*Egypt. J. Chem.* **Vol. 68**, No. 6 pp. 623 - 636 (2025)

**Egyptian Journal of Chemistry**

**http://ejchem.journals.ekb.eg/**

# Utilization of an unconventional oxidative degradation catalyst for converting plastic wastes to petroleum like fractions

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## Abstract

Plastic waste is one of the serious problems that comprehend a negatively impact on the environment. The objective of the current work is to develop a process for converting some disposals such as wastes of polyethylene (WPE), polypropylene (WPP) and polystyrene (WPS), mixed with used lubricating oil (ULO) as carrier media, to more valuable products. This technique is currently put into practice under atmospheric pressure, using lignin, as a catalyst. The results demonstrated that the largest magnitude of gasoline can be attained from the dissociated WPS/ULO product, as it constitutes approximately 90 wt.% of the cracked sample, On the other hand, 52.3 wt.% of the prepared gasoline-like is available in the liquefied WPP/ULO, whereas the least quantity of this fraction (42.3 wt.%) is accessible in the degraded WPE/ULO. The distillates analogous to kerosene, the recorded data reveal that this fraction attained from the degraded WPP/ULO and WPE/ULO is approximately similar in amounts, comprising about 26-25 wt.%.On contrast, the product received from WPS/ULO cracking is characterized by the scarcity of this cut, as it represents 2.6 wt.% from the yield .the constituents of the heavier fraction petroleum gas oil the quantities of these components are equal to 16.6 and 20.8wt.% for degraded WPP/ULO and WPE/ULO, successively, whereas the lowest value of 7.4 wt.% was derived from the cracked WPS/ULO.

***Key words:*** Cracking, polyolefin wastes, oxidative degradation, plastic wastes, natural phenolic catalyst, used lubricating oil.

## 1. Introduction

Several researches studied the conversion of polyolefins plastic waste to fuel and other products by oxidative cracking process [1-12]. The oxidative degradation is more favorable than the traditional processes, which used various acidic and basic catalysts, such as silica-alumina, HY and HZSM-5 zeolites, mordenite, or alkaline compounds like zinc oxide[13–19].The main problems of the catalytic cracking operations are the high level of catalysts prices, aside from their fast deactivation and coke deposition which cannot be recovered and regenerated perfectly, that lead to premium in the cost of the waste plastic cracking to unacceptable point economically [20]. This has motivated us to use the oxidative reactions for cracking several hydrocarbon compounds, including the three waste polyolefins, i.e. polyethylene (WPE), polypropylene (WPP), and polystyrene (WPS) [21–25]. In this method, air is used as a cheap source of oxygen, and phenol is used as a catalyst. Furthermore, lignin was employed specifically in the current study in the capacity of a phenolic compound, since it encloses numerous units of phenols in its chemical structure[26]. It is also distinguished by several preferable qualities, as it is available, non-volatile, and an inexpensive material. The reaction mechanism of the atmospheric oxidative cracking appears to be similar to the mechanism that occurs at high reaction pressure. It starts at a moderate temperature at less than 200°C, where the oxygen of air extracts hydrogen from the phenol molecules, forming phenoxy radicals and water, accompanied by a rise in the reactants' temperature (making it an exothermic reaction). Then, the phenoxy radicals at a temperature of 300°C induce more extraction of the labile hydrogen from the hydrocarbon molecules of the polymer, randomly generating hydrocarbon free radicals. This free radical degraded into two molecules, one of which is a free radical and the other is an unsaturated hydrocarbon molecule. The hydrocarbon free radicals can extract the labile hydrogen that is connected with secondary and

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Received date 07 October 2024; revised date 31 October 2024; accepted date 03 November 2024
DOI: 10.21608/ejchem.2024.326579.10594

tertiary carbon atoms in the other polymer chain molecules (propagation radicals) to give new polymer radicals, etc. At the end of the process, the free radicals will repaired with each other to terminate the reaction [27].

Therefore, this work is concerned with the oxidative cracking of polyolefins plastic wastes to be converted to fuel and other products through new less expensive technique in which lignin extracted from animal manures was employed as a catalyst at atmospheric pressure.

## 2. Materials and Methods

### 2.1. Materials:

Polyolefins plastic waste was purchased from recycling market in Shobra city, Egypt. The animal manures were kindly supplied from a farmer in Benha city, Egypt. Used Lubricating Oil was collected from Kamilia fuel stations in Benha city, Egypt.

### 2.2. Methods*:*

### *2.2.1 Preparation of spent lubricating oil sample.*

Used Lubricating Oil (ULO) was mixed with 0.5 wt.. % aqueous potassium hydroxide and heated to 85 °C aiming to remove the insoluble compounds from the oil. The mixture was stirred (300 rpm) for one hour and then left to settle for 5 hours. The oily layer was heated to 40 °C, separated by decantation, and then filtered by a press filter to give the dray, pre-treated spent lubricating oil.

### *2.2.2 Preparation of the catalyst.*

Lignin is a very complex molecule which is found in loew, palms Bara, beech wood, animals dung, industrial black liquor, etc. All these materials are mainly large phenolic compounds. In this research, we degraded the lignin from animal manures to use its small fragments as non-volatile phenolic catalysts in the present oxidative degradation reaction. The degradation of lignin was performed in the fallowing steps.

I- 350 g of animals dung was washed with an HCl solution of 1.5 pH, stirred for 5 minutes, and then centrifuged for separating the solid lignin from the acidic solution.

II- The separated solid lignin was washed with distilled water several times.

III- The solid lignin was charged inside an autoclave with 200 g $H_2O$ and 25 g $H_2O_2$ (con. 30%). The reactor was then sealed, heated up to 340 °C and held at that temperature for 1 hour during the reaction.

IV- The reactor was left to cool and then opened to obtain the degraded fragments of lignin.

V- The degraded lignin obtained was dried and pulverized in a mixer for 10 minutes, and used as a catalyst in the form of small phenolic fragments.

### *2.2.3 Blank Experiment*

500 g of polyethylene and 500 g of used lubricating oil were charged into a steel reactor. The reactor was closed, charged with $N_2$ gas three times, and heated to 460 °C. The reaction was carried out for 75 minutes without any catalyst at atmospheric pressure. The obtained volatile gases were passed during the reaction through 4 condensers to get the liquid products and gases. The reactor was left to cool and opened to get the residue inside its vessel and estimated.

### 2.2.4 *Cracking process*

500 g of waste polyolefin (polyethylene, poly propylene, polystyrene) was charged individually into a steel reactor with 2 wt.% of phenolic compounds as a catalyst. 500 g from pretreated spent lubricating oil was added also inside the reactor as a carrier medium, and then the reaction was performed in the following steps:

Step 1: The reactor was sealed, charged by 4 liters of air (the actual volume of reactor is 5 liters) and heated to 220 °C with continuous stirring (100 rpm). Then the autoclave was opened for releasing volatile gases to condensers. This step was performed to permit the reaction of oxygen with phenolic lignin molecules to give phenoxy radicals without escaping from the reaction medium.

Step 2: The volatile gases evolved from the reactor are released through the outlet valve to facilitate the atmospheric pressure reaction during the residence time of the reaction. The reactor is heated up to the desired operating temperature range (450-465°C) and then maintained at these temperatures for 75 minutes. The evolved gases are reserved by 4 condensers to condense the condensable gases into a liquid, while the non-condensable gases (permanent gases) are collected and subjected to gas chromatographic analysis (GC analysis). Finally, the autoclave was left to cool to room temperature with continuous stirring, and then opened to obtain the residue by dissolving in ethylene chloride, filtered to separate the charge substance, dried using a rotary evaporator, estimated and then subjected to gas chromatographic analysis (GC analysis) for finding its carbon number distribution.

The obtained liquid products from the degradation of the three polymers were subjected to a distillation process to fractionate them into gasoline, kerosene, and diesel oil fractions, similar to petroleum-like fractions.

### 2.2.5 *The physicochemical characterization.*

The physicochemical properties of all these samples were determined according to the following ASTM test methods: D240 (calorific value), D1298 (Density), D4294 (sulfur content), D97 (pour point), D93 (flash point), D57 (carbon residue), D445 (kinematic viscosity), D482 (ash content), D3238 (for CP, CN and CA), D86 (IBP and FBP), and D-130 (copper corrosion).

### 2.2.6 *Infrared Spectra*

The structural characteristics of the cracked polymers were investigated through Fourier Transform Infrared (FTIR) analysis, using a Nicolet iS-FTIR, Nicolet iS-10. The spectra were recorded in the wave number range of 4000-400 $cm^{-1}$.

### 2.2.7 *Gas Chromatographic analysis.*

#### 2.2.7.1 *The liberated gases from the cracked process:*

The mixed hydrocarbons and $CO_2$ gases evolved from the oxidative cracking of various waste polymers were analyzed using a Varian CP-800 type C gas analyzer, equipped with a thermal conductivity detector (TCD) and a flame ionization detector (FID).

HP-1 capillary column, 60 meters in length and 0.25 mm in diameter, was used in addition to a stainless steel packed column, Propack Q, 10 feet in length and 1/8 inch in diameter. The elution of the investigated gases was accomplished with temperature programming, from50°C to 200°C, at a rate of 6°C $min^{-1}$. Nitrogen (oxygen-free) was employed as a carrier gas, flowing at a rate of 30 ml $min^{-1}$, and was measured from the end of the column with a soap bubble flow meter. Methane and air were used as markers to correct the dead volume in the columns in case of FID and TCD respectively. The injector temperature was 200°C and the detector temperature was 250°C [28, 29]

The quantitative analyses of the liberated gases utilized a standard natural gas sample of known composition and according to the standard ASTM method [29]. The data were estimated by integration of the area under the resolved chromatographic profiles, using HP computer of software chemstation. The calorific values of the gaseous fuels were calculated using the standard method [29]. These calculations were based on the gas composition of the liberated gases.

#### 2.2.7.2 *The liquid products.*

The liquid products obtained (degraded oil and its corresponding distilled petroleum- like fractions) were analyzed using an Agilent 6890-A plus HP gas chromatograph, equipped with an FID using the HP-1 capillary column, 60 meter in length and 0.25 mm in diameter, attached with FID. The injector and detector temperatures were 300°C and 320°C, respectively. The elution of the studied liquid products was achieved with temperature programming, starting from 50°C isotherm for 5 minutes, then ramping up to 300°C at a rate of 5°C $min^{-1}$. Pure nitrogen gas, free from oxygen, was used as the carrier gas, flowing at a rate of 30 ml/min.

## 3. Results and Descussion

### 3.1. FTIR spectroscopic analysis:

FTIR technique was utilized to identify the structural information of the degraded liquids of WPE, WPP, WPS and used lube oil carrier (ULO) in the wave number range of 4000-400 cm-1. The spectra of the liquid products obtained from the oxidatively cracked polymers, as well as ULO are shown in Figure1. The demonstrated patterns of the examined samples exhibit a broad signal in the high-frequency area around 3600-3200 cm-1, that dominated by the stretching mode of OH groups, likely derived from atmospheric air. The extreme broadness of this band may be ascribed to the presence of hydrogen bonding [30].

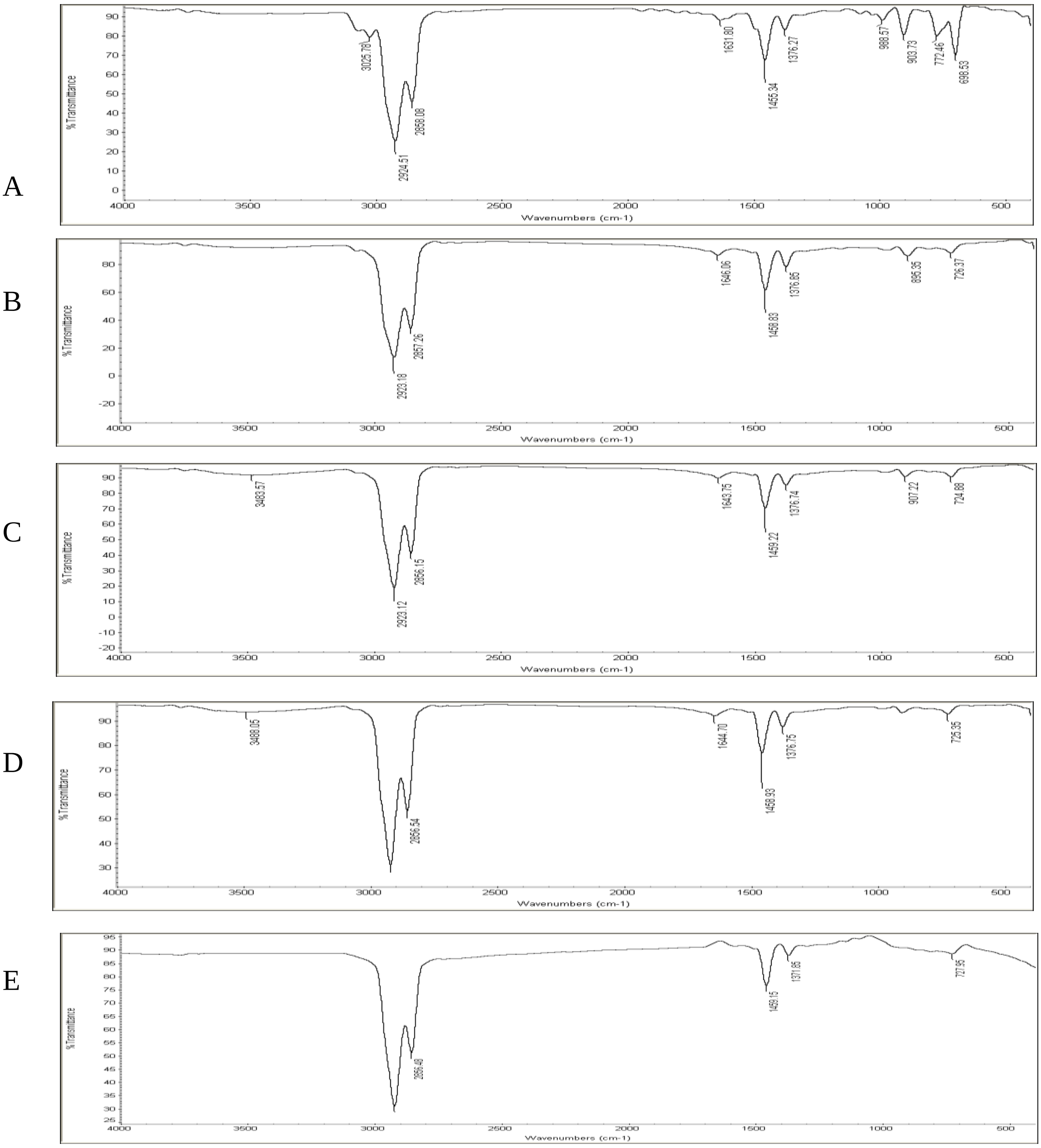

**Figure 1:** FTIR spectra of the liquefied waste polymers: A (WPS/ULO), B (WPP/ULO), C (WPE/ULO), D blank (WPE), and E (ULO)

The strongest and sharpest peaks, appearing close to 2923 cm$^{-1}$ and 2857 cm$^{-1}$, correspond to the stretching vibrations of carbon-hydrogen groups in alkanes, such as $CH_2$ and $CH_3$. Furthermore, two medium signals were recorded at 1459cm$^{-1}$ in the IR spectra of all evaluated samples, except for WPS/ULO, where it shifted to 1455cm$^{-1}$. The second peak is visible at about 1376 cm$^{-1}$. These peaks are assigned for the carbon-hydrogen bonding absorption of $CH_2$ and $CH_3$, respectively.

Several bands were observed in the lower wavenumber region between 989 cm-1 and 699 cm-1, in addition to a small shoulder at approximately 3100 cm-1, specifically in the spectrum of cracked WPS/ULO polymer (Fig 1A). These peaks are imputed to the out-of-plane bending as well as the stretching vibration of the benzene, successively. The emergence of these signals can be attributed to the aromatic nature of the waste polystyrene sample. However, the spectral analysis of dissociated paraffinic polymers (WPP/ULO and WPE/ULO) also reveals certain weak signals at 725cm-1 and 907cm-1, corresponding to the out-of-plane vibrations of the benzene ring (Fig1 B, C). These peaks likely arise from the contaminated ULO carrier, which embraces some aromatics, as evidenced by the unresolved band in the same region of the waste lubricant oil pattern (Fig 1D).

The depicted spectra of the degraded waste polymers under investigation display a weak signal in the wavenumber range between 1659 $cm^{-1}$- 1631 $cm^{-1}$, resulting from the bending mode of C=C double bonds of olefins. Moreover, another peak is conspicuous at about 3026 $cm^{-1}$ in the pattern of WPS/ULO, which proceeds from the stretching vibration of alkenes. The weakness of these bands indicates the efficiency of saturation of the olefin molecules throughout the autocatalytic hydrogenation during the cracking process.

Furthermore, there were no pronounced signals detected in the alkene region in the spectra of the assayed samples. Consequently, the degraded output of the inspected waste polymers consisted mainly of saturated hydrocarbons. Likewise, the IR pattern of ULO also elucidated the absence of any representative peaks for olefinic compounds (Fig.1E). Hence, the cracked products of the investigated distillates did not require further costly hydrogen treatment for their upgrading.

On the other hand, the exposed spectra of the oxidatively cracked specimens did not show any noticeable signals in the oxygenated group region at 1750-1540 $cm^{-1}$, and 1300-1000 $cm^{-1}$ related to C=O and C-O groups, unceasingly. This substantiates the non-existence of such oxygenated constituents resulting from this process, despite of use of oxygen from the charged air, which may be discarded as water. So, water is considered the only oxygenated compound resulting from this reaction.

### 3.2. Gas chromatographic analysis of the investigated cracked waste polymers:

The three liquids obtained during the oxidative cracking of polyethylene (WPE), polypropylene (WPP), and polystyrene (WPS), mixed with used lubricating oil (ULO), as well as the blank WPE/ULO, were subjected to gas chromatographic analysis to determine their constituents and the distribution of their hydrocarbons. The composition of each component was estimated in terms of mol% and wt.% by applying the normalized peak area technique.

The qualitative and quantitative data of these products are demonstrated in Figure 2 and Table 1 and in Tables S1, S4.

**Table 1**: Gas chromatographic analysis of liquefied obtained from oxidative cracking of WPS/ ULO, WPP/ULO, WPE/ULO and blank WPE/ULO

| Fraction | Boiling range | Range of n-paraffins | WPS /ULO | WPP /ULO | WPE /ULO | Blank WPE /ULO |
|---|---|---|---|---|---|---|
| | | | Wt., % | Wt., % | Wt., % | Wt., % |
| Gasoline | 40-180 | C4 –C10 | 89.177 | 52.262 | 42.319 | 38.888 |
| Kerosene | 180-280 | C11 - C15 | 2.601 | 26.448 | 25.385 | 23.963 |
| gas oil | 280-380 | C16 – C23 | 7.352 | 16.635 | 20.844 | 24.367 |
| Residue | > 380 | > C23 | 0.87 | 4.655 | 11.452 | 12.782 |

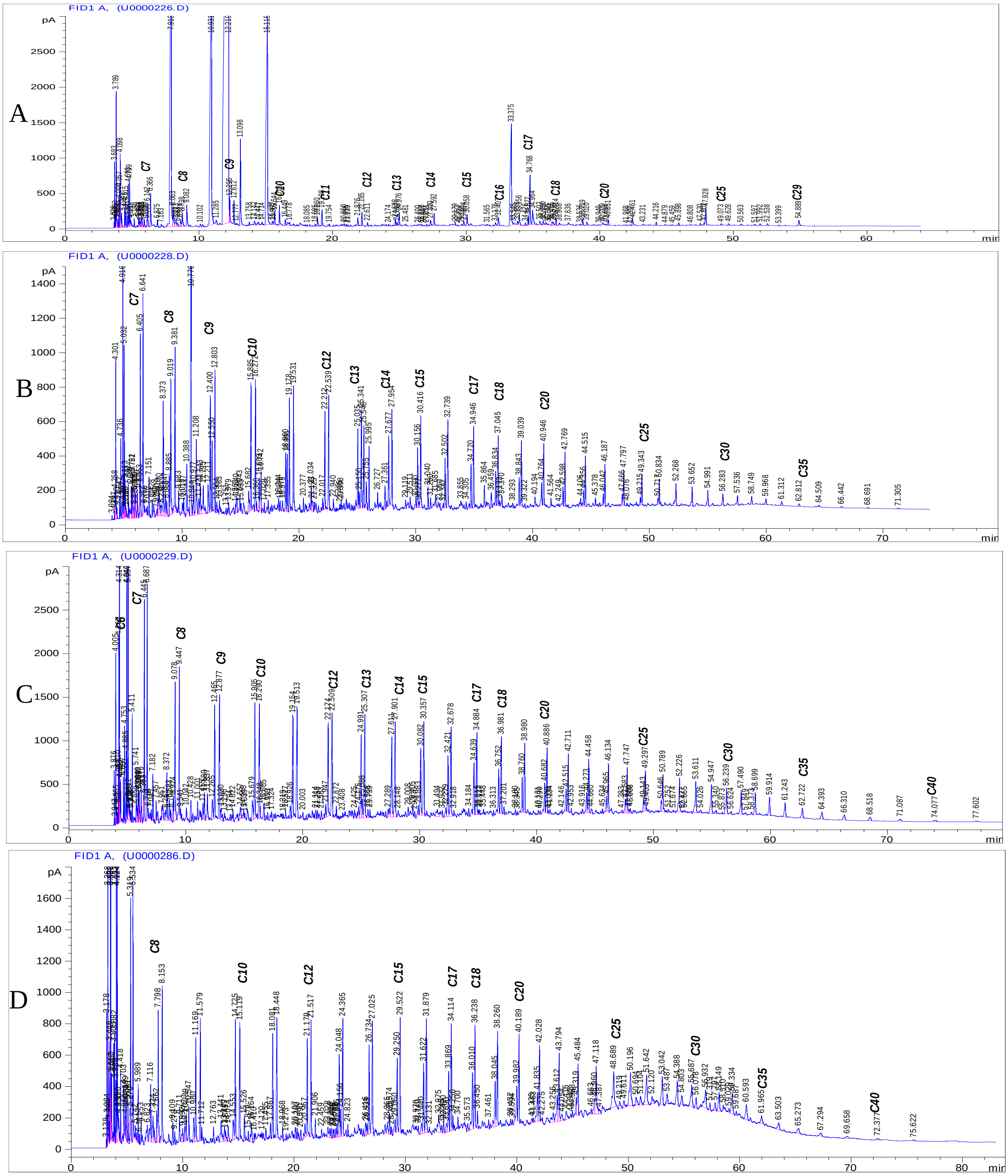

**Figure 2:** Gas chromatographic analysis for liquefied samples: A (WPS/ULO), B (WPP/ULO), C (WPE/ULO), and D blank (WPE/ ULO).

The provided information shows that each of the three examined samples includes light, middle, and heavy fractions contaminated with some residuals. According to their boiling points, they are classified as gasoline, kerosene, and gas oil, corresponding to those originated from petroleum crude oil.

The gasoline-like fraction, with a boiling point started from the ambient temperature up to 180°C, involves hydrocarbon components with carbon numbers ranging between $C_5$ to $C_{10}$, present in varying proportions depending on the nature of the tested waste polymer.

It is clearly obvious that the largest magnitude of gasoline can be attained from the dissociated WPS/ULO product, as it constitutes 89.177 wt.% of the cracked sample, It could be noticed that nonane($C_9$) represents the most abundant hydrocarbon in this specimen, with an amount coming to >60 wt.%. In addition, considerable amounts of octane ($C_8$) and decane ($C_{10}$) as much as 11.9 and 10.2 wt.% are also detected, respectively (Fig 2 A and Table 1).

On the other hand, 52.3 wt.% of the prepared gasoline-like is available in the liquefied WPP/ULO, whereas the least quantity of this fraction (42.3 wt.%) is accessible in the degraded WPE/ULO. It is noticeable that the hydrocarbon components of these two gasolines-like are distinguished by their different concentrations (Table 1).

The distillates analogous to kerosene, those boiling at 180°C to 280°C, embrace hydrocarbons in the range of C11 to C15. The recorded data in Table ( 1) reveal that this fraction attained from the degraded WPP/ULO and WPE/ULO is approximately similar in amounts, comprising about 26-25 wt.%.

On contrast, the product received from WPS/ULO cracking is characterized by the scarcity of this cut, as it represents 2.6 wt.% from the yield (Table 1).

The exposed results in Table (1) exhibit the constituents of the heavier fraction that boils in the range of 280°C - 380°C, and is considered to be identical to petroleum gas oil. This fraction encloses hydrocarbon components, with carbon numbers fluctuating between $C_{16}$, and $C_{23}$. The quantities of these components are equal to 16.6 and 20.8wt.% for degraded WPP/ULO and WPE/ULO, successively, whereas the lowest value of 7.4 wt.% was derived from the cracked WPS/ULO.

It is obvious that the amounts of gasoline and kerosene-like fractions acquired from the blank test are, to some extent, lower than those achieved from the cracked WPE/ULO polymer, whereas the heavier cuts (gasoil and residues) are relatively higher in magnitude (Table 1).

The fractions boiled above 380°c are considered residuals. Their amounts can be arranged in descending order as follows: WPE/ULO (blank 12. 8) >WPE/ULO (11.5 wt. %) > WPP/ULO (4.66 wt. %) >WPS/ULO (0.97wt. %).

### 3.3. Gas Chromatographic analysis of the liberated gases

The gases liberated from the oxidative degradation of WPS/ULO, WPP/ULO, WPE/ULO and Blank (WPE/ULO) were also analyzed via gas chromatography. The received data, expressed in mol% and wt.%, are described in Table S5.

It is so noticeable that the emanated gases are composed mainly of light hydrocarbons, ranging from methane up to hexane and beyond, contaminated with restricted amounts of carbon dioxide. The carbon dioxide levels ranged between 7.67-1.39 mol% (14.58 - 3.11 wt.%) for the WPS/ULO and WPP/ULO samples, respectively, but dropped to become just traces, not exceeding (0.211 mol%) (0.5 wt.%) for the WPE/ULO (blank) sample.

The ensuing hydrocarbon gases comprised primarily methane, which represents the most prevalent component, accounting for 66.71, 80.5, 77.9 and 83.32 mol% (corresponding to 46.23, 65.57, 61.85 and 71.69 wt.%) of the dissociated WPS/ULO, WPP/ULO, WPE/ULO, and WPE/ULO (blank) samples, respectively. Considerable amounts of ethane, ranging from 10.38 to 11.33 mol% (15.85-16.86 wt.%), are also included in the evolved gases.

A lower concentration of n-propane come to 3.62-1.38 mol% (between 7.89 -3.27 wt. %.), was existed as revealed from Table S5. The remaining alkanes (from $C_4$ to $C_6$) were rarely existed, with total proportions amounting to 0.35-2.25 mol% (ie 1.23 - 6.63 wt. %).

The emitted gases resulting from the oxidative cracking of the investigated waste polymers incorporate some isomerized hydrocarbons such as iso-$C_4$, and iso-$C_5$. They are accessible but with infrequent concentrations, getting to 0.47-1.07 mol % (1.36–2.83 wt.%) for those related to WPE/ULO and WPS/ULO, successively. In contrast, the blank WPE/ULO shows a scarcity of isomerized constituents, likely due to the absence of the catalyst during the process applied to this sample.

The depicted data demonstrate the detection of some unsaturated hydrocarbons in the evolved gases, including ethylene, propylene, and butenes. These olefins are available in amounts ranging between 9.23-3.424 mol%, i.e, 12.17- 5.03 wt.%, for WPS/ULO and WPP/ULO in succession.

It could be observed that all the investigated cracked polymers produced n-paraffins in higher amounts than the isomers for the same carbon number. This can be attributed to the fact that the applied circumstances do not favor the reforming of the degraded molecules.

The emanated gases acquired from the cracked polymers offer considerable calorific values that permit their use as a fuel gas.

### 3.4. Evaluation of the cracked distillates:

The application of unconventional oxidative cracking was found to be a promising method for converting several waste polymers to more valuable products. The effectiveness of this technique in transforming the wastes of PS, PP and PE mixed with spent lube oil into lighter products can be clarified from the present study.

The investigated waste polymers and used lubricating oil (ULO) were degraded under experimental operating conditions at 260°C reaction temperature, 75 min residence time and at atmospheric pressure, using oxygen of air and lignin as a catalyst.

For comparison, a blank test was also performed for the mixed WPE and ULO at similar conditions, but in the absence of oxygen and catalyst, to deduce the influence of these two parameters on the properties of the obtained products.

The physicochemical properties of the investigated samples are exposed in Table (2). The depicted data reveal that the degraded WPS/ULO feedstock offered the largest magnitude of liquid products, with restricted amounts of gases and residue, accounting for 86%, 5.26% and 8.74% respectively.

**Table 2**: Physicochemical characteristics of the cracked liquids of WPS/ULO, WPP/ULO, WPE/ULO and blank WPE/ULO.

| Test | Method | WPS/ULO | WPP/ULO | WPE/ULO | Blank WPE/ULO |
|---|---|---|---|---|---|
| **Calorific value, Kj/Kg** | ASTM D-240 | 45156 | 46076 | 46432 | 45680 |
| **Density @ 15.56ºC**<br>**Specific gravity**<br>**API gravity** | ASTM D-1298 | 0.8705<br>0.8713<br>30.9 | 0.8065<br>0.8073<br>43.77 | 0.7806<br>0.7814<br>49.59 | 0.8263<br>0.8271<br>39.57 |
| **Total sulfur ,wt.%** | ASTM D-4294 | 0.27 | 0.33 | 0.34 | 0.09 |
| **Pour Point, ºC** | ASTM D-97 | -30 | 21 | 30 | -15 |
| **Flash Point, ºC** | ASTM D-93 | <-20 | <-20 | <-20 | >-20 |
| **Carbon Residue, wt.%** | ASTM D-527 | NIL | NIL | NIL | NIL |
| **Kinematic viscosity @40ºC,cSt** | ASTM D-445 | 1.19 | 2.65 | 3.01 | 3.64 |
| **Ash content, wt.%** | ASTM D-482 | NIL | NIL | NIL | NIL |
| **%CP**<br>**%CN**<br>**% CA** | D3238<br>ASTM D- | 52<br>10.92<br>37.08 | 71.59<br>19.92<br>8.49 | 82.31<br>10.38<br>7.31 | 46.85<br>10.89<br>42.26 |
| **Gases** | | **5.26%** | **9.80%** | **8.90%** | **13.80%** |
| **Liquid yeld (Condensate liquid in condenser)** | | **86%** | **77.40%** | **73.80%** | **58%** |
| **Residue** | | **8.74%** | **12.80%** | **17.30 %** | **28.20%** |

WPE: Waste of polyethylene, WPP: Waste polypropylene, WPS: Waste polystyrene, ULO: Used lubricating oil, API = American petroleum institute, %CP= paraffin chains, %CN= Naphthene ring structure, % CA= Aromatic ring structure

The dissociated distillates comprehend the highest concentration of aromatics in as much as >37%, added to approximately 11% naphthenes. This can be ascribed to the aromatic nature of the WPS feedstock, which is abundant in benzene rings. Therefore, the resulting liquid include only 52% paraffins, which considered as the minimum amount among the three examined polymers.

Concerning the cracked WPP/ULO products, data in Table (2) shed light on its ingredients, which were composed from liquid residue and gas in intermediate proportions between those recorded for degraded WPE and WPS. The proportions were 77.4% liquid and 12.8% residue, in addition to 9.8% gases.

On contrast, cracked WPE/ULO exhibited the least quantity of liquid yield, as it comprised 73.8%. This noticeable decrement is associated with the relative abundance of residue at the expense of the liquefied product, since they come to 17.8% respectively. Furthermore, the distillate of this sample consisted principally of aliphatic hydrocarbons. They represent more than 82% of the distillate, while aromatics and naphthenes make up only 7.3% and 10.38% respectively. This can be attributed to the paraffinic origin of this polymer.

From the reported information in Table (2), it can also be recognized that the WPE/ULO sample exhibits conspicuous advantages in terms of the amount of fluid yield arising from the oxidative degradation, which is higher than that of the blank sample. The fluid yield from the WPE/ULO sample comprised 73.8% compared to 58% for the blank sample. Meanwhile, there are considerable reductions in the amounts of residue, char, and evolved gases realized upon implementation of this type of cracking, compared to the uncatalyzed process. In addition, the data reveal a distinct increase in the aromatic content accompanied by an explicit reduction in the amount of paraffinic constituents in the assayed blank WPE/ULO sample.

More advantages are available from putting this oxidative cracking method into practice, as revealed in Table (2). The presented data expound the differences between the general specifications of the degraded WPE/ULO that was cracked with and without oxygen and catalyst.

It could be observed that there was an intelligible decrement of density and flash point, accompanied by a remarkable improvement in calorific value, aside from a limited decrease in kinematic viscosity of the oxidatively cracked WPE/ULO sample compared to those acquired from the blank test. The amelioration of all these properties may be ascribed to the autocatalytic hydrogenation occurring during the reaction [27]. However, a slightly higher percentage of sulfur content (amounting to 0.34 wt.%) was determined for the catalytically processed WPE/ULO polymer, against 0.09 wt.% achieved for the thermally cracked one. This may be imputed to the responsibility of sulfur derived from the spent lubricating oil, which incorporates a relatively high proportion of sulfur compounds, which reached 1.05wt. Table S6. It seems that the sulfur included in the appended ULO is probably dissociated thermally easier than in the presence of oxygen and /or the employed catalyst.

On the other hand, both samples are characterized by a lack of ash content and carbon residue. The aforementioned results reflect the substantial role of oxygen and lignin catalyst for upgrading the physicochemical properties of the yields obtained from the oxidatively cracked waste polymer, in comparison with those accomplished from the thermal dissociation method.

It is obvious that the data in Table (2) concerns the traits of distillates achieved from the degradation of the three inspected waste polymers. The recorded information elucidates certain variations in the physicochemical properties of the assayed samples. The depicted calorific values range from 46432 to 45156 kJ/kg, whereas the densities fluctuate between 0.7806 and 0.8705 at 15.56°C.

The highest calorific values, along with the lowest density, are related to the liquefied WPE/ULO polymer, which is characterized by the predominance of paraffinic hydrocarbons. In contrast, the worst results for both properties are found in WPS/ULO, which is rich in aromatics. The perceptible divergence can be attributed to the incongruity of the aliphatic and aromatic proportions in the constituents of the samples.

Moreover, the data manifest the direct relation between the increments of kinematic viscosity values, from 1.19 to 3.01 cSt, with increasing the paraffinic concentrations in the cracked waste polymers, which can be arranged in the following manner: WPE/ULO > WPP/ULO >WPS/ULO,

Reversely, the depression of pour points from 30°C to -30°C for WPE/ULO and WPS/ULO respectively can be attributed to the diminution of saturated paraffins associated with the prevalence of aromatics in the liquid accomplished from the degraded waste polymers, as confirmed from the FTIR spectra Figure1 and the data in Table (2).

Accordingly, it could be deduced that the quality of the processed products is mostly dependent on the constituents and the nature of the sample, whether it is generated from paraffinic or aromatic origin.

On the other side, it has been found that certain characteristics are common to all the examined distillates. These include the same flash points (< -20°C) along with the absence of both carbon residue and ash content. Nevertheless, limited concentrations of sulfur content were determined, ranging from 0.27 – 0.34 wt.%, which may have arisen from the contaminated ULO carrier.

### 3.5. Evaluation of the various fractions obtained from the investigated cracked waste polymers.

The oxidatively cracked WPE, WPP, and WPS feedstock, when mixed with ULO, was fractionated into three cuts with boiling ranges starting from ambient temperature to 180°C, 180- 280°C, and 280-380°C, which were classified as gasoline, kerosene, and gas oil, analogous to those derived from petroleum oil, respectively. The fraction boiling above 380°C was considered as residuals[29]. The various distillates were evaluated and compared with their corresponding fractions get out from petroleum crude.

#### *3.5.1 Evaluation of gasoline-like fraction obtained:*

Table (3) demonstrates the general physicochemical characteristics of gasoline prepared from the degraded waste polymers, along with those recorded for straight-run gasoline.

The exposed ASTM information indicates that the majority of the physicochemical properties, such as densities, flash points, copper corrosion, and kinematic viscosity values, as well as the lack of carbon residues and ash contents for the three produced gasolines, are almost identical to those achieved for the commercial petroleum fraction. Meanwhile, there are some improvements in the total sulfur content related to the gasoline acquired from dissociated WPS/ULO and WPE/ULO, which diminished to 0.04 wt.% and 0.02 wt.% respectively. However, a trivial increase to 0.06 wt.% has occurred for the sample arising from the degraded WPP/ULO, when compared to 0.05 wt.% pertaining to the natural gasoline.

It is also noticeable the considerable reduction of the determined gum content, which has minified to 0.63-0.53 wt.% instead of 1.0 wt.% that was depicted for the petroleum analogue.

Furthermore, the pour point of the sample derived from the cracked WPS/ULO is coincident with that of the petroleum counterpart (-30°C), whereas an explicit amelioration to -13°C was realized from the specimen derived from WPE/ULO. Nevertheless, an increment in pour point temperature to -15°C was accomplished for the gasoline-like product originated from WPP/ULO.

**Table 3:** Physicochemical characteristics of petroleum-like distillates obtained compared with petroleum refinery gasoline distillates.

| **Test** | **Method** | **WPS/ULO** | **WPP/ULO** | **WPE/ULO** | **Petroleum** |
|---|---|---|---|---|---|
| **Calorific value, Kj/Kg** | ASTM D-240 | 45359 | 47154 | 46810 | **49360** |
| **Density @ 15.56ºC** | ASTM D-1298 | 0.7381 | 0.7379 | 0.7451 | 0.725-0.748 |
| **Total sulfur, wt.%** | ASTM D-4294 | 0.04 | 0.06 | 0.02 | 0.05 |
| **Pour Point, ºC** | ASTM D-97 | -30 | -15 | -12 | -30 to -40 |
| **Flash Point, ºC** | ASTM D-93 | <-20 | <-20 | <-20 | - |
| **IBP, ºC**<br>**FBP, ºC** | ASTM D-86 | 45<br>178 | 53<br>180 | 50<br>180 | |
| **Carbon Residue, wt.%** | ASTM D-527 | NIL | NIL | NIL | NIL |
| **Kinematic viscosity @40ºC,cSt** | ASTM D-445 | 0.50 | 0.56 | 0.62 | 0.5-1 |
| **Ash content, wt.%** | ASTM D-482 | NIL | NIL | NIL | 0.1 |
| **Copper corrosion** | ASTM D-130 | 1a | 1a | 1a | 1a |
| Refractive index | ASTM D-1218 | 1.431 | 1.418 | 1.411 | 1.4100 |
| Gum content | ASTM D-381 | 0.531 | 0.621 | 0.633 | 1.00 |

IBP: Initial boiling point, FBP: Final boiling point, WPE: Waste of polyethylene, WPP: Waste polypropylene, WPS: Waste polystyrene, ULO: Used lubricating oil

On the other hand, the data presented in Table (3) elucidate that the magnitude of the calorific value of the natural gasoline, comprising 49360 Kj/kg, is somewhat higher than those measured for the prepared light fractions, as they possess values ranging between 45359 and 47154 Kj/kg, which can be categorized as follows:

WPP/ULO > WPE/ULO > WPS/ULO.

The variation in the calorific values of the three produced gasoline-like fractions may be imputed to the existence of partial olefines, naphthenes, and aromatics, specifically those derived from the degraded WPS/ULO polymer, as confirmed by the FTIR spectra Figure1.

Referring to the amounts of the lightest fraction produced from cracked polymers, and as deduced from the gas chromatographic data (Table 1) they can be arranged descendingly in the order of WPS/ULO> WPP/ULO > WPE/ULO.

Eventually, and on the basis of the aforementioned results, gasoline-like fractions derived from the various oxidatively cracked waste polymers under study are compatible with that produced from petroleum sources, as they have approximately the required specifications of natural gasoline.

### *3.5.2 Evaluation of kerosene-like fraction obtained:*

Concerning the kerosene-like that get from the cracked WPS/ULO, WPP/ULO and WPE/ULO Table (4) bring out the general characteristics of these distillates, along with those of their corresponding distillates derived from petroleum oil, for comparison.

**Table 4**: Physicochemical characteristics of petroleum-like distillates obtained compared with petroleum refinery kerosene distillates.

| **Test** | **Method** | **WPS/ULO** | **WPP/ULO** | **WPE/ULO** | **Petroleum** |
|---|---|---|---|---|---|
| **Calorific value, Kj/Kg** | ASTM D-240 | 45148 | 46414 | 45855 | 0.47220 |
| **Density @ 15.56oC**<br>**Specific gravity**<br>**API gravity** | ASTM D-1298 | 0.8397 | 0.7895<br>0.7903<br>47.54 | 0.8277 | 0.775-0.840 |
| **Total sulfur ,wt.%** | ASTM D-4294 | 0.15 | 0.12 | 0.13 | 0.3 |
| **Pour Point, ºC** | ASTM D-97 | -30 | -12 | -12 | -40 to -47 |
| **Flash Point, ºC** | ASTM D-93 | 50 | 58 | 55 | 58 |
| **IBP, ºC**<br>**FBP, ºC** | ASTM D-86 | 183<br>233 | 185<br>227 | 180<br>234 | |
| **Carbon Residue, wt.%** | ASTM D-527 | NIL | NIL | NIL | 1.5 |
| **Kinematic viscosity @40ºC,cSt** | ASTM D-445 | 1.25 | 1.25 | 1.32 | 1.5 |
| **Ash content, wt.%** | ASTM D-482 | NIL | NIL | NIL | NIL |
| **Copper corrosion** | ASTM D-130 | 1a | 1a | 1a | 1a |
| **Smock point** | **ASTM D-1322** | 33 | 34 | 34 | 34 |
| **Refractive index** | **ASTM D-1218** | 1.4460 | 1.4453 | 1.4473 | 1.4458 |

WPE: Waste of polyethylene, WPP: Waste polypropylene, WPS: Waste polystyrene, ULO: Used lubricating oil, API = American petroleum institute, IBP: Initial boiling point, FBP: Final boiling point, The submitted information brings to light that the properties of the prepared kerosene seem to be in agreement with those of petroleum analogue. The kerosene is devoid of carbon residues and ash content, has suitable densities, and exhibits the same result of copper corrosion test as typical kerosene. Meanwhile, it displays certain improvement with regard to the sulfur content, which dropped from 0.3 wt.% to the range of 0.12-0.15 wt.%, as well as a reduction in the kinematic viscosity values, which decreased to 1.25-1.32 cSt compared to 1.5 cSt for the natural kerosene.

Nevertheless, the produced specimens reveal some elevation in pour point temperatures that attained the range of -30°C to -12°C particularly for those get from cracked WPE/ULO and WPP/ULO. This may have ascribed to the paraffinic nature of the original feedstock. Moreover, the prepared kerosene has somewhat lower calorific values, ranging from 45148-46414 cSt, compared to the corresponding straight-run petroleum fraction that comprised 47220 Kj/kg. This is mainly imputed to the presence of some olefins and aromatics, as detected from the FTIR spectra (Fig1), particularly with that received from WPS/ULO sample, which exhibits the smallest calorific value amongst the acquired distillates in as much as 45148 Kj/ kg.

Remembering that, as set forth from the gas chromatographic data, the kerosene yield derived from degraded WPE/ULO and WPP/ULO are nearly similar, with percentages amounting to about 26 wt.%, whereas the cracked WPS/ULO sample suffers from the scarcity of this fraction, since it represents only less than 3 wt.%. Therefore, the kerosene-like fuel ensued from the dissociation of the investigated waste polymers in this study has properties almost in conformity with those of the corresponding cut generated from petroleum oil.

### 3.6. Evaluation of the gas oil-like fraction obtained.

The gas oil-like products drawn out from the oxidative dissociation of the three scrutinized waste polymers were also subjected upon the tapis. From the analytical gas chromatographic data in Table 1, these fractions incorporate paraffinic hydrocarbons with magnitudes come to 24.4 wt.%, 16.6 wt.% for WPE-ULO and WPP-ULO, successively, whereas the least amount, equivalent to 7.3 wt.%, was donated from WPS-ULO.

The physicochemical features of the prepared gas oils were determined, and their data are exposed in Table (5). The propounded information established that the produced distillates have entirely identical results for the copper corrosion test, along with the absence of carbon residues and ash contents, as those reported for the typical gas oil, while the assigned densities are within the range.

**Table 5**: Physicochemical characteristics of petroleum-like distillates obtained compared with petroleum refinery gas oil distillates.

| Test | Method | WPS/ULO | WPP/ULO | WPE/ULO | Petroleum |
|---|---|---|---|---|---|
| **Calorific value, Kj/Kg** | ASTM D-240 | 44880 | 45792 | 45809 | 44300 |
| **Density @ 15.56ºC** | ASTM D-1298 | 0.8531 | 0.8610 | 0.8593 | 0.8385-0.87 |
| **Total sulfur ,wt.%** | ASTM D-4294 | 0.27 | 0.33 | 0.25 | 1.174 |
| **Pour Point, ºC** | ASTM D-97 | -24 | -12 | -15 | 0 |
| **Flash Point, ºC** | ASTM D-93 | 56 | 61 | 68 | |
| **IBP, ºC**<br>**FBP, ºC** | ASTM D-86 | 233<br>350 | 230<br>350 | 235<br>350 | 250<br>350 |
| **Carbon Residue, wt.%** | ASTM D-527 | NIL | NIL | NIL | NIL |
| **Kinematic viscosity @40ºC,cSt** | ASTM D-445 | 3.98 | 4.11 | 4.33 | 5.4 |
| **Ash content, wt.%** | ASTM D-482 | NIL | NIL | NIL | NIL |
| **Copper corrosion** | ASTM D-130 | 1a | 1a | 1a | 1a |
| **Refractive index** | **ASTM D-1218** | 1.47 | 1.469 | 1.477 | 1.462 |

WPE: Waste of polyethylene, WPP: Waste polypropylene, WPS: Waste polystyrene, ULO: Used lubricating oil, IBP: Initial boiling point, FBP: Final boiling point

However, upgrades of certain properties, such as pour paint, kinematic viscosity, calorific values, and sulfur content have been detected.

It is noticeable that the pour points were dropped from zero to the range of -12°C to -15°C with WPE/ULO and WPP/ULO samples, those derived from paraffinic feedstocks, and a more significant depression took place to -24°C for that ensued from aromatic source, (i.e.WPS/ULO). So the decrease of pour points is related to the proportions of saturated paraffins included in the samples. Thereby, it is plausible to attribute the responsibility of the paraffinic concentration of the examined distillates on their obtainable pour point values.

Meanwhile, the improvement of the kinematic viscosities via the diminution of their magnitudes was recognized for the gas oil prepared from the WPS/ULO polymer, compared to the two other distillates. This can be ascribed to the relative abundance of aromatics on account of the paraffinic constituents of this sample.

In contrast, the significant increase in the calorific values of the examined specimens is noteworthy, as they reached a range of 44880 - 45809 kJ/kg compared to 44 300 kJ/kg for petroleum gas oil. The degree of calorific value improvement for this fraction derived from various cracked waste polymers can be categorized in descending order as follows:

WPE/ULO >WPP/ULO >WPS/ULO.

The enhancement in calorific value can be attributed to the increased saturation of paraffins in the samples, along with the auto-hydrogenation of degraded products during the reaction, which appears to be unparalleled in the saturation of petroleum products.

Finally, the data reflect a significant reduction in total sulfur content from 1.17 wt. % to a range of 0.25-0.33 wt. % for the natural gas oil and the corresponding prepared fractions, respectively. This reduction can be attributed to the sulfur content in the original feedstock of each specimen.

Minifying the sulfur concentration of the obtained yields conveys a dropping of the emitted sulfur trioxide generated during the oil combustion. This sequel is concordant with the imperative restricted value for the emitted hazardous gases.

Modification of all the aforementioned characteristics can be ascribed to the in situ upgrading of the dissociated feedstock, which takes place during the course of the oxidative cracking process.

As for the excited results, the gas oil obtained from the degradation of WPE/ULO, WPP/ULO, and WPS/ULO has similar properties or may be better than that acquired from petroleum crude oil.

## 4. **Conclusion**

The unconventional oxidative cracking technique was modified and utilized to convert three waste polymers (WPE, WPP, and WPS) and a ULO carrier to more valuable products. The results demonstrated that the largest magnitude of gasoline can be attained from the dissociated WPS/ULO product, as it constitutes approximately 90 wt.% of the cracked sample, On the other hand, 52.3 wt.% of the prepared gasoline-like is available in the liquefied WPP/ULO, whereas the least quantity of this fraction (42.3 wt.%) is accessible in the degraded WPE/ULO. The distillates analogous to kerosene, the recorded data reveal that this fraction attained from the degraded WPP/ULO and WPE/ULO is approximately similar in amounts, comprising about 26-25 wt.%.On contrast, the product received from WPS/ULO cracking is characterized by the scarcity of this cut, as it represents 2.6 wt.% from the yield .the constituents of the heavier fraction petroleum gas oil the quantities of these components are equal to 16.6 and 20.8wt.% for degraded WPP/ULO and WPE/ULO, successively, whereas the lowest value of 7.4 wt.% was derived from the cracked WPS/ULO.. The obtained hydrocarbons i.e. gasoline, kerosene, and gas oil–like fractions, have the same advantages as being usable as substitutes for the fossil fuel. Gas chromatographic analysis has shown that the liberated gases during the cracking process are mainly composed of combustible hydrocarbon gases. The FTIR data show that the cracked products contained no oxygenated compounds, despite the use of air in the process. Conspicuous advantages connected with the amount of liquid yield arose from dissociative WPE/ULO, which is higher than that received from the blank test, aside from the considerable diminution of residue, char, and evolved gasses. Furthermore, the decrement of density and viscosity, and the improvement in CV value of the oxidatively cracked WPE/ULO compared to the thermally dissociated one (blank), can be attributed to the autocatalytic hydrogenation occurring throughout the reaction. This reflects the substantial role of O2 and the lignin catalyst for upgrading the properties of the obtained yield. Fractionation of the liquefied polymers produces fuels that have almost the same characteristics as the corresponding petroleum fractions.

## 5. Conflicts of interest

"The authors declare that they have no competing interests"

## 6. Formatting of funding sources

The present investigation has not been funded for all experiments and analyses, including publishing.

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