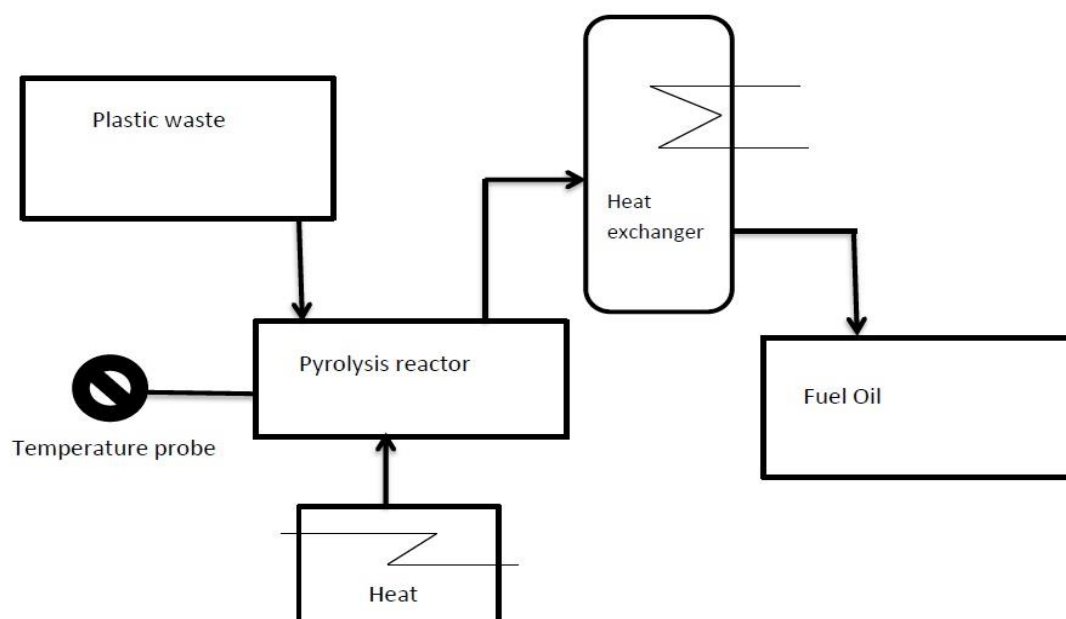


Figure 1: Flow diagram for batch pyrolysis process of LDPE plastic wastes into useful hydrocarbon products

2.3 Synthesis and characterization of mesoporous local clay composite (LCC3)

Mesoporous clay composite from Nigerian bentonite clay was synthesized by hydrothermal process followed by intercalation with ferric oxide precursor to attain the needed surface area and pore diameter for efficient cracking process. Detailed process for catalyst synthesis has been reported elsewhere (Okon et al. 2019).

2.4 Thermogravimetric analysis of LDPE

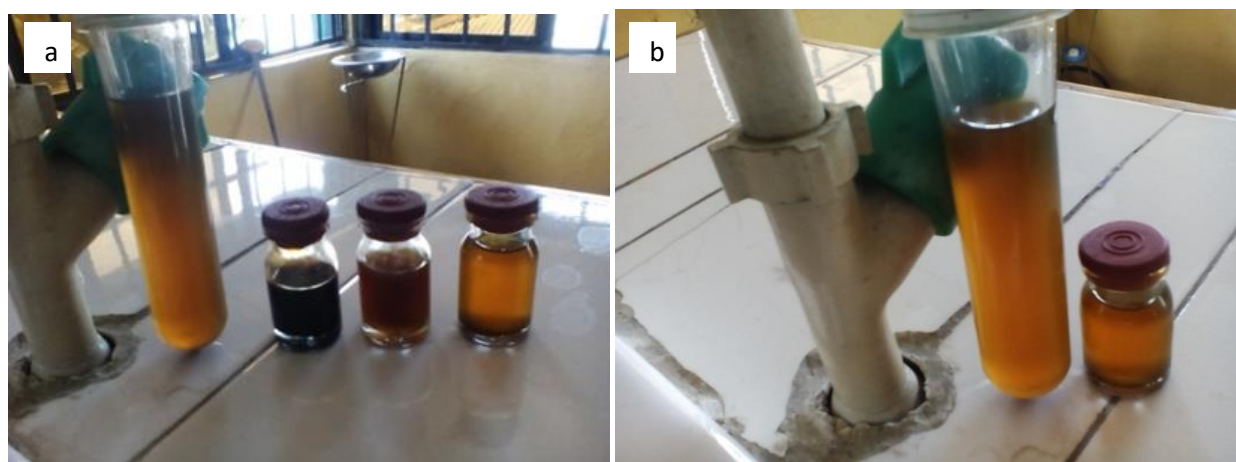
Hundred grams (100 g) of the LDPE waste was placed in the pre-weighed fabricated tubular pyrolysis reactor and pyrolyzed at temperature ranges of 250 °C, 300 °C, 350 °C, 400 °C 450 °C and 500 °C. The weight losses of the plastic waste at different temperatures were recorded according the report by Sarker et al. (2012).

2.5 Non-catalytic pyrolysis of LDPE sachet wastes into useful chemical products

The prepared the LDPE waste sample (100 g) was fed into the batch reactor and closed to prevent escape of gases. The sample was then pyrolyzed at 450 °C for 4 hours and the volatiles were allowed to condense in a heat exchanger unit connected to the reactor. The condensed products were collected in a 100 ml measuring cylinder placed directly under the condenser outlet. The products were stored in glass vials for further analysis.

2.6 Catalytic pyrolysis of LDPE sachet wastes into useful chemicals

The prepared LDPE sachet waste feedstock (100 g) was mixed with 20 g of the mesoporous clay composite and charged into the reactor and pyrolyzed at 450 °C for 3 hours and the volatiles were condensed in a heat exchanger unit connected to the reactor. The condensed products were collected in a 100 ml measuring cylinder placed directly under the condenser outlet. The products were stored in glass vials for further analysis. This was repeated with the mesoporous clay intercalated with ferric oxide according to the method reported by Sarker et al. (2012). Samples of liquid products obtained are presented in Figure 2.



Figures 2: Sample of product of, a: non-catalytic pyrolysis and b: catalytic pyrolysis processes

2.7 Characterization of products

The products from both the non-catalytic and catalytic pyrolysis processes were characterised using Fourier transform infra-red spectroscopy (FTIR) and gas chromatograph with a flame ionisation detector (GC-FID) to identify the functional groups and chemical composition.

FTIR analysis: FTIR analysis of the pyrolysis products obtained at different temperatures were taken on a Perkin-Elmer Fourier transformed infrared spectrophotometer (FTIR) with resolution of 4 cm⁻¹, in the range of 400-4000 cm⁻¹ to know the functional group composition.

GC-FID analysis: Exactly, 1 mL of the products was used for liquid-liquid extraction using n-hexane as solvent. Exactly, 1 ml of the extract was measured into a glass vial and placed in sample holder for auto-injection. The sample was analysed for total petroleum hydrocarbon using GC-FID (GC-FID-QP 2010 Shimadzu) with column temperature: 70 °C; injector temperature: 200 °C; injection mode: split; split ratio: 10; flow control mode: linear

velocity; column flow: 1.51ml/min; carrier gas: helium: (99.9995 % pure); length of column: 30.0 m; diameter: 0.25 mm.

2.8 Determination of physicochemical properties of products

The pyrolytic liquids obtained at the maximum liquid yield condition were characterized for their physical properties. These properties were determined according to the standard of American Society for Testing and Materials (ASTM) test methods. The properties determined were kinematic viscosity, pH, flash point, pour point, higher heating value, colour, density, and fire point.

Kinematic viscosity: Kinematic viscosity of the products was obtained using Oswald viscometer at 30 °C. About 50 mL of oil was sucked into the lower bulb of the viscometer, the oil was then allowed to flow through the marked points on the upper bulb. The time of flow of the pyrolysis product through this point were recorded and multiplied with the instrument conversion factor to obtain the kinematic viscosity. This was according to ASTM D445 test method.

Ash content: The ash content of the derived liquid was determined according to ASTM D482 test method. The liquid sample was burned in an open crucible and the carbonaceous residue was reduced to ash at a high temperature of 732 °C.

Flash point: The flash point of the obtained products was measured using Cleveland open cup tester. About 20 ml of the product was placed in the cup and heated with an electric hot plate. An ignition source was brought close to the cup at one minute interval. The temperature at which the ignition source first ignites vapour from the chemical products was recorded as its flash point. This was according to ASTM D92 test method.

Pour point: About 20 mL of the product sample was poured in a pour point test jar and cooled in an ice bath. The pour point of the product was recorded as the point when the liquid could not flow in respond to gravity. This was according to ASTM D97.

Calorific value: The calorific value of the chemical products was determined using a Parr adiabatic bomb calorimeter. About 20 mL of the product was placed in the cup and electrical energy was used to ignite the fuel; as the fuel is burning, it will heat up the surrounding air, which expands and escapes through a tube that leads the air out of the calorimeter. When the air is escaping through the copper tube it will also heat up the water outside the tube. The change in temperature of the water allows for calculating calorie content of the fuel. This was according to the test method of ASTM D426.

Aniline point: Equal volumes of aniline and the products were stirred continuously in a test tube and heated until the two merge into a homogeneous solution. Heating is stopped and the tube is allowed to cool. The temperature at which the two phases separate out is recorded as aniline point according to the method reported by Okon et al. (2019).

Specific gravity: The specific gravity of the products was measured using specific gravity bottle of 50 ml capacity with capillary hole. The bottle was weighed empty and recorded as W1. Exactly, 10 ml of water was placed in the bottle and the weight was recorded as W2. The water was poured out and the bottles cleaned thoroughly. Exactly 10 ml of the liquid product was placed in the bottle and the weight was recorded as W3. The specific gravity of the liquid product was calculated from the Equation (1), according to the method reported by (Qureshi et al., 2020).

$$\text{Specific gravity} = ([W2-W1])/([W3-W1]) \quad \text{Equation (1)}$$

3. Results and Discussion

3.1 Thermogravimetric analysis of LDPE under non-catalytic and catalytic pyrolysis

The thermogravimetric curves for LDPE pyrolysis under non-catalytic and catalytic processes are presented in Figure 3. The effect of temperature on plastic degradation showed that optimum temperature for degradation for non-catalytic pyrolysis and catalytic process were 500 °C and 490 °C, respectively for non-catalytic and catalytic processes, after which secondary reactions will occurs leading to significant changes in product distribution.

These optimum temperatures agree with those reported by another researcher (Ali & Qureshi 2011). The temperature (T50) where 50 % weight loss occurs were 450 °C and 440 °C for the non-catalytic and catalytic processes respectively. This result shows that the intercalated clay as a catalyst can reduce the pyrolysis temperature and enhance product quality (Ali & Qureshi 2011).

The product distribution (Figures 4) for the non-catalytic and catalytic processes shows increase in liquid fraction vis-à-vis low residue due to secondary cracking on the catalysts active site. However, there was decrease in the amount of gas due to decrease in pyrolysis temperature and short residence time recorded in the catalytic process (Sonawane et al., 2015).

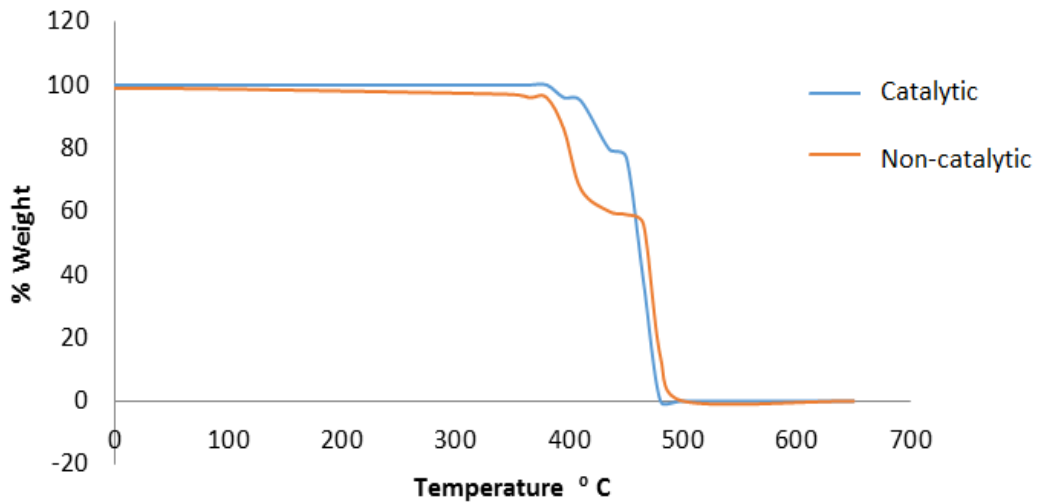


Figure 3: Thermogravimetric plots for catalytic and non-catalytic pyrolysis of LDPE

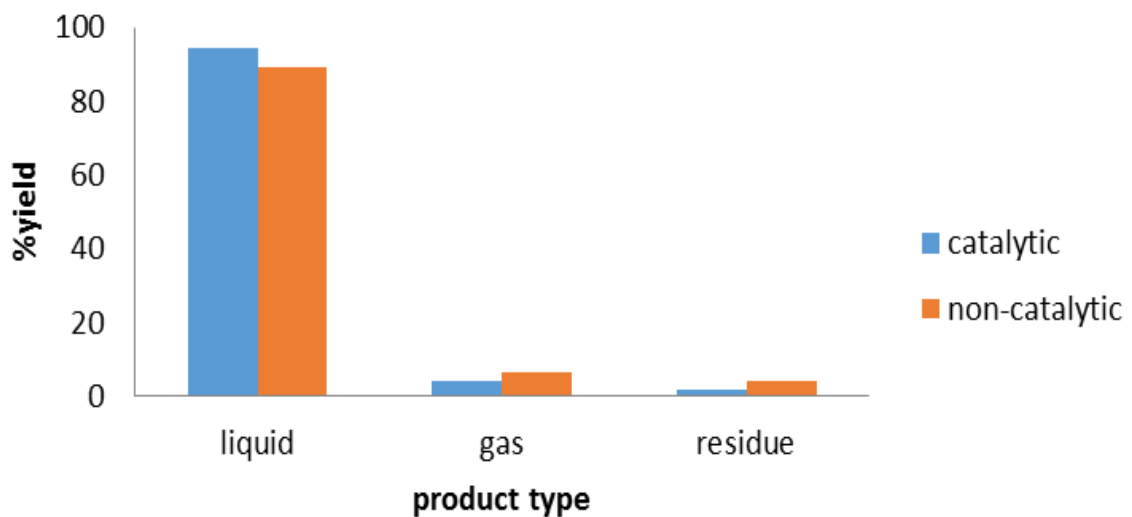


Figure 4: Product distribution for catalytic and non-catalytic pyrolysis of LDPE

3.2 Effect of temperature on product distribution

The degradation products were classified into three groups namely, gaseous, liquid and solid residue. Figure 5 shows the distribution of different fractions produced from non-catalytic pyrolysis process. The amount of liquid products increases with increase in temperature. The recovery of liquid fraction was 2 % at 340 °C, 33 % at 400 °C and 84.4 % at 500 °C. At low temperature, the residence time was higher, because secondary cracking of the pyrolysis product occurred inside the reactor and resulted in a yield of volatile/gaseous product which was not possible at higher temperature due to low residence time of the reaction inside the reactor (Panda et al. 2014).

Figure 6 shows the distribution of different fractions from the catalytic process. The amount of liquid product (condensable fraction) increases and residence time decreases due to the presence of the LCC3 at all temperature range. From the result presented in Figure 6, the optimum yield for liquid product was 89.5% for the catalytic product which was 5.1% higher that the yield of non- catalytic process (84.4 %). this was due to the catalytic effect of the catalyst which was expected to possess acidic sites and metallic site which facilitate isomerization and cracking process at each site, respectively (Sharuddin et al. 2016).

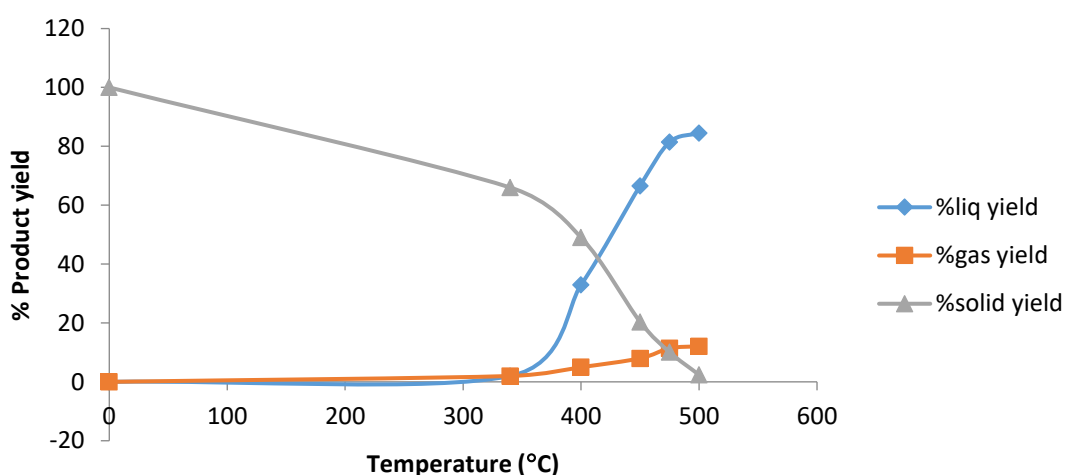


Figure 5: Product yield in percentage for non-catalytic pyrolysis of LDPE at different temperatures

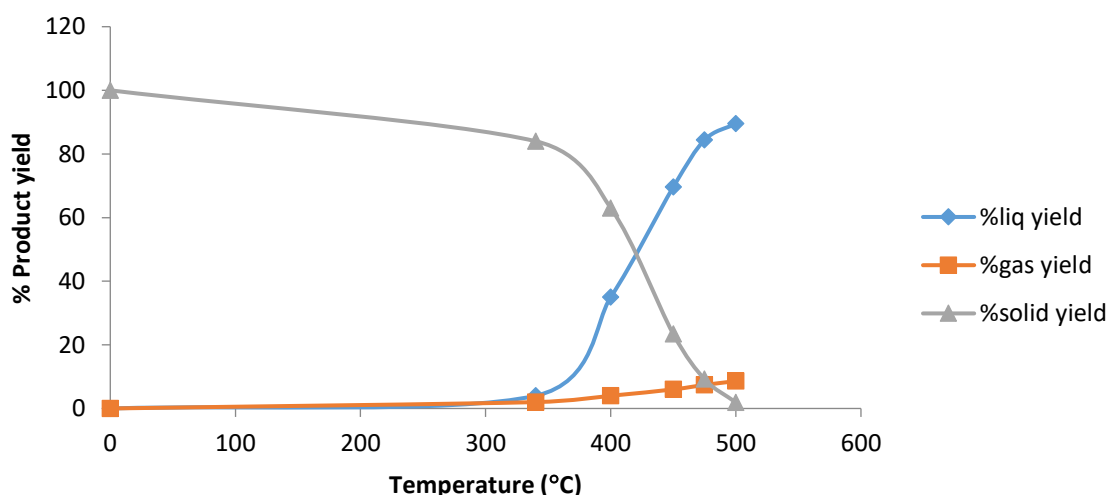


Figure 6: Product yield in percentage for catalytic pyrolysis of LDPE under different temperatures

3.3 Characteristics of liquid hydrocarbon products

Two liquid products were obtained from both non-catalytic and catalytic processes and were characterized using FTIR and GC-FID for functional group composition and hydrocarbon profile as well as physicochemical properties respectively.

Functional group profile of products: The FTIR spectra of the liquid products obtained from the non-catalytic and catalytic pyrolysis process of LDPE at 500 °C is presented in the supplementary data section (Fig S2 and S3). The important assigned peaks are summarized in Table 1 and 2 with reference to Sigma Aldrich IR tables and charts. The result indicates the presence of different hydrocarbons in the products in association with other organic molecules. From the FTIR spectra of the oils, it was observed that there was variation in the composition of the products in non-catalytic and catalytic processes. There was medium peak intensity for mono-substituted aromatics in the catalytic process showing higher amount of this component in the product as against its relative low amount in the non-catalytic process with weak peak in that region. The presence of strong alkenyl stretch signal at 1648 cm⁻¹ which was absent in the non-catalytic pyrolyzed product also indicates that isomerization occurred on the surface of the catalyst during the catalytic pyrolysis process. The weak peaks appearing at 3746-3671 cm⁻¹ in the spectra of the non-catalytic product are characteristic peaks for organic silicones and were absence in spectra for the product of catalytic pyrolysis (Kyaw & Hmwe 2015).

Table 1: Principal assigned peaks from FTIR spectra of products obtained from non-catalytic pyrolysis of LDPE at 500°C

S/N	Wave number (cm ⁻¹)	Functional group assignments
1	3746-3671	Si-O-R stretch (from organic silicones)
2	3078	Aromatic C-H stretch
3	2922-2855	Methylene C-H asym/sym stretch
4	1828	Carbonyl group (from a five membered ring) anhydride
5	1643	C=C (conjugated)
6	1461	Methylene C-H bend
7	1379	Methylene C-H asym/sym bend
8	1304	Vinylidene C-H (in plane bend)
9	1077	Organic siloxane and silicone (Si-O-C)
10	965	Trans C-H out of plane bend
11	909	Vinyl C-H out of plane bend
12	723	Mono-substituted Aromatics (Phenyl)

Table 2: Principal assigned peaks from FTIR spectra of products obtained from non-catalytic pyrolysis of LDPE at 500 °C using LCC3

S/N	Wave number (cm ⁻¹)	Functional group assignments
1	3078	Aromatic C-H stretch
2	2922-2855	Methylene C-H asym/sym stretch
3	1828	Carbonyl group (from a five membered ring) anhydride
4	1648	Alkenyl C=C stretch
5	1461	Methylene C-H bend
6	1379	Methylene C-H asym/sym bend
7	1304	Vinylidene C-H (in plane bend)
8	965	Trans C-H out of plane bend
9	909	Vinyl C-H out of plane bend
10	723	Mono-substituted aromatics (phenyl)

GC-FID analysis of liquid products: The detected hydrocarbons present in the liquid products are summarized in Tables 3 and 4 for the non-catalytic and catalytic pyrolyzed products, respectively. The products contained mostly alkanes, alkenes and a few aromatics with carbon number range of C₁₀-C₂₃ and C₁₀-C₂₁ for non-catalytic and catalytic products, respectively. The major difference between the product of the non-catalytic and catalytic process are the isomerization of saturated hydrocarbons (mainly decane, dodecane, tridecane, hexadecane and docosane) into aromatics (mainly naphthalene, acenaphthene, fluorene, pyrene and picene). This product isomerization resulted to changes in the properties and application of the product in as energy fuel and as starting material for industries. Product isomerization in catalytic pyrolysis process has been reported by other researchers (Sharuddin et al. 2016; Kyaw & Hmwe 2015).

Table 3: Composition of liquid product obtained from non-catalytic pyrolysis of LDPE at 500 °C by GC-FID

Peak	Retention time (min)	Area %	Name of composition	Molecular Formula
1	4.313	2.93	Decane	C ₁₀ H ₂₂
2	4.967	3.78	Undecane	C ₁₁ H ₂₄
3	5.313	5.02	Dodecane	C ₁₂ H ₂₆
4	6.118	6.24	Tridecane	C ₁₃ H ₂₈
5	6.666	8.24	Tetradecane	C ₁₄ H ₃₀
6	7.180	8.57	Pentadecane	C ₁₅ H ₃₂
7	7.663	8.30	Hexadecane	C ₁₆ H ₃₄
8	8.096	2.62	Heptadecane	C ₁₇ H ₃₆
9	8.123	8.00	Benzo(a)fluorene	C ₁₂ H ₁₂
10	8.560	9.75	Octadecane	C ₁₈ H ₃₈
11	8.589	5.76	Chrysene	C ₁₈ H ₁₂
12	8.974	7.73	Nonadecane	C ₁₉ H ₄₀
13	9.368	6.42	Eicosane	C ₂₀ H ₄₂
14	9.743	5.56	Heneicosane	C ₂₁ H ₄₄
15	10.104	4.31	Docosane	C ₂₂ H ₄₆
16	10.481	6.18	Tricosane	C ₂₃ H ₄₈
Total		100		

Table 4: Composition of liquid product obtained from catalytic pyrolysis of LDPE at 500 °C using LCC3 by GC-FID

Peak	Retention time (min)	Area %	Name of composition	Molecular Formula
1	4.314	3.81	Naphthalene	C ₁₀ H ₈
2	4.965	4.68	Undecane	C ₁₁ H ₂₄
3	5.576	4.82	Acenaphthene	C ₁₂ H ₁₀
4	6.107	7.96	Flourene	C ₁₃ H ₁₀
5	6.652	9.92	Tetradecane	C ₁₄ H ₃₀
6	7.165	9.97	Pentadecane	C ₁₅ H ₃₂
7	7.648	8.41	Pyrene	C ₁₆ H ₁₀
8	8.107	8.71	Heptadecane	C ₁₇ H ₃₆
9	8.137	6.98	Benzo(a) fluorene	C ₁₈ H ₃₆
10	8.543	7.42	Octadecane	C ₁₈ H ₃₈
11	8.571	5.69	Chrysene	C ₁₈ H ₁₂
12	8.958	6.21	Nonadecane	C ₁₉ H ₄₀
13	9.352	6.65	Eicosane	C ₂₀ H ₄₂
14	9.729	6.92	Heneicosane	C ₂₁ H ₄₄
15	10.090	4.45	Picene	C ₂₂ H ₁₄
Total		100		

3.4 Physicochemical properties of liquid products

The results for the fuel properties of the product from non-catalytic and catalytic pyrolysis of LDPE at 500 °C are presented in Table 5. The boiling range of the non-catalysed fuel is 74 °C – 401 °C while that of the catalytic pyrolysis product is 71 °C - 385 °C. The specific gravity, viscosity, aniline point, calorific value, flash point, fire point, cloud point, pour point and boiling points for both non-catalytic and catalytic products are given in Table 5. The specific gravity for both non-catalytic and catalytic pyrolysis products were 0.77 and 0.78, respectively, which is similar to that of kerosene fuel. Viscosities for both liquid products were 2.18 and 2.26 respectively and were close in value to that of kerosene which is 2.20 m²/s.

Flash point and fire points for both non-catalytic and catalytic fuels vary from 47.5 °C – 49.7 °C and 49 °C- 52 °C respectively and were higher than the value for gasoline but lower than those of kerosene and diesel. The values for pour point for the non-catalytic and catalytic fuels – 11 °C to -14 °C respectively and were higher than that of kerosene. Values for cloud point for both catalytic and non-catalytic products were 14 °C and 22 °C respectively. Cloud point was not detected for the commercial products. The boiling range of the non-catalysed fuel is 74 °C – 401 °C while that of the catalytic pyrolysis process is 71 °C - 385 °C. Both oil from their boiling points infer the presence of mixtures of different fuel components namely gasoline, kerosene, diesel in the products. The values of aniline point were found to increase for product of catalytic process compared to the non-catalytic process. Again, these values were in agreement with those of kerosene and diesel fuel as reported in Table 5. There was decrease in their flash point and fire points due to increase aromaticity in the liquid product from catalytic pyrolysis process using LCC3. The flash point values indicate that the catalytic oil obtained using LCC3 catalyst resembles kerosene while non-catalytic oil has its flash point closer to the diesel range fuel. There was a little increase in net calorific value for the catalytic pyrolysis product (39.22 MJ/Kg) over that of non-catalytic pyrolysis product (39.15 MJ/Kg). These calorific values were also found to correspond to that of diesel fuel (Kyaw & Hmwe 2015; Saptoadi & Pratama 2015; Usman et al. 2014). From the result, it is concluded that the fuel properties of the non-catalytic and catalytic liquid products resemble the properties of commercial fuels. The liquid product obtained can be distilled into different fraction for used as fuels after proper quality assurance.

Table 5: Fuel properties of liquid products obtained and compared with commercial products in the market

Fuel properties	Non-catalytic	Catalytic	Gasoline	Kerosene	Diesel
Colour	Colourless	Golden yellow			
Viscosity(m ² /s)	2.18	2.26	1.04	2.2	3.7-5.0
Calorific value (MJ/Kg)	39.15	39.22	42.8	43.5	41-42.9
Ash content(g)	0.002	0.00	0.00	0.002	0.003
Flash point(°C)	49.7	475	37.8 – 38	50-55	55-60
Cloud point(°C)	14	22	-	-	-
Fire point (°C)	52	49	-	32-72	32-78
Pour point(°C)	-11	-18	-	-47	
Aniline point	54	69	65	62	71
Specific gravity	0.77	0.78	0.70	0.78	0.85
Initial BP(°C)	74	71	49	110	120
Final BP(°C)	329	314	112	220	305

4. Conclusion

The present study revealed that local clay and its modified composites used in this study are efficient catalysts for degrading LDPE into liquid hydrocarbons and has the potential to reduce plastic waste burden in the environment. Spectroscopic characterization of the clays reveals important changes in their texture and functionality which enhance their catalytic efficiency in the plastic degradation process. Generally, products yields increased with application of the catalyst with decrease in pyrolysis temperature. Product distribution also showed changes in hydrocarbon profile with less number of carbon atoms in the catalytic pyrolysis product stream and increased unsaturated and mono-substituted aromatic components. This makes the catalytic

pyrolysis product of higher calorific value and shows physicochemical properties in the gasoline fuel range and other commercial fuels such as kerosene and diesel.

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