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Catalytic conversion of waste low density polyethylene into fuel oil

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Abstract

Waste Low density polyethylenes (LDPE) represent a source of energy and valuable chemicals, were pyrolysed catalytically in a batch reactor under atmospheric pressure. Calcium carbide was used as a catalyst to explore its effect on pyrolysis product distribution. The effect of temperature, amount of catalyst and time on the yields of the pyrolysed products was investigated. The effect of catalyst on the liquid yield was also studied. The results demonstrate that temperature has a promising effect on the yield; however high temperature, as well as high catalyst loading, caused a decline in liquid yield. The liquid obtained from catalytic pyrolysis were also characterized by physical and chemical tests. Among the physical tests Density, Specific gravity, API gravity, Viscosity, Kinematic viscosity, Aniline point, Flash point, Watson Characterization Constant, Freezing Point, Diesel Index, Refractive Index, Gross calorific value, Net calorific value and ASTM Distillation were determined according to IP and ASTM standard methods for fuel values. From the physical tests it was observed that the results for the liquid fractions are comparable with the standard results of physical tests for gasoline, kerosene and diesel fuel oil. Phenols and carbonyls were quantitatively determined by spectrophotometric methods using Folin-Denis and Phenyl Hydrazine reagents respectively. The components of different hydrocarbons in the oil mixture were separated by using column chromatography and fractional distillation followed by characterization with FT-IR spectroscopy.

Keywords: Catalytic pyrolysis; Waste LDPE; Liquid fuel; Calcium carbide.

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1. Introduction

Waste LDPE from household and industries are recognized to be a major environmental problem and a source of energy. There are several methods for disposal of municipal and industrial LDPE wastes, i.e. landfill, incineration, true material recycling, and chemical recovery. Landfill treatment and incineration destruction are quite expensive and may raise problems with unacceptable emissions. True material recyclin i.e. to convert the waste material into products that can be reused, can significantly reduce the net cost of disposal. However, it is generally accepted that true material recovery is not a long-term solution to treat waste LDPE.

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Uddin *et al* [1], Sharratt *et al* [2], Park *et al* [3] , Masuda *et al* [4], Mclntosh *et al* [5], Kagayama *et al* [6], and Kaminsky *et al* [7], were investigated chemical recycling, through which waste plastics can be converted into fuel oil and valuable chemicals. Thermal and catalytic degradation of waste plastics are two kinds of chemical recycling processes. The main drawbacks of thermal degradation are wide product distribution and requirement of high temperatures, typically more than 773K and even up to 1173K. Since thermal degradation demands relatively high temperatures and its products require further processing for their quality to be upgraded, catalytic degradation of polymer waste offers considerable advantages. Catalytic pyrolysis gives a means to solve these problems. Suitable catalysts have the ability to control both the product yield and product distribution from polymer degradation as well as to reduce significantly the reaction temperature.

Luo *et al* [8] were studied catalytic degradation of polyethylene into liquid fuel in a powder-particle fluidized bed. The optimum temperature they used was comparatively high that is 823K. Lee *et al* [9], Dawood *et al* [10], and Monos *et al* [11], were also studied the effects of catalysts on the catalytic degradation of plastic by contacting melted plastic with catalyst in fixed bed reactors. Zhang *et al* [12], reported the current situation of recycling waste plastics and technology of converting waste plastics into oil. Koç *et al* [13], were also worked on catalytic and thermal oxidative pyrolysis of LDPE in a continuous reactor system. LDPE was both decomposed thermally and catalytic in a continuous reactor via as an oxidative media in the temperature range of 673–773K. So for the studies reported in the literatures about waste LDPE pyrolysis have been carried out in the presence of various expensive catalysts and derived oils of high boiling point range at comparatively high temperature.

The objective of the present study was to convert waste LDPE catalytically at relatively low temperature as well as to derive oil in the boiling range of commercial fuel oil. In this paper calcium carbide (CaC2) was used as a catalyst for conversion of waste LDPE into fuel oil. Calcium carbide consists of pi (π) electrons and at high temperature energies of pi (π) electron changes [14]. Vibration of pi (π) electrons increases the vibrational energies of LDPE up to resonance and results in pyrolysis of polyethylene.

2. Experimental

2.1. Material and methodology

For the preparation of polyethylene representative sample, fine cuttings of polyethylene shopping bags of $2cm^2$ area were cut with the help of scissor and used for analysis. Thermogravimetric analysis of polyethylene sample was performed using a Perkin –Elmer TGA7 type instrument in which about 10 mg plyethylene samples was heated. The temperature was increased from 313K to final temperature of 1273K at a rate of 293K/minute. The sample temperature was measured with a thermocouple directly at the crucible very close to the sample. For investigation of the optimum temperature for thermal analysis of polyethylene, five grams by mass fine cuttings of polyethylene sample were placed in pyrex glass tube and heated it in the heating assembly for 60 minutes at different temperature i.e. 523, 573, 623 and 673K, respectively. The cracking products at each temperature were collected and based on maximum conversion into liquid oil optimum temperature was determined.

For catalytic pyrolysis of polyethylene five gram of fine cutting of polyethylene sample of $2cm^2$ area and known amount of CaC_2 catalyst (100μm mesh size) were mixed and added to the reaction assembly for the catalytic pyrolysis experiment. The pyrolysis experiments were carried out in a batch reactor under atmospheric pressure [15-18]. A precisely weighed amount of both the sample and catalyst (Calcium Carbide) was loaded in a preweighed pyrex glass tube of 8.0 cm I.d. x 18 cm length, which was then put into the batch reactor. The reactor was heated with an electrical furnace. The temperature was measured by thermocouple and controlled by a temperature controller. The outlet of the glass tube was connected to trap in order to condense and weigh the liquid products. The glass tube was removed from the reactor at the end of catalytic pyrolysis and cool down to room temperature. The mass percentage of product composition could then be calculated for % conversion, % oil, % residue and % gas according to the following formula given below.

$$\% \text{ Conversion} = \frac{\text{(mass of polyethylene - mass of residue)}}{\text{mass of polyethylene}} \times 100$$

$$\% \text{ Oil} = \frac{\text{mass of oil}}{\text{mass of polyethylene}} \times 100$$

$$\% \text{ Residue} = \frac{\text{mass of residue}}{\text{mass of polyethylene}} \times 100$$

$$\% \text{ Gas} = \% \text{ Conversion} - (\% \text{ Oil} + \% \text{ Residue})$$

Reflux study of the parent oil derived from catalytic pyrolysis of polyethylene using calcium carbide as catalyst

2.2. Procedure

120 mL of the parent oil product, which was obtained during the catalytic pyrolysis process, and 20 g of the CaC_2 catalyst were transferred to a pyrex glass tube and then the tube was inserted into the reactor, the temperature of the reactor was adjusted at 623K and the mixture was refluxed for 2 hours. The refluxed oil was characterized using physical and chemical tests given below.

2.3. Determination of physiochemical properties of the liquid product

The liquid column chromatography was used to separate the different groups of hydrocarbons present in the derived oil. The silica gel 60 (63-200 μm grain size, supplied by Merck) was packed into borosilicate glass column and the pyrolysis oil applied to the top of the column. The column was then eluted with n-hexane, benzene and methanol to produce aliphatic, aromatic and polar fractions of the pyrolysis oil, respectively.

Fractional distillation was carried out to separate different boiling point hydrocarbon fractions from the liquid derived from catalytic pyrolysis of waste LDPE. The oil was distilled at a specific temperature until no more distilled products were collected. Infrared spectroscopy was used for identification of liquid product measuring absorption from 400 to 4400cm^{-1}. Phenols and carbonyls were quantitatively determined by spectrophotometric methods using Folin-Denis and Phenyl Hydrazine reagents, respectively.

The fractions obtained by this method were also characterized by using density, refractive index, refractive index parameter (I) and refractivity intercepts (RI). The refractive indexes were calculated with Abbe's refractometer (P20-Warsaw, Poland) at 293K. Refractive index parameter and refractivity intercepts were calculated for all the fractions by using the formulas:
Refractive index parameter (I) = η^2-1/ η^2+2
Refractivity Intercept (RI) = $\eta-d/2$
η= Refractive index, d= density.

Some physical properties of pyrolytic oils were determined by using the following standard methods: Flash point by Cleveland open cup method IP-36/84 and ASTM-D92-78. Density IP-59/82, API gravity IP-160/87 and ASTM-D1298-85, Kinametic viscosity IP-711/87 and ASTM-D445-87 and Distillation IP-191/83 and ASTM-D216-77 were determined according to IP and

ASTM standard methods for fuel. Gross calorific value was determined using Gallenkamp Auto Bomb Calorimeter (England).

3. Results and discussion

The mass loss verses temperature curve for thermal decomposition of polyethylene under a linear heating rate Fig.1 shows that the decomposition was a single step process with offset and end temperature of 448.5K and 673K, respectively. The curve shows that maximum mass loss related to volatilization of hydrocarbons occurred at 673K. Total 99.98 % mass changes occurred from 448.5K to 673K. The residue mass was 0.2 % and was found constant upto 1273K. This indicates that certain amount of char like carboneous material was formed during the decomposition of polyethylene. Along with the volatilization process, other reactions like cracking of side chains from aromatic rings, isomerization and polycondensation also occur. Therefore it indicates that all volatile matter of polyethylene has been decomposed to volatile hydrocarbons.

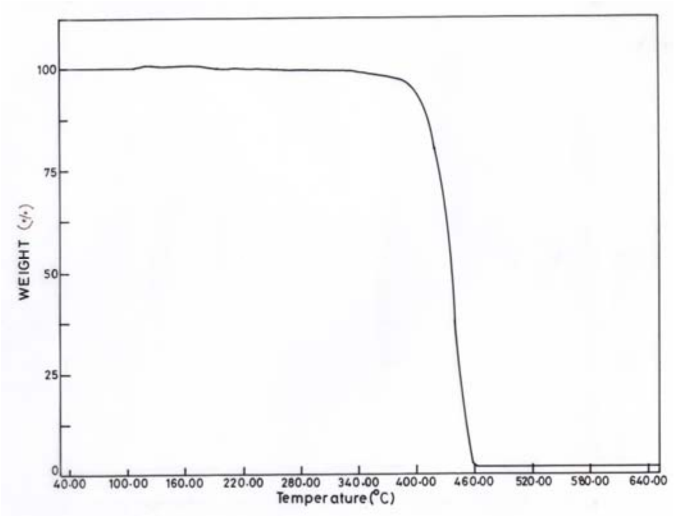

Fig. 1. TGA curve of waste LDPE.

Polyethylene sample was also pyrolysed at different temperatures. The results are given in Fig. 2. It can be seen from these results that maximum conversion was obtained at 673K. At this temperature conversion into liquid oil product was 12.0 ± 0.2 %, while conversion into gaseous product was 13.36 ± 0.32 %. Total conversion at this temperature was 25.36 ± 0.52 %.

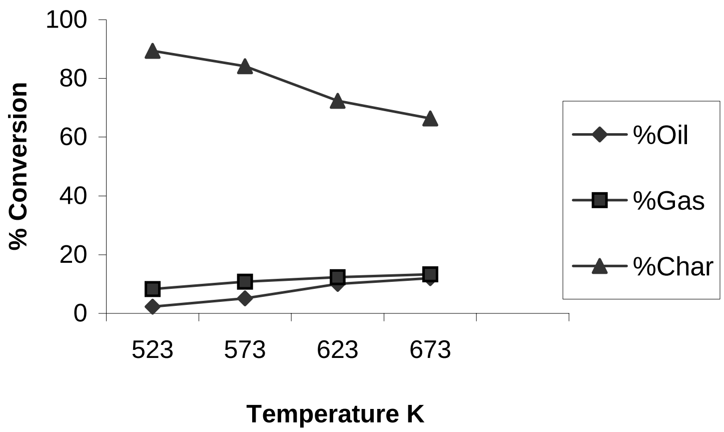

Fig 2. Temperature optimization for pyrolysis of waste LDPE

In order to investigate the optimum temperature for catalytic pyrolysis of polyethylene, polyethylene sample was catalytically pyrolysed at varied temperatures. The results are given in Fig.3. The product obtained was liquid oil, gases and solid residue. The purpose of temperature optimization was to find out suitable temperature at which maximum liquid product could be obtained. Total conversion was based on the amount of liquid and gas formed during catalytic pyrolysis. It can be seen from the results that maximum conversion into liquid product 69.73 % was achieved with this catalyst at 623K temperature. Beyond the optimum temperature a decrease in the liquid fraction was observed which could be due to further conversion into volatile product leading to higher fraction of gases and decrease in liquid fraction.

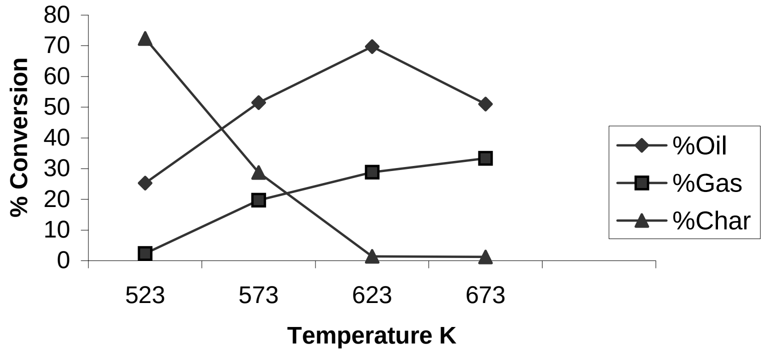

Fig 3. Temperature optimization for catalytic pyrolysis of waste LDPE

In the next step the effect of catalyst mass on the catalytic pyrolysis of polyethylene was investigated in the range of 0 to 2.5 g at optimum temperature. The results are given in Fig. 4.

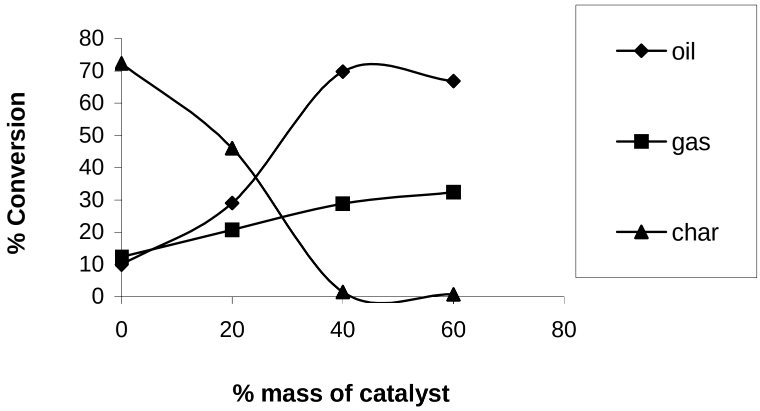

Fig 4. Catalyst mass optimization for catalytic pyrolysis of waste LDPE

In the absence of catalyst the conversion into liquid was only 10.0 %, while in the presence of catalyst the yield of liquid increased up to 69.73 % with 1g of catalyst (10 %). So 1g of catalyst weight (40 %) was found as optimum catalyst mass for it. Below the optimum catalyst mass conversion into liquid oil was minimum, while higher mass of catalyst lead to higher gaseous product. This is due to the fact that at higher catalyst mass the rate of reaction increases the process of cracking, which results in maximum gaseous product. The effect of residence time on the catalytic pyrolysis was also studied at optimum conditions of temperature and catalyst mass and the results are given in Fig.5.

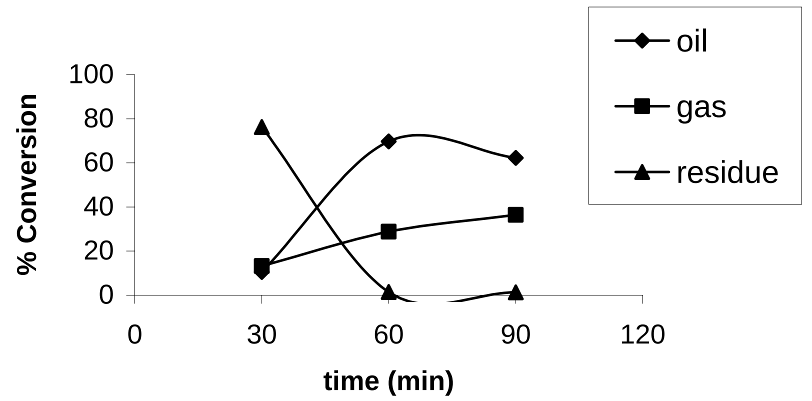

Fig 5. Time optimization for catalytic pyrolysis of waste LDPE

It was investigated that 60 minutes reaction time sufficient for maximum conversion into liquid product. At this optimum time the conversion into liquid was 69.73 %, which was the maximum conversion. At optimum conditions the liquid product was collected and analyzed for different fuel properties.

3.1. Characterization of liquid product

The liquid obtained in polyethylene catalytic pyrolysis is a liquid product, usually termed oil. In order to better analyze the potential uses of the oil, separation of oil on silica gel column with the aim to separate and get an idea of the nature and type of compounds present in such oil. The sample was loaded on to the column and elution was done using different solvents like n-hexane, benzene and methanol. It was expected that aliphatic fraction will be eluted with n-hexane, aromatic with benzene and polar hydrocarbons with methanol. The results are given in Table 1.

Table 1

Percentage of different fractions collected by column chromatography of waste LDPE using CaC_2 catalyst.

S.No	Solvent system	% age of fraction
1	n-hexane	40
2	Benzene	30
3	Methanol	25

It can be seen from these results that the derived oil has maximum fraction of non-polar (40 %) followed by relatively non-polar (30 %) and minimum polar fraction (25 %). The hydrocarbon groups of the derived oil having a boiling range of 353K-490K were also separated into different boiling point range by fractional distillation. The results are given in Table 2. As can be seen from the table that 15 wt % oil are below 443K and 40 wt % between 443-523K.

This shows that maximum concentrations of hydrocarbons are with in the range of initial boiling point 353-523K. The concentration of hydrocarbons are 13.33 wt % and 30 wt % for boiling point in the range of 523-573K and greater than 573K, respectively. This indicates that major fraction of the oil contained components are in the boiling point up to 573K which is the range of commercial petrol. All physical parameters like density, refractive index, refractive intercept, refractive index parameter and specific refraction for each fraction were also measured and are given in Table 3. It can be seen from the results that the densities and refractive indexes of oil increased from 0.812 to 0.921g/cm^3 and 1.416 to 1.456, respectively.

Table 2
Fractional distillation study of oil obtained using CaC_2 catalyst.

S.No	Temperature (K)	% of each fraction
1	353-443	15
2	443-523	40
3	523-573	13.33
6	>573	30

Table 3
Physical parameters of various fractions collected at different temperatures from fractional distillation of oil obtained using CaC_2 catalyst.

Temp (K)	%	Density (g/ml)	Refractive index (η)	Refractive intercept (RI)	Refractive index Parameter (I)	Specific refraction (γD)
443	15	0.812	1.416	1.010	0.250	0.125
523	40	0.898	1.442	0.993	0.265	0.132
573	13.33	0.899	1.446	0.996	0.266	0.133
Residue	30	0.921	1.456	0.996	0.272	0.136
Parent	25	0.900	1.441	0.991	0.264	0.132

There is pronouncing increase in refractive indexes and densities with an increase in boiling point of the fraction. At lower temperature low molecular mass hydrocarbons are present and with increase in boiling point the molecular mass of the compounds are also increased. Low values of refractive indices are associated with paraffins and higher values with aromatic and intermediate with naphthanic hydrocarbons. The nature of the hydrocarbon group present in each fraction was further identified from the refractivity intercept (RI) and it gives specific value for each hydrocarbon group. The RI value of paraffins lies in the range of 1.048-1.05, for naphthenic 1.03-1.046 and for aromatics 1.07-1.105. The RI values for fractions obtained in the boiling point range from 443-573K lie in between 0.993-1.010 which show the presence of nephthenic and paraffins hydrocarbons in the derived oil.

ASTM Distillation test at atmospheric pressure was carried out, for the oils obtained at optimum conditions. The results are presented in Fig.6. It was observed that 60 % volume of such oils was easily distillable fraction with boiling range between 353-473K which is the boiling point range specified for commercial petrol. On the other hand Fig.6 shows that the remaining 40 % vol. of the oils has a boiling point under 490K, which is the boiling range for the 50 % of distilled product in commercial kerosene oils.

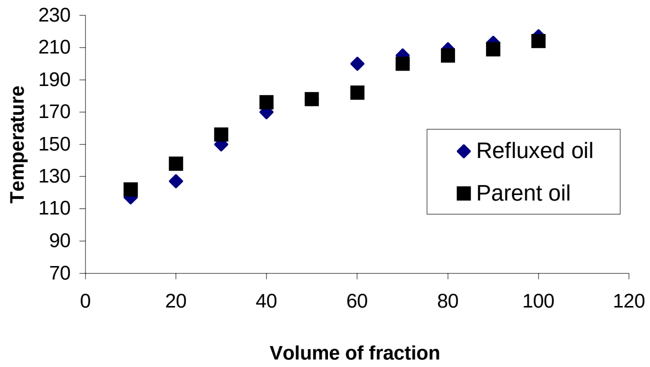

Fig 6. ASTM Distillation for catalytic pyrolysis of waste LDPE

The oil obtained from catalytic pyrolysis with different catalysts without any further process has been named as parent oil. To find out if there is any further cracking, each parent oil has been refluxed with respective catalyst at temperature 623K for 2 hours time. The fuel properties like density, specific gravity, API gravity, Viscosity, Kinematic viscosity, aniline point, flash point, Watson characterization constant, freezing point, calorific value, diesel index, sulfur and ASTM distillation for both the parent and refluxed oil in each case has been measured and are given in Table 4, and were compared with standard values for gasoline, kerosene and diesel which are given in Table 5.

Table 4

Typical properties of oil derived from catalytic pyrolysis of oil obtained from catalytic pyrolysis of waste LDPE.

S.No	Parameters	Oil derived with CaC_2 catalyst	Refluxed Oil
1	Density (g/mL)	0.759	0.765
2	Specific gravity	0.767	0.766
3	API gravity	52.93	53.12
4	Viscosity (Centipois)	1.084	1.096
6	Kinematic viscosity (mm^2/s)	1.429	1.432
7	Aniline Point (K)	341	338
8	Flash Point (K)	359	317
9	Watson characterization constant	12.163	12.179
10	Freezing point (K)	255	254
11	Gross calorific value (MJ/kg)	43.1	43.3
12	Net calorific value (MJ/kg)	41.2	41.5
13	Diesel index	81.719	79.144
14	Sulfur (%)	0.68	0.69

The density for the parent oil was 0.759 g/cm^3 and 0.765 g/cm^3 observed for the respective refluxed oil. On the basis of the value of densities both parent and the refluxed oil can safely be classed in between gasoline and kerosene (densities 0.736 to 0.82 g/cm^3).

Table 5
Standards parameter of gasoline, diesel and kerosene oil.

S.NO	Parameters	Gasoline	Diesel	Kerosene
1	Density (g/mL)	0.736/0.725	0.834	0.780-0.82
2	Specific Gravity	0.70	0.85	0.78
3	API gravity	65	23-30	41.7-39.66
4	Viscosity (Centipois)	0.7750-0.8394	2.0-4.5	0.9-1.5
5	Kinematic viscosity (mm^2/s)	5.0	3.77 -5.0	2.2
6	Aniline point (K)	338	344	335
7	Flash point (K)	310.8-311	328-333	323-328
8	Watson characterization constant	12.45	11.28	12.126
9	Freezing point (K)	215	219	-
10	Diesel index	83.44	54	59.88
11	Bromine number	60	1-10	
12	Gross calorific value (MJ/kg)	45.6	43.5-55.7	46.5
13	Sulfur (%)		0.70	
S.No	ASTM Distillation	Temp (K)	Temp (K)	Temp (K)
0	Dew point	322	393	383
1	10mL	342	498	451
2	20mL	351	511	453
3	30mL	355	525	453
4	40mL	359	545	455
5	50mL	363	554	463
6	60mL	367	563	465
7	70mL	371	572	471
8	80mL	375	573	473
9	90mL	379	574	483
10	100mL	385	578	493

Specific gravity values for the parent oil 0.767 and for the refluxed oil were 0.766 respectively. Again no change in specific gravity was observed when parent oil was refluxed with respective catalyst. Again these values fall in the range in between of gasoline and kerosene (0.70 to 0.78).

The values for API gravity were found to be in the range of 52.93 of parent oil and 53.12 for refluxed oil. It was observed that API gravity has slightly increased on refluxing. The API gravity values also fall with in the API gravity values in the range in between gasoline and kerosene (39.66 to 65). Viscosity and kinematic viscosity values both for parent and refluxed oil were measured and 1.084 centipoises, 1.429 mm^2/s for parent oil and 1.096 centipoises, 1.432 mm^2/s for refluxed oil. It was observed that there is slight increase both in viscosity and kinematic viscosity upon refluxing with CaC_2 as a catalyst. Viscosity and kinematic viscosity values are both in agreement with the viscosity and kinematic viscosity values for kerosene (0.9–1.5centipoise and $2.2mm^2/s$).

The values for aniline point were found to be 341K and 338K both for the parent and refluxed oil, respectively. A decrease in aniline point was observed on refluxing. The aniline point values are in agreement with gasoline (338K). The values of flash point for parent and refluxed oils vary from 316 and 317K. It was observed that there was a slight increase in the flash point. The flash point values indicate that the oils obtained using CaC_2 as a catalyst in the range of gasoline and kerosen (293.8-311K to 323-328K), respectively.

The Watson characterization constant values were found to be 12.163 and 12.179 for both parent and refluxed oil. No appreciable change in these values was observed on further refluxing with catalyst. From the value of watson characterization constant the oil may be considered aromatic and nephthenic in nature.

The values of freezing point, gross calorific value and net calorific values lies in the range of 255K and 254K, 43.1 and 43.3 MJ/Kg and 41.2-41.5 MJ/Kg, respectively. In case of freezing point it was observed that there was a slight change in the value upon refluxing. The values of freezing point are in agreement with freezing point value of kerosene.

Incase of gross calorific value and net calorific value a slight increase in the value was observed in all cases upon refluxing. In all cases, these values are in agreement with the gross calorific and net calorific values of gasoline and kerosene respectively. The value for diesel index both for parent and refluxed oil vary in the range of 81.719–79.144. It was observed that these values decrease in case of refluxing. The results of the diesel index lie in the range of gasoline (83.44). Sulpher contents for parent and refluxed oil vary in the range of 0.68–0.69 %. These values are in agreement to the sulphur contents for gasoline (0.7 %).

The results for ASTM distillation are given in Fig.6. It can be seen that the initial boiling point is far beyond the initial point of gasoline which is 310-313K in summer grade gasoline oil. In some cases, the dew point is very high which indicates that the oil under study mostly lie with in kerosene range. The final boiling point lies in the range of kerosene oil (523K).

It can be seen that the dew point of the refluxed oil is lesser than the parent oil. This shows that the catalytic activity of this catalyst might have cleaved heavy molecular weight configuration into light oil which cause initial boiling point to occur at temperature less than the parent oil. The final boiling point observed in the range of 490-487K, which again is the range of kerosene oil.

Table 6

Quantitative determination of total phenol and carbonyl compounds in fractions collected at different temperatures.

Temp (K)	Phenol (μg/mL)	Carbonyl (μg/mL)
443	2400	35
523	1620	48
573	4450	20
Residue	450	19
Parent	8920	123

The oxygenated compounds were determined quantitatively in terms of total phenols and carbonyls. The results are given in Table 6. It can be seen from the results that the parent oil with CaC_2 catalyst shows 8920 μg/mL of total phenols and 123 μg/mL of carbonyls. The presence of small concentration of phenols and carbonyls indicates that it may be due to the pyrolysis, cyclization and interaction of cracking product with atmospheric oxygen at high temperature in the presence of catalyst.

The interpretation of FTIR spectra shows that catalytic pyrolysis of LDPE leads to the formation of a complex mixture of alkanes, alkenes, carbonyl group containing compounds like aldehydes, ketones, aromatic compounds and substituted aromatic compounds like phenols.

4. Conclusion

It could be concluded, that catalytic pyrolysis of LDPE leads to the production of fuel oil, valuable resource recovery and reduction of waste problem. Catalytic pyrolysis of waste LDPE has also several advantages over other alternative recycling methods. It has been shown that the conversion at lower temperature in the presence of catalyst into liquid is a feasible process. An important difference is that the oil obtained relatively with greater volume and low boiling range in the presence of catalyst as compared to pyrolysis in the absence of catalyst. The total pyrolytic oil can be blended with the gasoline or kerosene. Consequently, evaluation of waste LDPE by catalytic pyrolysis is very important from economic and environmental point of view. However, further studies are necessary to utilize pyrolytic oil as liquid fuel or feedback.

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