CONVERSION OF WASTE POLYETHYLENE TEREPHTHALATE (PET) INTO HYDROCARBONS BY PYROLYSIS USING LOCAL MATERIALS AS CATALYSTS

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Abstract

Polyethylene terephthalate (PET) have become a general use for various applications such as storage for food and beverages because of its excellent tensile and impact strength, chemical resistance, and thermal stability. However, due to its non-degradability, its waste has continued to generate great concern globally. The recycling of PETs has become a good option for the clean-up of waste PETs in the environment. To this effect, this research aimed at converting waste PETs by pyrolysis into hydrocarbons using local materials as catalyst. Varying ratio of plantain peel and kaolin clay, corn shaft and kaolin and banana peel and kaolin were the local materials used. Agilent 7820A Gas chromatograph attached to a 5977E Mass Spectrometer was used for the analysis. The results indicated that hydrocarbons such as biphenyl, benzonitrile, benzaldehyde, styrene, phenol, benzene, ethylbenzene and naphthalene were among the prominent compounds detected. The results were similar to other research works of PETs pyrolysis using inorganic materials as catalysts hence, the local materials proved to be equally efficient catalysts for PET pyrolysis.

Keywords: Pyrolysis, Local materials, Hydrocarbons, waste PETS, Catalyst

1.0 Introduction

Polyethylene terephthalate (PET), commonly referred to as 'polyester' in the textile industry, is considered to be one of the most important thermoplastic polyesters (Incornato, 2000). It is produced by the polymerization of ethylene glycol and terephthalic acid which are derived from crude oil. It is widely used for various applications such as bottles, fibres, moldings, and sheets because of its excellent tensile and impact strength, clarity, process ability, chemical resistance, and thermal stability (Pawlak, 2000; Gupta and Bashir, 2002). Unlike natural polymers, it is a non-degradable polymer in the natural environment (Edge, 1996) leading to environmental pollution when it is discarded after use. The increasing consumption and non-biodegradability of PET plastics used for packaging of food and drinks has created serious environmental and economic concerns hence, management of plastic waste has become an important social issue (Bartolome, 2012). Landfill of PET is undesirable because of space limitations and ground pollution. Incineration is also not the preferred option, because of the inevitable emission of toxic gases that are generated from the decomposition of PET molecular chains and residual additives, although incineration produces energy. Recycling and energy recovery methods have been developed as part of plastic waste management. Chemical recycling of PET by pyrolysis using inorganic materials (both synthetic and local) as catalysts for energy recovery have been effective in the conversion of waste PET to useful product (Ogunyemi *et al*., 2019). In view of these, this research aimed at converting waste PET by pyrolysis into useful hydrocarbons using local materials as catalyslts.

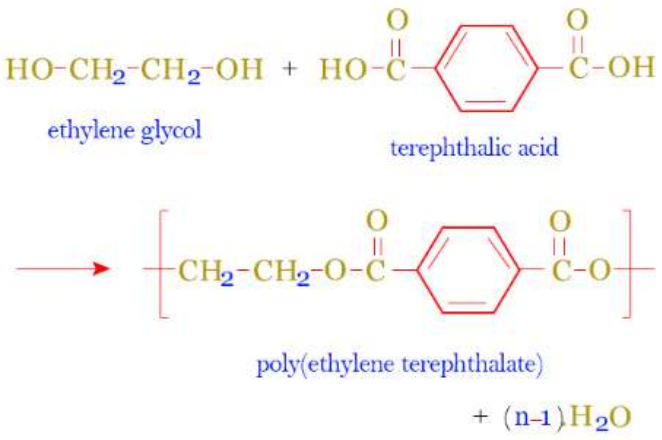

Fig. 1: Structure of PET

2.0 Materials and Methods

2.1 Sampling

2.1.1 PET Bottles

The post-consumer PET bottles were sourced around Yaba College of Technology environment. The caps and label on the bottles were removed (with hand), washed and sun-dried to remove any trace of moisture. The quantity collected was then shredded to form pellets (of non-uniform sizes) with scissors.

2.1.2 The kaolin clay

The kaolin clay (local chalk) was purchased from an herbal seller at Oyingbo market Lagos State. The whole chalk was crushed and ground to powdery form using pestle and mortar and stored in a clean air tight container.

2.1.3 Plantain Peels

The plantain peels were sourced for and gathered from the sellers within Sabo and Oyingbo markets, Lagos. The peels were washed with distilled water, placed on a clean and dry surface and left to sun-dry for about three weeks before grinding to powdery form. They were then stored a clean foil prior to use.

2.1.4 Corn Shaft

Corn shafts was collected from the local pap sellers and sun dried for a period of two weeks till it was completely free of moist and then stored in a clean air tight container.

2.1.5 Banana Peels

Banana peels were obtained locally within Sabo and Oyingbo markets, Lagos from banana sellers and those that produce banana flour. The peels were washed with distilled water, drained and sliced into small pieces, it was then sun dried for about three weeks. After that, the peels were then grounded into powdery form using a blender and then stored into a clean foil.

2.2 Method

The production method for the conversion of plastics to liquid fuel was based on the pyrolysis of the plastics according to Sarker et al., 2011 and Moinuddin and Mohammed, 2011.

2.2.1 Sample A: PET (pellets) and plantain peels (10g:40g)

The total weight of the PET (pellets) and Plantain peel was 50g. 40 g of the dried plantain peel was divided into two. 20 g of the dried plantain was introduced into the 2 necked round bottom flask. The waste PET pellets (10 g) was added into the flask and the remaining half (20 g) of the dried plantain peel was then introduced to cover the sample (PET) so as to prevent having the PET samples sticking to the flask upon heating and also to ensure that the PET samples have proper contact with the plantain peels when pyrolysis starts. A mercury in glass thermometer was passed through one neck to measure the rate of temperature during the heating process while the other was connected to a lie-big condenser (which condenses all the vapourised gases into liquid fuel) using a T-shaped connector. The flask was then placed in the heating mantle and allowed to heat until the PET was completely degraded at the same time turning on the stop clock. The vapour travelled through the condenser pipe and was collected in the collection flask until the end of the experiment. The whole content was heated for 32 minutes at a temperature range of 150ºC.

2.2.2 Sample B: PET (pellets), Kaolin clay and plantain peels (20g:20g:20g)

The same procedure as for sample A except that 20 g of PET (Pellet), 20 g of Kaolin clay and 20 g plantain peels totaling 60 g were used. The Kaolin and plantain peel were mixed thoroughly and divided into two and the above procedure was used. The whole content was heated for 32 minutes at a temperature range of 150ºC.

2.2.3 Sample C: PET (pellets), Kaolin clay and plantain peels (40g:20g:40g)

The same procedure as B was carried out. The whole content was heated for 42 minutes at a temperature of 280 ^{0}C.

2.2.4 Sample D: PET (pellets), Kaolin clay and plantain peels (40g:30g:40g)

With the same procedure as for sample B, the heating flask and its content were heated to 160 ºC and the process after 45 minutes.

2.2.5 Sample E: PET (pellets), Kaolin clay and plantain peels (40g:40g:40g)

Following the same procedure as sample B, the flask was heated at the room temperature of 30ºC to 170ºC and the whole process was completed in 48 minutes.

2.2.6 Sample F: PET (pellets), Kaolin clay and plantain peels (40g:50g:50g)

The same procedure as for sample B was also employed. The flask was heated from 30ºC to 185ºC and the total time for the process was 55 minutes.

2.2.7 Sample G: PET (pellets), Kaolin clay and corn shaft (10g:30g:30g)

Thirty grams (30 g) of corn shaft and 30 g of kaolin clay was thoroughly mixed and then divided into equal halves. The first half (30 g) was introduced into the 2 necked round bottom flask. Waste PET pellets (10 g) was added into the flask and the remaining half (30 g) of the mixed corn shaft and kaolin clay was then introduced to cover the sample (PET).

The procedure continued as with other samples above. The flask was heated at the room temperature of 30ºC to 270ºC and the whole process was completed in 30.44 minutes.

2.2.8 Sample H: PET (pellets), Kaolin clay and corn shaft (20g:40g:40g)

Following the same procedure as sample G, the flask was heated at the room temperature of 30ºC to 190ºC and the whole process was completed in 26.50 minutes.

2.2.9 Sample I: PET (pellets), Kaolin clay and corn shaft (30g:40g:40g)

Following the same procedure as sample G, the flask was heated at the room temperature of 30ºC to 200ºC and the whole process was completed in 52.12 minutes.

2.2.10 Sample J: PET (pellets), Kaolin clay and corn shaft (40g:40g:40g)

Following the same procedure as sample G, the flask was heated at the room temperature of 30ºC to 270ºC and the whole process was completed in 58.57 minutes.

2.2.11 Sample K: PET (pellets), Kaolin clay and banana(20g:30g:30g)

The procedure used for the above was used except that banana was used in place of corn shaft. the flask was heated at the room temperature of 30ºC to 165 ^{0}C and the whole process was completed in 45.58 minutes.

2.2.12 Sample L: PET (pellets), Kaolin clay and banana (30g:30g:30g)

The procedure used for sample K was used. The flask was heated at the room temperature of 30 ^{0}C to 290 ^{0}C and the whole process was completed in 48.18 minutes.

2.3 Extraction / Clean-up of samples

Since the product of the pyrolysis was both aqueous and organic, the extraction method employed in this study was liquid-liquid extraction (LLE) according to EPA method 3510 with some modification. Hexane (35 mL) and 15ml of dichloromethane were added to the sample in a separatory funnel and the mixture was shaken gently at first and then vigorously with intermittent venting. Aqueous layer of the mixture was then released into a beaker while the organic layer was re-extracted twice and the organic extracts combined. The combined extract was concentrated in a rotary evaporator and transferred to glass columns containing 5 g silica gel and 2 g anhydrous Na_2SO_4 for cleaning up of the samples. The resulting eluate was further concentrated to about 1 mL and transferred 1 mL vial for GC – MS analysis.

2.4 Instrumental analysis

Qualitative analysis of the samples obtained from the pyrolysis was performed using Agilent 7820A Gas chromatograph attached to a 5977E Mass Spectrometer. The injection temperature was 240ºC. The time of the analysis was about 70 minutes. The analysis was done to investigate possible hydrocarbons after purification of the samples and the results obtained from each sample were interpreted based on the chromatogram compared with the library.

3.0 Results and Discussion

3.1 PET, Plantain peel and Kaolin

The summary of the yield of the pyrolysis of PET, Plantain, corn shaft, banana and Kaolin clay is as shown in Table 1. From the table below, it can be seen that the quantity of the local materials, the more the yield, and the longer the time it took for the pyrolysis with varying temperature which is an indication that the local materials play a major role in the thermal degradation of PET. The optimum yield for PET, plantain and Kaolin was ratio 4:4:3 which implies that Kaolin clay is important and the ratio of the plantain peel to kaolin should be more than half but not equal to one. The yield for corn:PET:Kaolin clay (1:1:1) also gave higher yield for same ratio 1:1:1 of combination of banana and plantain with kaolin clay.

Table 1: Showing the summary of the Experiment Data

Samples		Mass	Mass of PET (g)	Mass of Kaolin (g)	Ratio of products	Initial temperature (T_1°C)	Final temperature (T_2°C)	Time taken (min)	Volume of product (ml)
Plantain peel (g)	A	40	10	-	4:1	30	190	35	15
Plantain peel (g)	B	20	20	20	1:1:1	30	150	32	8
Plantain peel (g)	C	40	40	20	2:2:1	30	280	42	15
Plantain peel (g)	D	40	40	30	4:4:3	30	160	45	22
Plantain peel (g)	E	40	40	40	1:1:1	30	170	48	18
Plantain peel (g)	F	50	40	50	5:4:5	30	185	55	22
Corn shaft (g)	G	30	10	30	3:3:1	30	270	31	16
Corn shaft (g)	H	40	20	40	2:2:1	30	190	27	21
Corn shaft (g)	I	40	30	40	4:3:4	30	200	52	24

Corn shaft (g)	J	40	40	40	1:1:1	30	270	59	25
Banana peels(g)	K	30	30	30	1:1:1	30	290	48	9
Banana peels(g)	L	30	20	30	3:2:3	30	165	46	10

Table two below shows the list of products obtained from Sample A. From the result below, n-Hexadecanoic acid had the highest percentage area of 3.19%; Benzonitrile had 1.55%; Biphenyl had 1.50%; n-Propylbenzoate had 0.44%; Ethyl 4-acetylbenzoate; Diphenylmethane had 0.86%; 2-Furanmethanol had 0.75%; Ethanone had 0.68%; 1,2-Benzenecarboxylicacid had 0.55%; Phenol had 0.49% amongst others, all at different retention times.

Table 2: Compounds detected for sample A (PET + Plantain peel = 50g)

PEAK NUMBER	RETENTION TIME (min)	AREA%	COMPOUND NAME
1	3.136	0.01	Toluene
7	3.455	0.01	1,5-Naphthalene
8	3.549	0.03	Cyclopentadienyl
25	4.548	0.02	Benzenesulfonic
27	4.606	0.07	2-Cyclopenten-1-yl
29	4.686	0.03	2- Cyclopenten-1-one
35	5.323	0.75	2-Furanmethanol
37	5.52	0.12	Benzene
38	5.585	0.03	p-Xylene
50	6.678	0.03	Hexabutyl
51	6.739	0.04	2,4-Hexadiene
53	6.891	0.09	3,4-Octadiene
57	7.506	1.55	Benzonitrile
58	7.598	0.49	Phenol
59	7.671	0.1	Cyclohexene

70	8.443	0.1	1,2,3,5,8-Hexahydronaphnthalene
78	9.269	0.24	1-Phenyl-1-butene
84	9.747	0.38	1-Propanone
88	9.969	0.68	Ethanone
106	11.015	0.33	Ethyl 4-methylbenzoate
107	11.05	0.22	Tridecane
108	11.073	0.35	Naphthalene
118	11.664	0.22	Benzeneacetamide
121	11.88	1.5	Biphenyl
125	12.033	0.27	4-Ethylbenzoic acid
130	12.267	0.99	N-propyl benzoate
131	12.331	0.81	Diphenylmethane
134	12.471	0.37	Eugenol
139	12.824	0.55	Pentadecene
141	12.898	0.29	1,4-Benzenecarboxylic acid
150	13.321	0.86	Ethyl 4-acetylbenzoate
157	13.715	0.27	4,4-Dimethylbiphenyl
160	13.846	0.33	1,4-Epoxynaphthalene
165	14.171	0.55	1.2-Benzenedicarboxylic acid
169	14.379	0.41	Heptadecane
198	15.785	0.29	Nonadecane
208	16.421	3.19	n-Hexadecanoic acid
248	18.811	0.52	9-Octadacanamide

Table 3 showed the lowest number of products obtained from sample B. From the result, Hexabutyl had the lowest area percentage of 0.3%, while Phenol had the highest composition of 1.70%; 3,4-Dichlorophenyl had 1.63%; Acetophenone had 1.12%; Dimethylphosphine had 1.04%; Ferrocene had 0.96%; 2-Methylphenyl had 0.87%; 2-propenoic acid had 0.76%; Hexadiene had 0.42%; Benzenamine had 0.36%; Hexa-2,4-diene had 0,31%; Biphenyl had 0.24%; Benzene had 0.15%.

Table 3: Compounds detected for sample B (PET + Kaolin + Plantain peel = 60g)

PEAK NUMBER	RETENTION TIME (min)	AREA%	COMPOUND NAME
2	3.302	0.3	Hexabutyl
4	3.388	0.36	Benzenamine
8	3.579	1.12	1,1-Dimethylpropyl, Acetophenone, 2-Naphthalenecarboximide
11	3.681	1.63	3,4-Dichlorophenyl
12	3.74	0.87	2-Methylphenyl
15	3.884	0.96	Ferrocene
26	4.515	0.42	Diethyl-3-methyl, Hexadiene
27	4.557	0.15	Benzene
34	5.143	0.31	Hexa-2,4-diene
39	5.367	1.7	Phenol
51	6.425	1.04	Dimethylphosphine
55	6.717	0.76	2-Propenoic acid
111	11.827	0.24	Biphenyl

The products obtained from sample C is as shown in Table 4 with Biphenyl having the highest area percentage of 2.32%; Ethanone had; 2.02%; 1-Octadecene had 1.83%; Ferrocene had 1.30%; Methylester had 1.23%; p-Terphenyl had 1.03%; Benzonitrile had 0.68%; Hexadecane had 0.66%; amongst others. Cyclopropane and Tetradecane had the same area percentage but varying retention time (with Cyclopropane having the greater retention time of 13.467 minutes to Tetradecane).

Table 4: Compounds detected for sample C (PET+ Kaolin+ Plantain peel =100g)

PEAK NUMBER	RETENTION TIME	AREA%	COMPOUND NAME
2	3.2	0.01	Toluene
12	4.144	0.01	Benzoic acid, Cyclopropyl
25	4.879	0.01	Benzhydrazide
38	5.501	0.01	Benzene
39	5.582	0.05	2,4-Methylphenyl
71	7.651	0.68	Benzonitrile
77	8.184	0.24	Methanol, oxo-benzoate
108	10.086	2.32	Biphenyl
120	10.945	0.29	Tetradecane
133	11.838	1.23	Methyl ester
134	11.899	0.32	4-Ethylbenzoic acid
142	12.45	0.5	Heptacosyl acetate, Cyclopentane
143	12.555	0.41	1,3-Hexadione
146	12.705	0.28	Phenol, Benzamide
147	12.743	0.31	Naphthalene
150	12.987	0.47	Toluic acid
152	13.138	0.24	Fluorene
157	13.399	0.66	Hexadecane
158	13.467	0.29	Cyclopropane

165	13.855	0.2	Diethylphthalate
183	15.077	2.02	Ethanone
200	17.127	1.83	1-Octadecene
207	17.823	1.05	p-Terphenyl, m-Terphenyl
212	18.34	1.3	Nonadecane, Tricosane

For sample D, some of the compounds detected include; Benzonitrile with percentage area 2.09%; Diethylphthalate had 1.08%; Benzoic acid had 0.64%; Flourene had 0.56%; Tricosane, Octosane and Nonadecane had 0.55%; Ethylbenzene and p-Xylene had 0.38%; Acetophenone had 0.25%; Hydrazine had 0.23%. Cyclopropane and 2-Pentene had the lowest composition of 0.04% (Table 5).

Table 5: sample D (PET+ Kaolin+ Plantain peel=110g)

PEAK NUMBER	RETENTION NUMBER(min)	AREA%	COMPOUND NAME
1	3.178	0.2	Toluene
7	3.812	0.02	2-Pentene, Cyclopropane
24	4.834	0.04	2-Cyclopenten-1-one
25	4.944	0.04	2,5-Diemthylfuran
28	5.416	0.38	Ethylbenzene, p-Xylene
34	6.411	0.2	Cyclobutene
35	6.486	0.11	Cyclopentane
41	7.315	0.25	Acetophenone
47	7.805	2.09	Benzonitrile
70	9.737	0.64	Benzoic acid

78	10.436	0.23	Hydrazine
130	13.841	0.56	Fluorene
137	14.674	1.48	1,4-Benzenedicarboxylic acid
145	15.559	0.12	Ethanone-1,1,1-biphenyl
153	16.233	1.08	Diethylphthalate
171	17.904	0.33	p-Terphenyl
176	18.435	0.55	Tricosane, Ocatosane, Nonadecane

Table six shows the list of products obtained from sample E. From the result, Benzonitrile had the highest area percentage of 1.81 %; Fluorene had 1.79 %; Naphthalene had 1.52 %; Diethylphthalate had 1.37; p-Toluric acid had 0.86 %; Ethanone had 0.82%; Phenanthrene had 0.78 %; Hydrazine had 0.71 %; Triphenylmethane had 0.66 %; Benzene had 0.56 %; Toluene had 0.54 %.

Table 6: Compounds detected for sample E (PET+ Kaolin+ Plantain peel = 120g)

PEAK NUMBER	RETENTION TIME (min)	AREA%	COMPOUND NAME
1	3.22	0.54	Toluene
19	4.66	0.02	2-Cyclopenten-1-one
20	4.71	0.04	2,5-Dimethylfuran
26	5.31	0.14	Ethylbenzene
27	5.41	0.56	Benzene
28	5.54	0.37	p-Xylene
32	6.229	0.04	2,4-Dimethylfuran
35	6.422	0.1	Cyclopentane
36	6.477	0.09	Cyclopropane
37	6.55	0.09	Cyclohexene

42	7.133	0.28	Benzaldehyde
46	7.712	1.81	Benzonitrile
50	8.387	0.37	Indene
57	9.057	0.29	Benzoic acid
65	10.024	1.52	Naphthalene
107	13.848	1.79	Fluorene
110	14.317	1.37	Diethylphthalate
118	15.139	0.71	Hydrazine, Ethanol
119	15.302	0.78	Phenanthrene
122	15.55	0.82	Ethanone,-1,1,1-bipheny-4-yl
132	16.875	0.24	2-Propenoic acid
144	18.429	0.86	p-Toluric acid
146	18.701	0.66	Triphenylmethane

Analysis of sample F showed that, Hexadiene had the highest compostion of 0.67%; Phenol had 0.59 %; Ferrocene had 0.45%; Biphenyl and Naphthalene had 0.42 %; Cyclodecasiloxane had 0.33 %; Hexabutyl had 0.17 %; Phosphine had 0.16 %; 1,5-Diaminonaphthalene had 0.15 %; Di-2-naphthalenyl had 0.12 % and 1,4-naphthalene had the lowest composition of 0.2 % (Table 7).

Table 7: Compounds detected for sample F (PET+ Kaolin+ Plantain peel=140g)

PEAK NUMBER	**RETENTION TIME**	**AREA%**	**COMPOUND NAME**
1	3.206	0.17	Hexabutyl
4	5.503	0.16	Phosphine
9	3.69	0.12	Di-2-naphthalenyl

13	3.882	0.59	Phenol
15	4.056	0.2	1,4-naphthalene
18	4.437	0.67	Hexadiene, Triphenylphosphanalydiene
22	4.586	0.45	Ferrocene
49	6.791	0.33	Cyclodecasiloxane
55	7.072	0.15	1,5-Diaminonaphthalene
56	7.097	0.45	Ethanolamine
78	10.571	0.42	Biphenyl, Naphthalene

Comparing Tables, A, B, C, D, E and F, toluene, benzene, phenol, biphenyl, benzonitrile, benzoic acid, nonadecane, naphthalene, diethylphtalate were the compounds that occurred most amongst the components listed in the tables. For samples A, C, D and E benzonitrile had the highest area percentages of 1.55, 0.68, 2.09 and 1.81 % respectively. Biphenyl had the highest area percentage of 2.32 for sample C. Naphthalene had the highest area percentage of 1.52 % for sample E, nonadecane had the highest area percentage of 1.3 % for sample C while phenol had the highest compostion of 1.7 % for sample B. The hydrocarbons listed were present in almost every minute amount most especially benzene and benzonitrile (Figure 2).

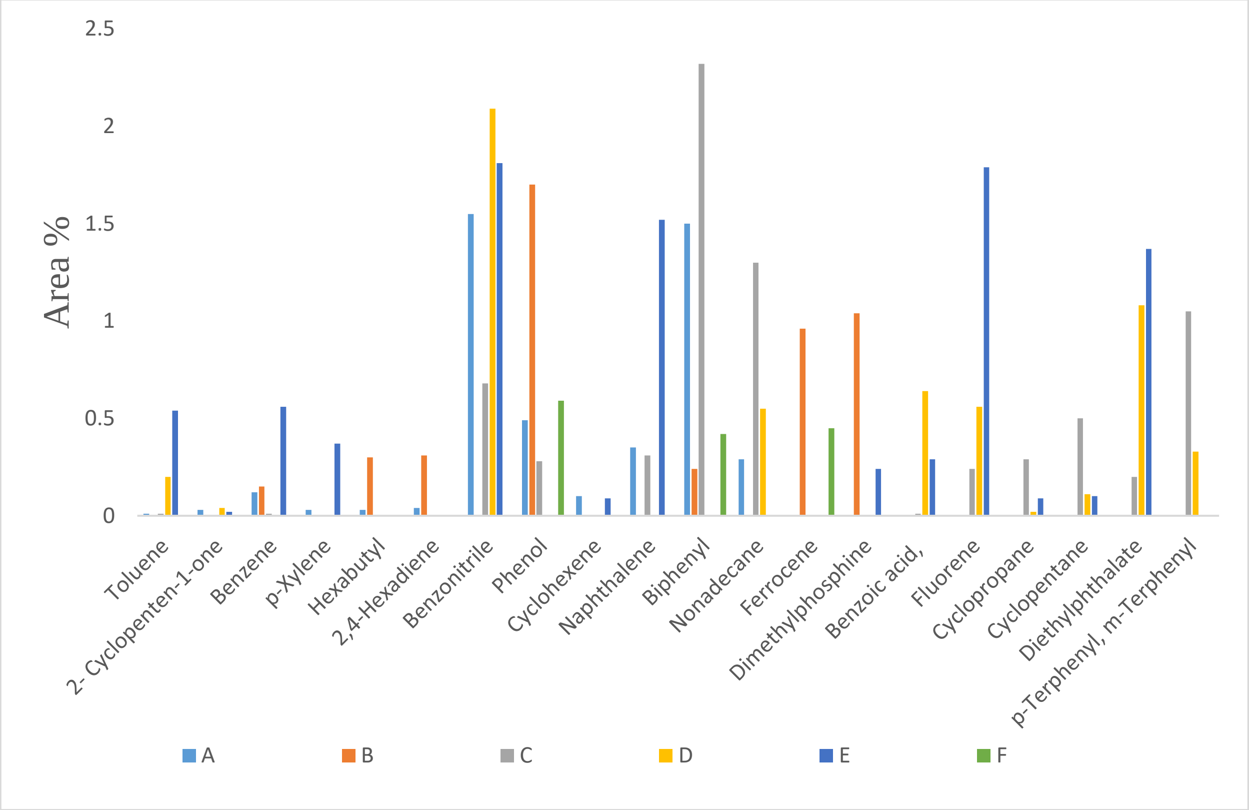

Figure 2: Comparison of samples A – F showing the most occurring components and their percentage area.

The analysis showed that apart from hydrocarbons, some other elements such as oxygen, nitrogen and alcoholic groups were present. This was similar to the work of Moinuddin and Mohammed, (2011) who used ZnO and Al_2O_3 as catalyst. That implied that plantain peel and Kaolin were suitable catalyst for PET pyrolysis. The advantage however with the plantain peel and kaolin over the use of ZnO and Al_2O_3 is that the latter temperature range for the pyrolysis was between 90°C and 405°C, while the plantain peel and kaolin clay temperature range was between 90°C and 300°C. Vakili and Fard, (2010), Siddiqui and Redhwi, (2011), Khaing and Chaw, (2015) and Ogunyemi et al., 2019 all got similar results using various inorganic catalysts.

Figures 3 and 4 shows the result of the yields from the thermal degradation of PET using Corn shaft/ kaolin clay and banana peel/kaolin respectively. Benzonitrile, biphenyls, benzoic acid, fluorene, 1,1 – biphenyl, benzaldehyde, styrene, phenol, benzene, ethylbenzene and naphthalene were among the compounds detected. These compounds are important industrial raw materials. These compounds were also detected when plantain peel was used. The results of this study was in agreement with most research works done on PET pyrolysis (Saha and Ghoshal, 2005; Al -Salam and Lettieri, 2010; Sarker et al., 2011; Claudinho and Ariza, 2017).

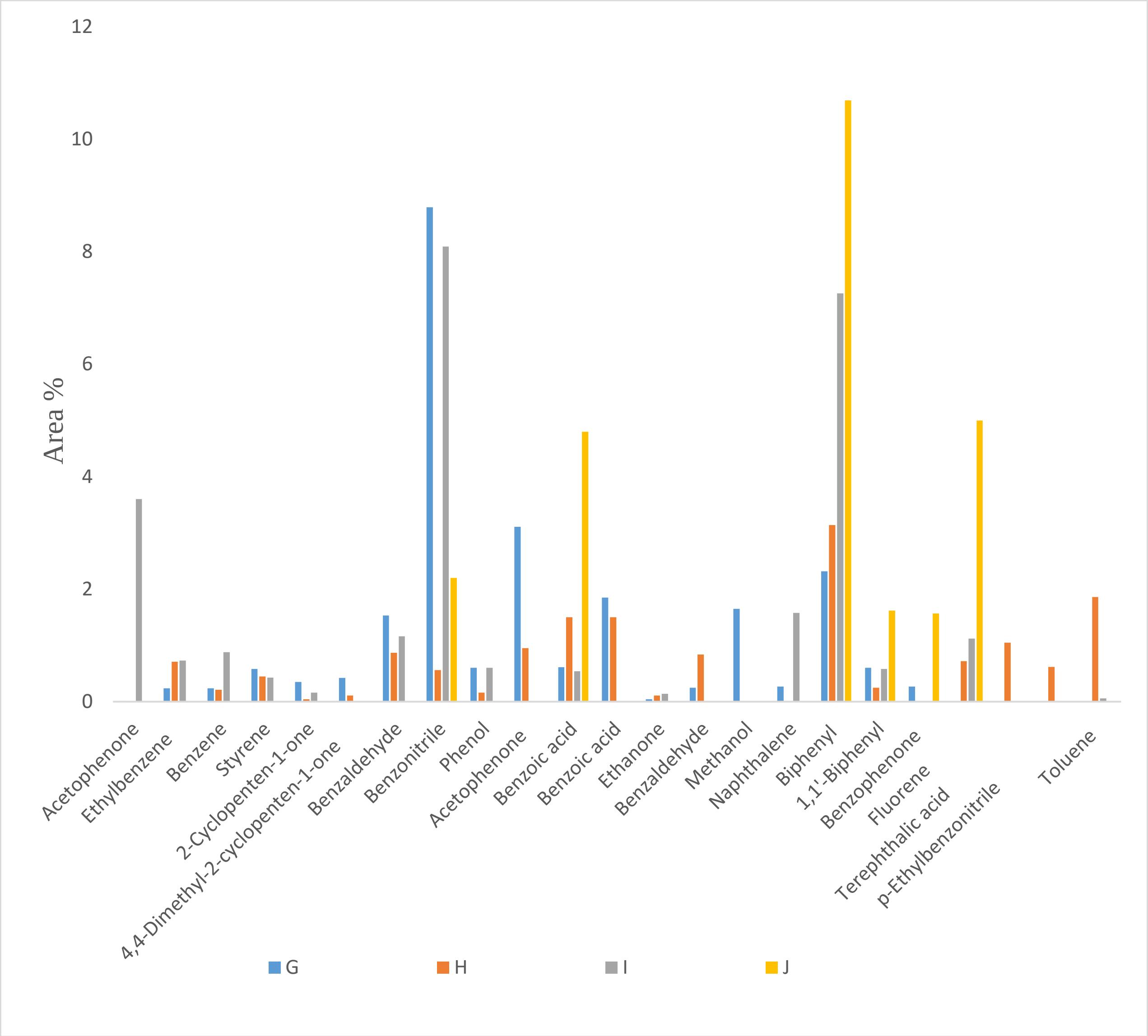

Figure 3: Thermal degradation of PET using Corn shaft and kaolin clay

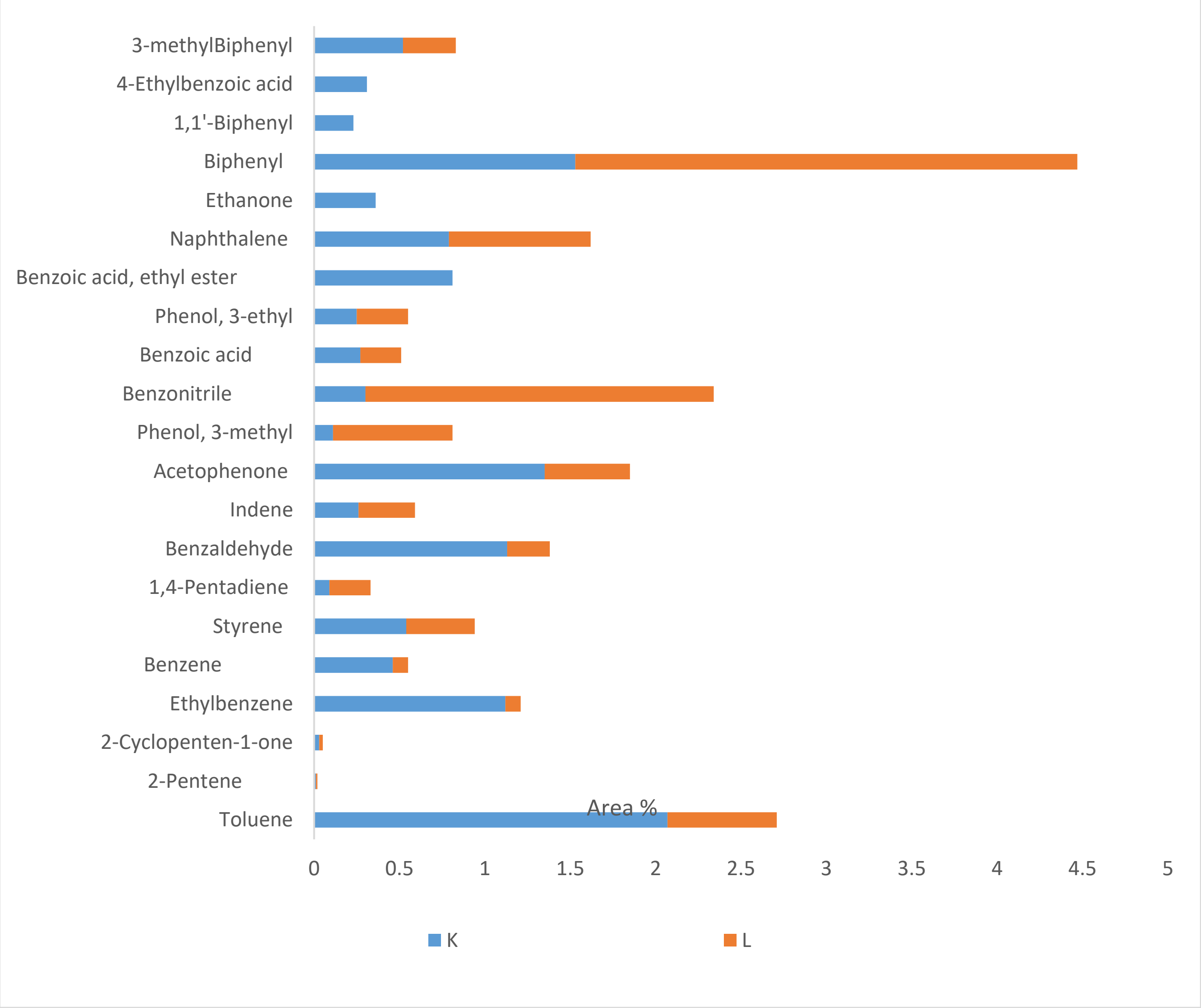

Figure 3: Thermal degradation of PET using banana peel and kaolin clay

Conclusion

Pyrolysis is a strong ally for revaluation of PET waste; it is of great advantage in the industrial production processes thereby generating a new purpose for this type of waste. The use of local materials such as plantain peel/kaolin; corn shaft/kaolin and banana peel/kaolin as catalyst have proved to be efficiency in the catalytic pyrolysis process by reducing the temperature and reaction time. Since plantain peels, banana peels and corn shafts are waste products, their use will serve as both reduction of solid waste and reducing the cost of PET pyrolysis. Hydrocarbons and other compounds produced by the pyrolysis of PET serves as primary feed stocks for petrochemicals industries such as pharmaceutical and pesticides. The GC – MS analysis of this present study using local materials indicated that waste PET can be converted into useful hydrocarbons and other petrochemicals.

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