

## THE EFFECT OF TEMPERATURE AND CATALYST MODIFICATION ON THERMOCATALYTIC DEGRADATION OF LOW DENSITY POLYETHYLENE

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### ABSTRACT

*Low density polyethylene (LDPE), which is non biodegradable, is a major constituent of municipal solid waste and source of light hydrocarbons when recycled using thermo-catalytic processes. In this study, the effects of temperatures (400 and 500 °C) and catalysts (commercial and modified mordenite) on the reforming of the vapour obtained from the pyrolysis of LDPE were studied. Commercial mordenite was acid treated and impregnated with 0.3 wt% Iron; the catalysts thus prepared were A1: commercial mordenite, A2: acid treated mordenite and A3: 0.3 wt% Fe loaded on mordenite. The catalysts were characterized using X-Ray Diffractometer and Brunauer-Emmett-Teller techniques, and then used to reform the pyrolysed LDPE at reforming temperatures 400 and 500 °C. The reformed products obtained were liquid and gases. The liquid products were analysed using GCMS. The results showed paraffins were predominant in the samples obtained from the use of A1. While the samples obtained using A2 and A3 had olefins and aromatics present. The carbon atoms in the hydrocarbons ranged from C<sub>5</sub> – C<sub>36</sub> for A1, and reduced to C<sub>5</sub> – C<sub>16</sub> and C<sub>5</sub> – C<sub>22</sub> for A2 and A3 respectively. The results showed the ability of mordenite to reform the vapours of pyrolysis to products within fuel range.*

*Keywords: Catalysts, LDPE, Pyrolysis, Reforming, Temperature.*

### 1.0 INTRODUCTION

Plastics have replaced to a large extent packaging materials; most of these packaging plastics are thermoplastic type of polymer which could be recycled. It is widely agreed that used and unwanted plastics become waste and are dumped at landfill site most often. Since most plastics are non - biodegradable, they constitute menace to the environment.

The most appropriate approach to eliminate or reduce the environmental problems created by waste plastics is recycling [Norena et al, 2012]. There are various kinds of recycling processes, of which the tertiary recycling process is an advanced and most desired. This is so because tertiary recycling process produces valuable chemicals and fuels [Puente et al, 2002].

Polyolefins such as polyethylene, polypropylene and polystyrene are produced and consumed massively due to their wide range of applications in recent time. Tertiary recycling of these polymers as they become waste has been studied under various approaches which include laboratory scale catalytic degradation whereby the polymer and the catalyst of choice are in contact in a reactor and enclosed; the set up is heated to a reaction temperature and allowed for a reaction time; then the products are collected and analyzed (Panda et al, 2008). On the other hand, thermal cracking otherwise known as pyrolysis has been the method in existence in which the

polymers are heated at very high temperature in order to breakdown the macromolecules of the waste polymer to smaller or micro molecules. Usually, the products of pyrolysis are a mixture of gaseous, liquid and solid hydrocarbons (Buekens et al, 1998) which are however, crude but can be upgraded. An advanced method of tertiary recycling is the two stage process involving the pyrolysis as the first stage and catalytic reforming as the second stage. Mochamad Syamsiro et al. (2013) reported on passing the products of pyrolysis through a catalytic reforming unit. They obtained and analyse wide range of products. The catalysts tested for the two stage recycling process include synthesized mordenite from Malaysian clay and commercial mordenite. This method caters for the shortcomings encountered in either of the previous two method earlier described.

Ishira et al. (1998) established the effective reforming of products of pyrolysis using several catalysts. They include zeolites like ZSM – 5, mesoporous MCM-41, USY, SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>, natural zeolites and so on; other forms of catalysts are metal impregnated on zeolite support like Nickel on ZSM -5, Cobalt on mordenite, etc.

In our study, , two stage recycling processes of pyrolysis and reforming were investigated using a commercial mordenite and its modified forms which included acid treatment and 0.3 wt% Fe impregnated on the

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mordenite. This is to enable comparative study of the performances of the catalysts.

## 2.0 Materials and Methods

A description of the materials and procedures adopted are outlined in this section.

### 2.1 Materials

The materials used in the work included commercial ammonium mordenite, which was purchased from Zeolysts International, Iron III nitrate nanohydrate (Guangdong Guanghua Chemical Factory Co., Ltd), distilled from petrochemical laboratory of National Research Institute for Chemical Technology, Zaria and waste water sachets (LDPE) collected from household wastes.

The equipment included X-Ray Diffractometer, VSorb APP surface area and porosity analyzer and Agilent Gas Chromatography/Mass Spectroscopy.

### 2.2 Preparation of catalysts

The commercial mordenite was first dried in an oven at 120 °C for 5 hours, and then calcined for 3 hours at 500 °C in a muffle furnace; this was named A1. The second catalyst, A2 was prepared by treating the ammonium mordenite with 500ml of 1M HCl at atmospheric pressure by stirring for 1hr. then, it was filtered and washed with distilled water, dried at 120 °C for 5 hours and calcined in a muffle furnace for 3 hours at 500 °C. A3 was prepared by impregnating Fe precursor i.e the iron III nitrate nonahydrate in the mordenite. 0.3 w/w% Fe was loaded on a 20 g of the mordenite. The quantity of the precursor, correctly calculated for loading, was dissolved in distilled water and poured in a beaker containing the 20g mordenite, it was heated at 75 °C and stirred slowly for 5 hrs. A3 was then dried in the oven at 120 °C for 5 hrs and calcined afterward in a muffle furnace at 500 °C for 3 hours.

### 2.3 Characterization of the catalysts

The crystal structures of the catalyst samples were determined using XRD; about 0.5 g of each sample was seeded on a ceramic substrate. The XRD patterns were captured using Rigaku MiniFlex 600 at 40 KV, 15 mA and; a scanning speed and step size of 10 degrees/min and 0.02° respectively for 2 theta values from 10 – 90. N<sub>2</sub> adsorption – desorption measurements were

conducted at 77 K (-196 °C) on a VSorb APP to obtain the BET specific surface area (S<sub>BET</sub>) as well as pore sizes and volumes of all the three samples.

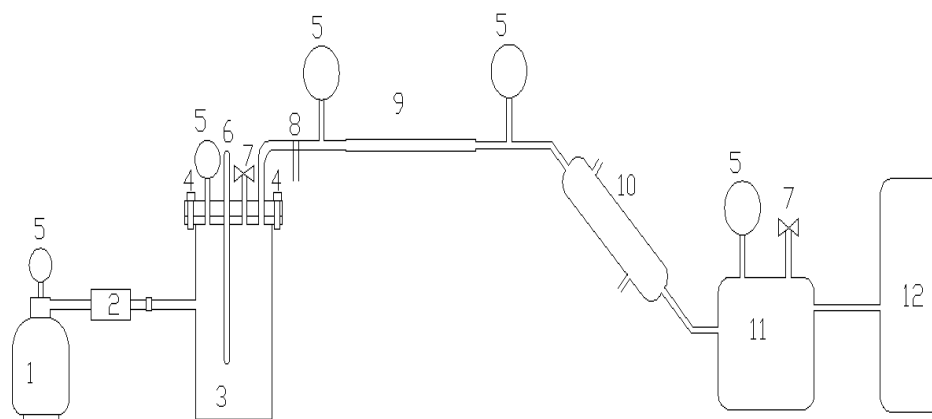
### 2.4 Preparation of the feedstock (LDPE)

- The waste plastics (LDPE) were gathered from houses,
- the plastic wastes are washed and sun dried,
- Size reduction of the waste plastics by shredding was done.

### 2.5 Experimental procedures:

The experimental set up comprised of a pilot scale pyrolysis/reforming reactor system which was designed and fabricated at National Research Institute for Chemical Technology, NARICT, Zaria. It is a two stage reaction set up for pyrolysis and reforming; the other components of the set up include the cooling and receiving sections. The schematic diagram of this set up is shown in Figure 1 with the component description. 10 g of the waste LDPE is weighed into the round bottom flask immersed in the heating mantle of heating rate of rate 5 °C /min, where the pyrolysis takes place. An external digital K type thermocouple as well as a nitrogen gas line was inserted into the flask.

Prior to the pyrolysis, the system is scrubbed with the nitrogen gas in order to create an inert system. The vapour produced from the pyrolysis stage is allowed to flow to the reformer where it was reformed by catalyst at varying temperatures. The reforming stage consists of the reformer which is a fixed bed reactor made of stainless steel and surrounded by heating element of heating rate of about 14 °C /min, the length of this reactor is 20.5 cm while the inner diameter is 2 cm. 1 g of catalyst was weighed and loaded in the reformer at every experimental run, the catalyst was guided by glass wool before and after in the fixed bed in order to maximise the gas phase contact and reaction. The reforming temperature was set at either 400 or 500 °C. The reformed vapours then were released into the separating flask immersed in an ice water bath that was continually chilled by the water circulating through a chiller. The vapours which consist of condensable and non condensable gases were separated; the condensable gases remained in the flask as the liquid product and the non condensable gases pass through a gas line into a gas bag.



**Figure 1: Schematic diagram of the experimental set up**

**Footnote: nitrogen tank (1), flowmeter (2), pyrolyser (3), pressure guage (5), temperature sensor (6), fixed catalytic bed (9), condenser (10), liquid collector (11) and gas collector (12).**

## 2.6 Analysis of Products

The chemical compositions of the liquid products were determined with Gas Chromatograph-Mass Spectrometer (GCMS) analyzer. The method described by Jin, Shen, Liu, & Xiao, (2016) was followed.

## 3.0 RESULTS AND DISCUSSION:

### 3.1 Characterization of catalysts:

The BET surface areas, pore volumes and pore sizes of the catalyst samples obtained from the  $N_2$  adsorption-desorption isotherms and calculated using the BET and Langmuir methods are illustrated in Table 2. Sample A1 has the least surface area ( $323\text{m}^2/\text{g}$ ) and pore volume ( $0.16\text{cm}^3/\text{g}$ ). Sample A2 has a higher surface area than A1, due to acid treatment and consequent dealumination of the mordenite which also indicate higher Si/Al ratio and more pore openings. The pore volume is also higher for A2 than A1. On iron impregnation, the surface area was smaller than that of the acid treated sample (A2), but still bigger than the initial mordenite sample (A1). The results on Table 1 also show that the pore size ranged from 2.0 – 2.7nm for all three catalysts (A1, A2 and A3). Hence, particles or molecules within the size

range of 2.0 – 2.7 nm can have access to the pores of the catalysts, where the active sites are situated, so as to be broken down or reformed (Syamsiro et al, 2013).

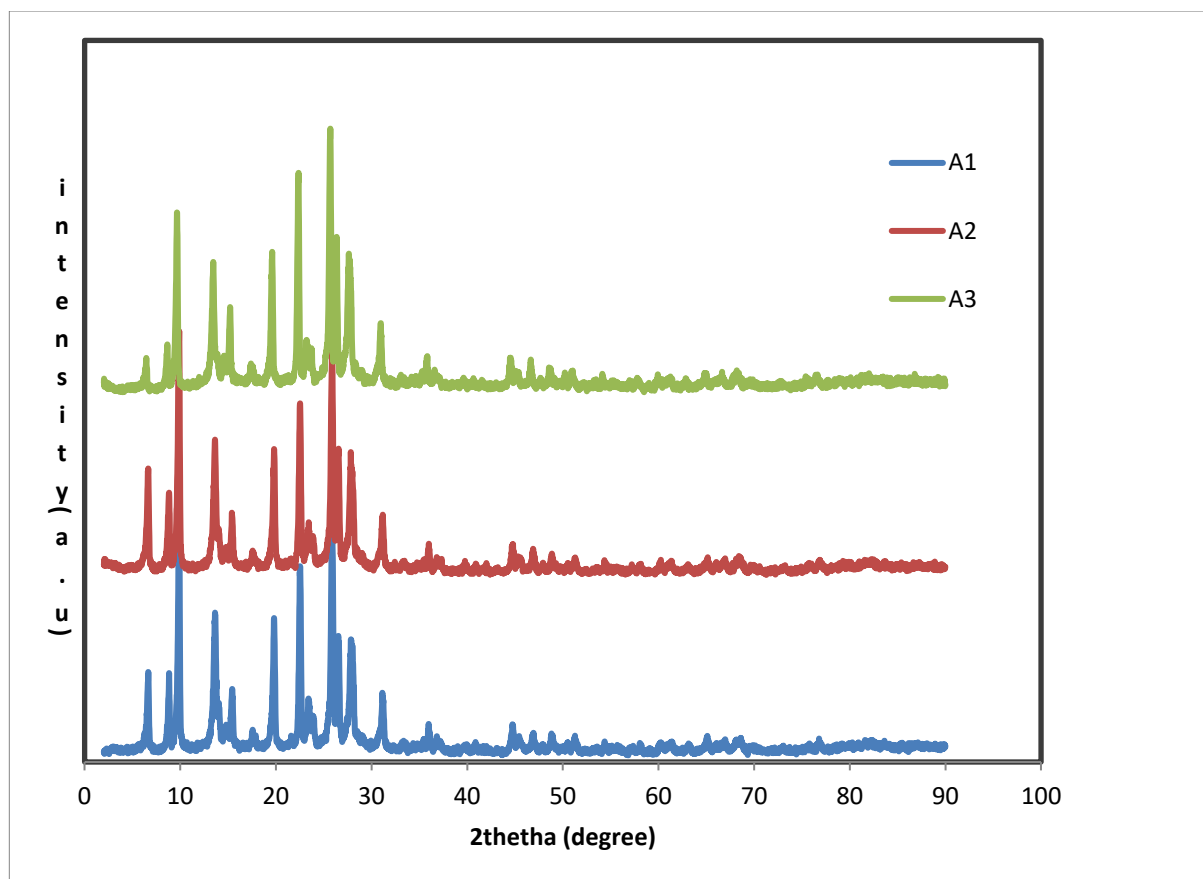
Figure 2 shows the crystalline structure of the three catalysts (A1, A2 and A3) as analysed by X-ray diffractometer. The diffraction patterns show peaks at  $2\theta$  values from  $10^\circ$  to  $32^\circ$ , corresponding to the structural framework of mordenite as described by Trisunaryanti et al., (1996). These peaks remained present despite the acid treatment and metal impregnation, indicating the crystalline nature of the samples. Therefore, the XRD analysis shows that the zeolite structure of mordenite was not damaged even after acid treatment and metal

impregnation. The presence of iron oxide tends to be indicated by peaks around  $2\theta$  values of  $33^\circ$  -  $37^\circ$  (Calsavara & Lucano, 2008). However, the diffraction peaks of iron oxide were not visible in Figure 2. This is probably because the amount of iron oxide impregnated in the mordenite was very small compared to the detectable amount of at least 5 w/w% reported in literature (Aziz et al., 2016).

**Table 1: BET report of the catalyst samples.**

S/N	CODE	$S_{\text{BET}}$ ( $\text{m}^2/\text{g}$ )	$S_{\text{L}}$ ( $\text{m}^2/\text{g}$ )	Pore size range (nm)	Pore volume ( $\text{cm}^3/\text{g}$ )
1	A1	324	428	2.1 – 2.6	0.156821
2	A2	421	554	2.0 – 2.6	0.200430
3	A3	384	506	2.1 – 2.6	0.177825

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**Figure 2: XRD patterns of: A1 (Commercial Mordenite); A2 (Acid Treated Mordenite) and A3 (0.3 w/w% Fe loading on Mordenite)**

### 3.2 PRODUCT ANALYSIS

Catalysts play significant roles in thermochemical reactions. They can improve the selectivity of desired products by driving the reaction towards a favourable reaction mechanism, which targets the desired product.

Tables 2 and 3 illustrate the chemical components of the liquid samples obtained from the GCMS analysis of the samples at 400 and 500 °C reforming temperatures respectively. Table 3 shows that the use of A1 results in a wide range of hydrocarbons from C<sub>6</sub> – C<sub>36</sub> identified to be majorly alkanes (paraffin). While the composition of the oil when A2 was used for reforming at 400 °C

includes a number of olefinic compounds (C<sub>5</sub>, C<sub>6</sub>, C<sub>7</sub>, C<sub>11</sub>) and paraffinic compounds with the highest carbon atom of C<sub>26</sub>. With the use of A3, a number of aromatic compounds (benzene), more olefins (C<sub>7</sub>, C<sub>11</sub>, C<sub>13</sub>) and paraffins with the highest carbon atom C<sub>23</sub> were obtained. This implies that as the reforming proceeds from no catalyst to A1 (without modification) to A2 (with acid treatment) to A3 (acid treatment and 0.3 wt% Fe), at the same temperature, cracking of the large hydrocarbons is enhanced progressively.

**Table 2: The most abundant component of the GC-MS analysis of the reformed pyrolyzed oil samples of LDPE at 400 °C**

Catalyst	Nomenclature	RT (min)	RA (%)	HC Type	No. of Carbon Atoms
A1	1,5-Heptadiene	8.85	0.28	Olefin	C <sub>7</sub>
	Decane	9.01	0.28	Paraffin	C <sub>10</sub>
	Undecane	10.89	0.93	Paraffin	C <sub>11</sub>
	Cyclodecane	12.47	0.67	Cycloparaffin	C <sub>10</sub>
	5-Dodecene	12.47	0.67	Olefin	C <sub>12</sub>
	1,6-Octadiene	12.69	0.22	Olefin	C <sub>10</sub>
	5-Tridecene	14.07	0.65	Olefin	C <sub>13</sub>
	Tridecane	14.18	1.39	Paraffin	C <sub>13</sub>

Catalyst	Nomenclature	RT (min)	RA (%)	HC Type	No. of Carbon Atoms
	1,5-Hexadiene	16.94	0.29	Olefin	C <sub>6</sub>
	Pentadecane	17.04	2.36	Paraffin	C <sub>15</sub>
	Hexatriacontane	25.82	2.97	Paraffin	C <sub>36</sub>
A2	Decane	9.01	0.63	Paraffin	C <sub>10</sub>
	4-Undecene	10.75	0.66	Olefin	C <sub>11</sub>
	Undecane	10.89	1.96	Paraffin	C <sub>11</sub>
	2,3-Hexadiene	12.08	0.28	Olefin	C <sub>6</sub>
	4-Dodecene	12.46	0.96	Olefin	C <sub>12</sub>
	Tridecane	12.61	4.74	Paraffin	C <sub>13</sub>
	3-methyl-1,4-pentadiene	13.84	0.35	Olefin	C <sub>5</sub>
	5-Tetradecene	15.55	1.18	Olefin	C <sub>14</sub>
	Tetradecane	15.67	6.30	Paraffin	C <sub>14</sub>
	1,5-Heptadiene	16.85	0.43	Olefin	C <sub>7</sub>
	Pentadecane	17.05	6.87	Paraffin	C <sub>15</sub>
	Octadecane	20.75	6.40	Paraffin	C <sub>18</sub>
	Nonadecane	21.86	5.36	Paraffin	C <sub>19</sub>
	Eicosane	22.91	4.73	Paraffin	C <sub>20</sub>
	Heneicosane	23.92	3.98	Paraffin	C <sub>21</sub>
	Docosane	24.88	2.96	Paraffin	C <sub>22</sub>
	Hexacosane	25.81	2.60	Paraffin	C <sub>26</sub>
A3	Nonane	6.95	0.40	Paraffin	C <sub>9</sub>
	Decane	9.01	1.49	Paraffin	C <sub>10</sub>
	2-ethyl-1,4-dimethylbenzene	10.61	0.42	Aromatic	C <sub>9</sub>
	4-undecene	10.75	1.25	Olefin	C <sub>11</sub>
	Undecane	10.89	2.99	Paraffin	C <sub>11</sub>
	2-methyl-2,3-hexadiene	11.74	0.62	Olefin	C <sub>7</sub>
	1-butylbenzene	11.92	0.65	Aromatic	C <sub>10</sub>
	Cyclopropylbenzene	13.87	0.42	Aromatic	C <sub>9</sub>
	1,5-Heptadiene	13.95	0.39	Olefin	C <sub>7</sub>
	Tridecene	14.06	2.4	Olefin	C <sub>13</sub>
	Hexadecane	18.35	6.70	Paraffin	C <sub>16</sub>
	Heptadecane	19.58	5.63	Paraffin	C <sub>17</sub>
	Octadecane	20.74	4.90	Paraffin	C <sub>18</sub>
	Nonadecane	21.85	3.81	Paraffin	C <sub>19</sub>
	Eicosane	22.89	3.35	Paraffin	C <sub>20</sub>
	Heneicosane	23.91	2.67	Paraffin	C <sub>21</sub>
	Triacosane	25.79	1.89	Paraffin	C <sub>23</sub>

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**Table 3: The most abundant component of the GC-MS analysis of the reformed pyrolyzed oil  
samples of LDPE at 500 °C**

Catalyst	Nomenclature	RT (min)	RA (%)	HC type	No. of Carbon Atoms
A1	1,5-Heptadiene	8.85	0.42	Olefin	C <sub>7</sub>
	4-Undecene	10.75	1.27	Olefin	C <sub>11</sub>
	Decane	10.89	1.23	Paraffin	C <sub>10</sub>
	5-Dodecene	12.42	1.50	Olefin	C <sub>12</sub>
	Cyclohexane	12.69	0.25	Cycloparaffin	C <sub>6</sub>
	6-Tridecene	14.07	1.89	Olefin	C <sub>13</sub>
	2,4,6-trimethyloctane	14.19	2.27	Paraffin	C <sub>11</sub>
	3-methyl-1,6-Heptadiene	15.46	0.57	Olefin	C <sub>7</sub>
	3-tetradecene	15.57	1.76	Olefin	C <sub>14</sub>
	2-methyl-2,3-Hexadiene	15.90	0.25	Olefin	C <sub>6</sub>
	Tetradecane	15.66	2.86	Paraffin	C <sub>14</sub>
	1-Pentadecane	16.94	1.43	Paraffin	C <sub>15</sub>
	Hexadecane	18.35	4.30	Paraffin	C <sub>16</sub>
	Octadecane	20.75	4.45	Paraffin	C <sub>18</sub>
	Nonadecane	21.86	4.48	Paraffin	C <sub>19</sub>
	Eicosane	22.92	4.24	Paraffin	C <sub>20</sub>
A2	Decane	9.01	0.82	Paraffin	C <sub>10</sub>
	4-Undecene	10.76	1.07	Olefin	C <sub>11</sub>
	4-Nonene	10.76	1.07	Olefin	C <sub>9</sub>
	Undecane	10.91	2.02	Paraffin	C <sub>11</sub>
	Benzene	11.95	0.64	Aromatic	C <sub>6</sub>
	Benzene	13.842	0.55	Aromatic	C <sub>6</sub>
	Benzene	14.70	0.43	Aromatic	C <sub>6</sub>
	4-Dodecene	12.48	1.38	Olefin	C <sub>12</sub>
	1,5-Heptadiene	12.72	0.33	Olefin	C <sub>7</sub>
	2,3-Hexadiene	13.33	0.37	Olefin	C <sub>6</sub>
	1-Tridecene	14.07	1.81	Olefin	C <sub>13</sub>
	1-Pentadecene	16.95	0.91	Olefin	C <sub>15</sub>
	Dodecane	12.63	5.19	Paraffin	C <sub>12</sub>
	Tridecane	14.22	7.91	Paraffin	C <sub>13</sub>
	Pentadecane	17.08	14.14	Paraffin	C <sub>15</sub>
	Hexadecane	18.37	11.31	Paraffin	C <sub>16</sub>
	Heptadecane	19.59	6.87	Paraffin	C <sub>17</sub>
	Octadecane	20.76	3.33	Paraffin	C <sub>18</sub>
	Nonadecane	21.86	2.05	Paraffin	C <sub>19</sub>
	Eicosane	22.91	1.47	Paraffin	C <sub>20</sub>
	Docosane	24.87	0.81	Paraffin	C <sub>22</sub>
A3	Decane	8.99	0.25	Paraffin	C <sub>10</sub>
	1,5-Heptadiene	10.74	0.5	Paraffin	C <sub>7</sub>
	4-Dodecene	12.45	0.84	Olefin	C <sub>12</sub>
	5-Dodecene	12.68	0.27	Olefin	C <sub>12</sub>
	4,4-dimethyl-2-	13.31	0.21	Olefin	C <sub>5</sub>

Catalyst	Nomenclature	RT (min)	RA (%)	HC type	No. of Carbon Atoms
	Pentene				
	5-tridecene	14.04	1.15	Olefin	C <sub>13</sub>
	1-butyl-Benzene	13.94	0.62	Aromatic	C <sub>6</sub>
	Dodecane	12.59	2.08	Paraffin	C <sub>12</sub>
	Tridecane	14.17	3.02	Paraffin	C <sub>13</sub>
	Pentadecane	17.02	2.43	Paraffin	C <sub>15</sub>
	Hexadecane	18.32	1.79	Paraffin	C <sub>16</sub>

Table 3 illustrates that at 500 °C, by using A1 the composition of the oil consists of hydrocarbons of majorly olefins (C<sub>7</sub> – C<sub>14</sub>) and paraffin (C<sub>10</sub> – C<sub>20</sub>) while using A2 and A3 result in aromatic compounds (benzene, methylbenzene and butylbenzene) and more olefinic compounds. The results show that the highest carbon atom obtained when A2 was used is C<sub>22</sub>, while with A3, C<sub>16</sub> was obtained; implying enhanced cracking in the presence of A2 and A3. Thus, the GC-MS analysis also confirms that increasing temperature from 400 to 500 °C played a key role in enhancing the cracking of the larger hydrocarbons.

Generally, the quality of the oil improved as catalyst was changed from A1 to A2 to A3 as well as increase in temperature from 400 to 500 °C. Also, the surface areas of A2 (421 m/g) and A3 (385 m/g) probably had accessible active sites where the reforming of the HCs took place. The aromatic compound present is mainly benzene, while the olefins are within the carbon number range of C<sub>5</sub> to C<sub>14</sub>; the paraffin reduced to C<sub>10</sub>- C<sub>16</sub>. The oil obtained when A3 at 500 °C was used consists of gasoline range HC (C<sub>5</sub> – C<sub>12</sub>), naphtha range HC (C<sub>8</sub> – C<sub>12</sub>), kerosene range HC (C<sub>11</sub> – C<sub>14</sub>) and diesel and fuel oils HC (C<sub>11</sub> – C<sub>20</sub>).

#### 4.0 CONCLUSIONS:

In this work, the performance of mordenite and Fe – mordenite on the reforming of pyrolysis vapours was investigated at varying temperatures. It can be concluded that the XRD results showed no structural deformation done to the mordenite despite the modification by acid treatment and iron loaded on the zeolite framework. The BET result showed the surface areas of the catalysts with the highest as 421 m<sup>2</sup>/g (A2) and lowest is 384 m<sup>2</sup>/g (A3); and pore sizes ranging from 2.0 – 2.7nm.

Two stage processes of pyrolysis and catalytic reforming was carried out on the waste LDPE. The composition of the liquid products analyzed by GCMS was largely paraffins of C<sub>6</sub> – C<sub>40</sub> for A1. The liquid products obtained for reforming over A2 and A3 contained some paraffin, olefin, diolefin and aromatic of C<sub>6</sub> – C<sub>16</sub> and C<sub>6</sub> – C<sub>22</sub> respectively. The aromatic compound present in the liquid is benzene.

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