



Thermal and Catalytic Pyrolysis of Plastic Waste: Catalysts Characterization and Properties of the Liquid Products

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Abstract

Nowadays, sustainable energies can be found by thermal and catalytic pyrolysis of plastic waste. This study uses high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET) to investigate thermal and catalytic pyrolysis using a stainless-steel semi-batch reactor at different conditions: Temperatures (350 - 500°C) and residence times (60, 90, and 120 min). The catalysts were undergone to different characterization techniques such as EDX, SEM, and BET that revealed the PR.9 catalyst consists of Cobalt, Molybdenum, and Platinum loaded on Al₂O₃. Whereas, the chemical compositions of Platinum RG-412 catalyst contain Chlorine and Platinum. The SEM analysis showed that the PR.9 catalyst is more active than RG-412 for the degradation of plastics. In addition, the properties of the liquid products like density, API, and viscosity were studied. As a result, the properties of the liquid produced by the plastic pyrolysis were similar that those produced from the petroleum refineries. The API findings declared that almost all of the liquids produced were light products, which contain mainly diesel oil, gas oil, kerosene, and gasoline.

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1. Introduction

Nowadays, the demand for plastic products rises as countries' economies develop, the worldwide plastics production almost reached 370 million tons. So with modern techniques, sustainable energies can be found by thermal and catalytic pyrolysis of plastic waste [1, 2]. The researches in this field are many [3-10]. Chemical recycling, as numerous research references suggest, should be a viable solution to environmental issues. Polymer wastes are the primary cause of these problems (plastics, biomass, *etc.*). Because of its wide temperature range, pyrolysis is a useful methodology (300 - 1000°C). Many different raw materials and catalysts might be employed in different reactors. Because of their composition and properties, the most apparent use for pyrolysis products is energy consumption. On the other hand, during these processes, determining the pollutant content and determining the optimal product-yield structure is a huge challenge. The raw material might be optimized to increase both the quantity and quality of the product. Catalysts or other therapies can help achieve this goal (e.g. catalytic hydrogenation, reforming, *etc.*) [11]. In thermal pyrolysis, Miandad et al. studied different PW effects on the yield and quality of the produced liquid in the thermal pyrolysis process using a small pilot-scale reactor at 450°C and

75 min and found that PS showed maximum liquid yield with a small number of gases and char [12]. Thahir et al. studied temperature effects and optimized liquid oil produced in pyrolysis of the PP PW using a fixed bed reactor at 500–650°C. They found that optimum yields of liquid oil and gas were 88 wt% and 5 wt%, respectively with the rest being char at 580°C [13]. In general, advantages of thermal cracking can be summarized as follows: using the product as a fuel alternative, and wax in steam cracking, while disadvantages are high-temperature requirements, long residence time required, wide-range product distribution, high manufacturing costs, and poor quality of products [14, 15]. In the catalytic pyrolysis, Claudinho & Ariza used catalytic degradation of the residual PET as a recycling technique for new valuable chemical products at different temperatures of 370–507°C and ratio proportions of the calcium hydroxide [10]. Diaz-Silvarray et al. investigated operating conditions such as temperature (450–600°C), residence time (20 sec), and catalyst to plastic ratio and their effects on the yields and properties of the product. they showed that benzoic acid was recovered in PET pyrolysis and found using a catalyst with different ratios, the oil yield was not promising [16]. Mangesh et al. used and compared four plastics in catalytic pyrolysis types to convert to liquid fuel. They investigated the behavior of each one and tested its physicochemical properties [17]. In general, the advantages of catalytic cracking can be summarized as follows: lowering residence time, reaching 100% conversion, reducing the net process costs, improving the quality of the products obtained, and the catalyst reducing the reaction temperature while the disadvantage is increasing the gaseous products when using some types of catalysts [18, 19]. We expanded over these studies by characterizing, such as the EDX, SEM, and BET surface area using different catalysts in plastic pyrolysis to observe which catalyst is more suitable for the pyrolysis process, as well as studying the properties of the liquid products like API gravity and viscosity.

2. Materials and Methods

HDPE, LDPE, PS, and PP were purchased from the local market, while PET was afforded as water bottles from a local company. The polymers were in the shape of pellets with an average particle size of 0.1 – 0.2 cm while the PET bottles were shredded and sieved into a size of 0.6 mm. Two types of commercial spent catalysts (Ketjenfine PR.9 and Platinum RG-412) have been utilized and obtained from the Iraqi Ministry of Oil/Midland Refineries Company MRC/Daura Refinery – Naphtha refining and Kerosene hydrogenation units, Baghdad, Iraq. Tables 1 and 2 show the properties of PR.9 and RG-412 respectively.

Table 1: Properties of the PR.9 catalyst.

Property	Value
MoO ₃ content (wt%)	12
CoO content (wt%)	4
Platinum content (wt%)	0.25
Re content (wt%)	0.25
Diameter (mm)	1.6
Surface Area (m ² /g)	270 – 280
Pore Volume (ml/g)	0.5 – 0.55
Average length (mm)	5
Density (Kg/m ³)	740

Table 2: Properties of the RG-412.

Property	Value
Platinum content (wt%)	0.35
Chlore content (wt%)	1
Diameter (mm)	1.8
Surface Area (m ² /g)	215
Total Pore Volume (m ³ /g)	0.57
Average length (mm)	5
Sock Loading Density (Kg/L)	0.66

A 1.5 L cylindrical stainless-steel semi-batch reactor with 10.8 cm internal diameter, 12 cm external diameter, and a height of 15 cm makes up the thermal pyrolysis setup. It also contains two pipes and a flange that connects to the top of the reactor and is sealed with metal bolts. The determined amount of nitrogen is delivered to the reactor

through one pipe, while the condensate products are collected through the other. Heating is provided by a 20 KW electric heating mantle, with the temperature being measured by a K-type thermocouple (0 - 600°C) fixed inside the reactor and controlled by a PID controller. To cool the condensing vapors generated from the reactor, a two-stage glass condenser cooled by chilled water was attached to the reactor. A stainless steel catalyst bed with a height of 6 cm and a mesh diameter of 0.1 cm was fixed with the inner diameter of the reactor in catalytic pyrolysis. Additionally, the weight of each plastic type fed into the reactor is 250 gm.

Table 3: Experimental thermal cracking work.

Exp. No.	Feed type	Temp. (°C)	Time (min)
1	HDPE	350	60
2		400	120
3		450	90
4		500	120
5	PS	400	120
6		450	120
7		500	120
8	LDPE	400	60
9		450	120
10		500	120
11	PP	400	60
12		500	120

Table 4: Experimental catalytic cracking work.

Exp. No.	Feed type	Catalyst type	Catalyst to polymer ratio (wt%)	Temp. (°C)	Time (min)
2	HDPE	PR.9	10	400	120
3		RG-412	10	450	120
4		PR.9	5	500	120
5	PS	PR.9	10	400	120
6		RG-412	15	450	120
7		PR.9	5	500	90
8	LDPE	PR.9	15	400	120
9		RG-412	10	450	120
10		PR.9	5	500	90
11	PP	PR.9	5	500	60

Density is a physical property of a material that can be defined as the mass of any substance divided by its volume. The density of the produced liquid was determined at a temperature of 25 °C using a 25 ml pycnometer with an inserted thermometer. The API gravity is defined as a measurement of how heavy or light liquid petroleum is concerning water, the lighter the compound, the greater the API gravity. It was measured as below [20]:

$$\text{Density} = \frac{\text{Mass of pycnometer with Liq. (gm)} - \text{Mass of empty pycnometer (gm)}}{\text{Volume of the pycnometer (ml)}} \quad (1)$$

$$\text{API} = \left[\frac{141.5}{\text{Specific Gravity}} \right] - 131.5 \quad (2)$$

The dynamic viscosity of liquid samples was obtained at a temperature of 43°C and a speed of 10 RPM using the BROOKFIELD RHEOMETER DV3T instrument while the kinematic viscosity was determined by dividing the dynamic viscosity on the density of the same sample according to the following equation [20]:

$$v = \frac{\mu}{\rho} \quad (3)$$

Where, v is the kinematic viscosity in cSt, μ is the dynamic viscosity in cP (mPa.s), and ρ is the density in kg/m³. The Scanning Electron Microscope (SEM) is a type of electron microscope that scans the surface with a focused beam of electrons to create sample images. Scanning electron micrographs of the PR.9 and RG-412 catalyst samples were taken by the TESCAN MIRA-3 microscope, which operated at an accelerating voltage of 30 kV. It is worth mentioning that Energy Dispersive X-Ray Analysis (EDX) has been done by the same instrument.

3. Results and Discussion

3.1. Catalyst Characterization

The PR.9 and RG-412 catalysts were characterized by Energy Dispersive X-Ray Analysis (EDX), Scanning Electron Microscope (SEM), and BET surface area as follows:

3.1.1 Energy Dispersive X-Ray Analysis (EDX)

Figures 1 and 2 represent the scanning electron image and chemical compositions of SEM analysis for PR.9 respectively. It can be observed from the figures and Table 5 that the PR.9 catalyst consists of Cobalt, Molybdenum, and Platinum loaded on Al₂O₃, which indicates that the analysis confirmed the properties of this catalyst given by the Ministry of Oil/ MRC/Al-Daura Refinery. Whereas, Figures 3, and 4 show the scanning electron image and chemical compositions of EDX analysis for RG-412 respectively. Table 6 shows the chemical compositions of Platinum RG-412 catalyst, which contains Chlorine and Platinum as confirmed by Al-Daura Refinery.

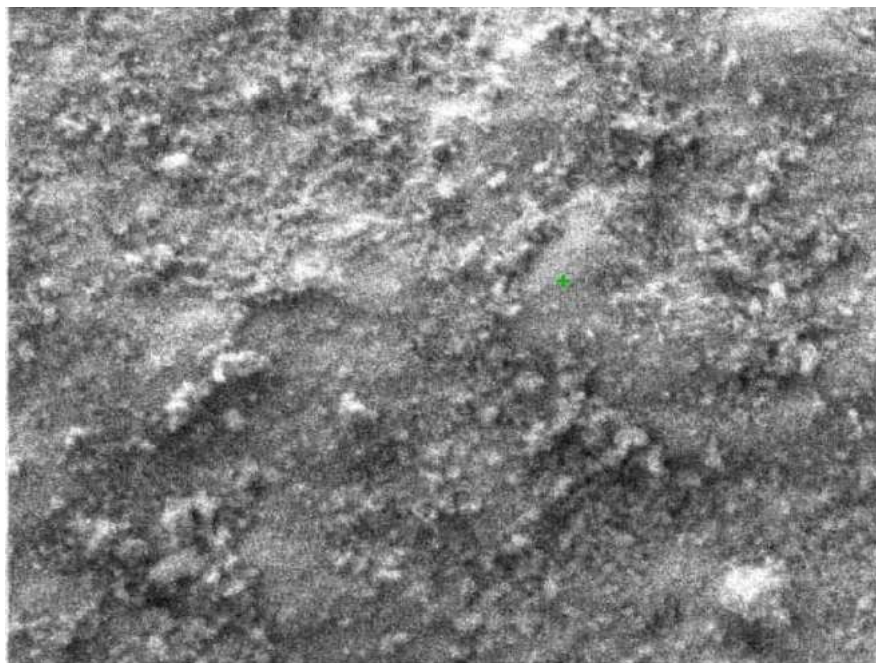


Figure 1: Electron image of EDX Analysis for PR.9 catalyst.

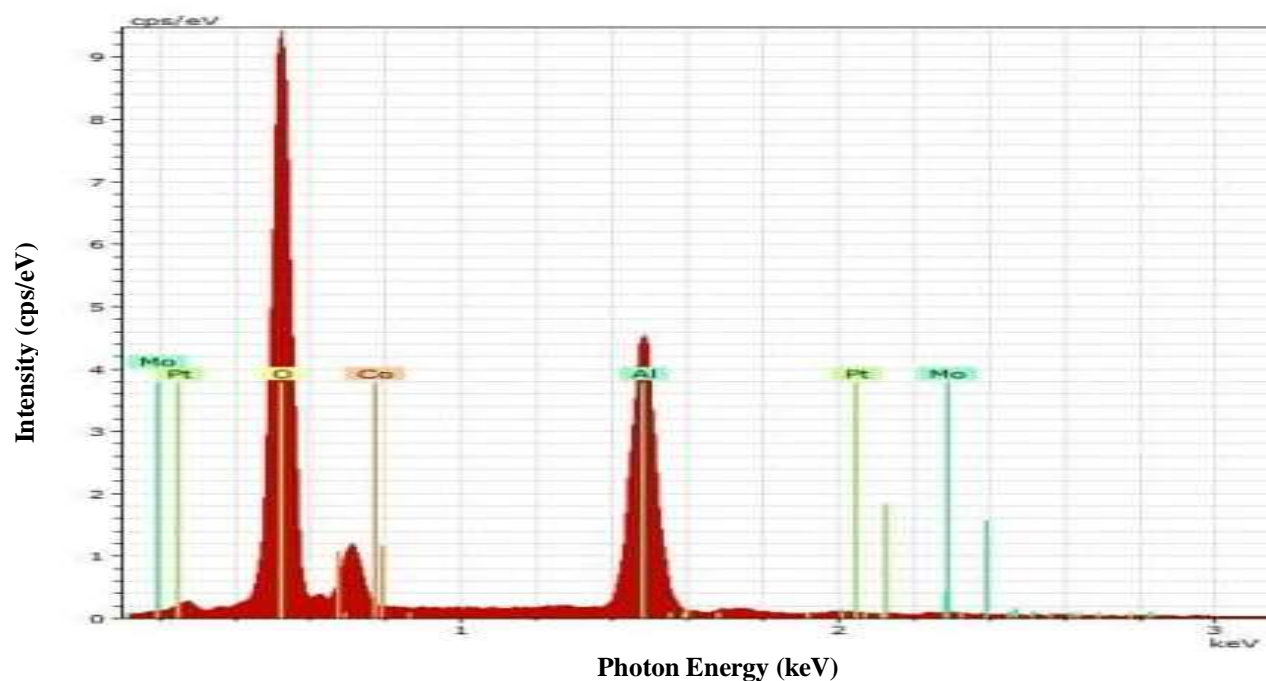


Figure 2: EDX Analysis for PR.9 catalyst.

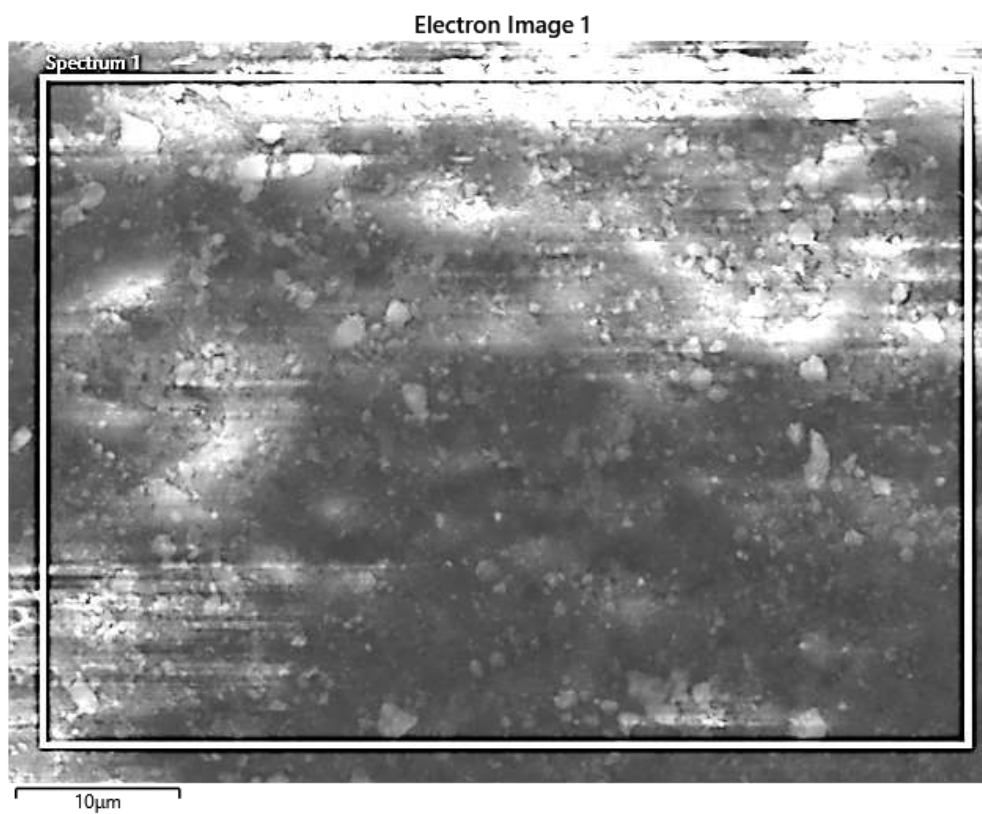


Figure 3: Electron image of EDX Analysis for RG-412 catalyst.

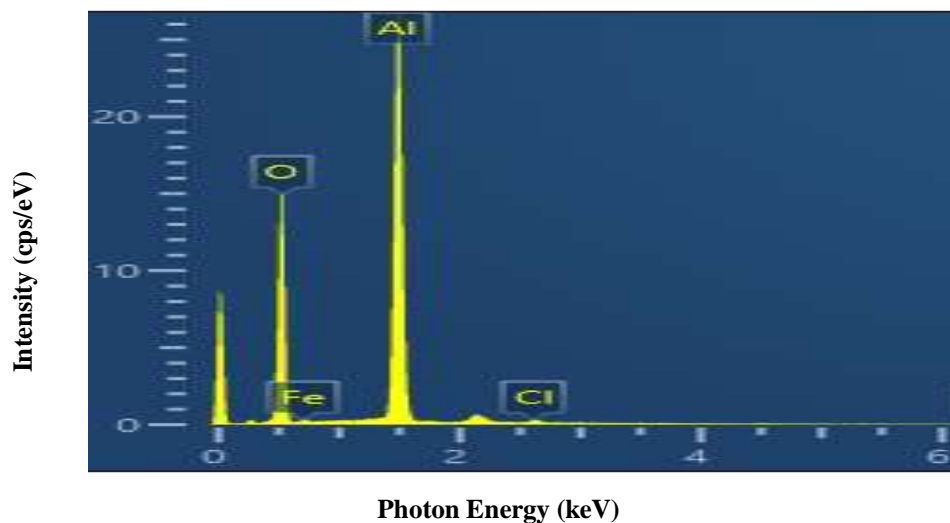


Figure 4: EDX Analysis for RG-412 catalyst.

Table 5: Chemical compositions of PR.9 catalyst.

Element	Line type	Weight %	Weight % Sigma	Atomic %
O	K series	58.57	4.43	72.67
Al	K series	34.45	0.95	25.34
Co	L series	4.27	0.68	1.44
Mo	L series	2.54	0.22	0.52
Pt	M series	0.18	0.06	0.02
Total		100.00		100.00

Table 6: Chemical compositions of RG-412 catalyst.

Element	Line type	Weight %	Weight % Sigma	Atomic %
O	K series	57.49	0.21	69.95
Al	K series	40.57	0.21	29.27
Fe	K series	1.43	0.11	0.50
Cl	K series	0.51	0.05	0.28
Total		100.00		100.00

3.1.2 Scanning Electron Microscope (SEM)

The morphological structure of the catalysts samples was obtained using this technique. Figure 5 shows the SEM images of the spent Ketjenfine PR.9 catalyst. From Image (a) in the figure below, it can be seen that the largest particle with average dimensions is 40 microns. As for other particles, they are less than one micron. This is evident in Image (b), where it was discovered that particles smaller than one micron were stuck together, which is also known as agglomeration. When these clusters were zoomed in the Image (c) below, it was found that they were nanoparticles, with sizes ranging from 30 to 40 nanometers, according to the measurements in the picture below. Figure 6 presents the SEM images of the spent Platinum RG-412 catalyst. In this Figure, image (c) presents the particles' sizes between 40 to 46 nm, which were larger than PR.9 catalyst; that means it contains more coke on its surface area.

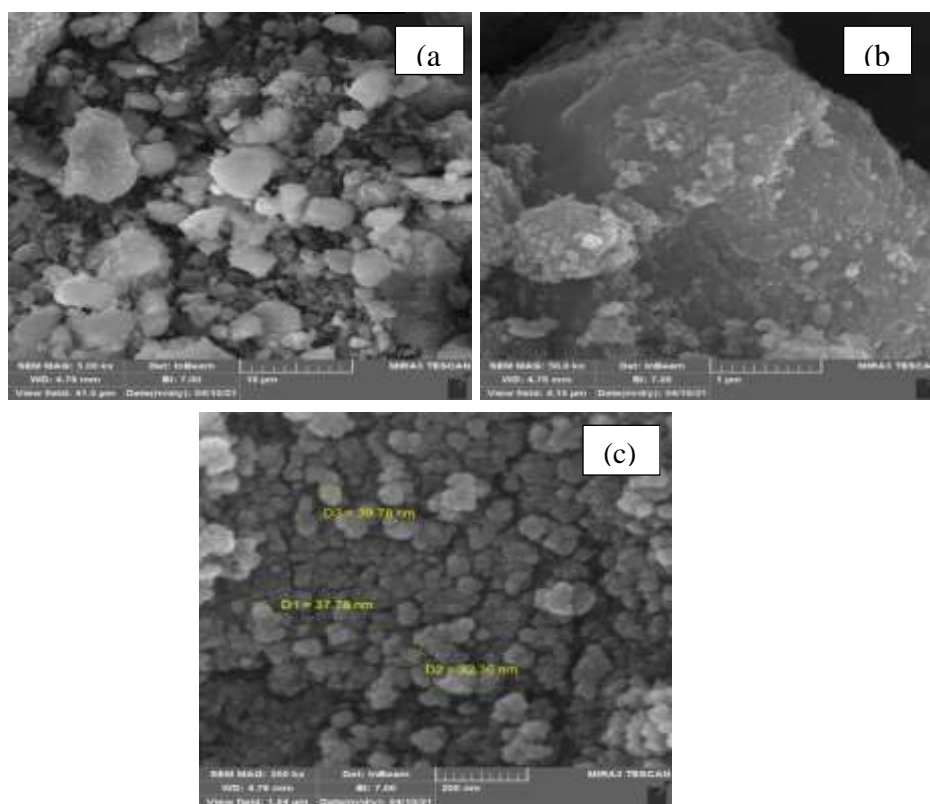


Figure 5: SEM images of spent Ketjenfine PR.9 catalyst.

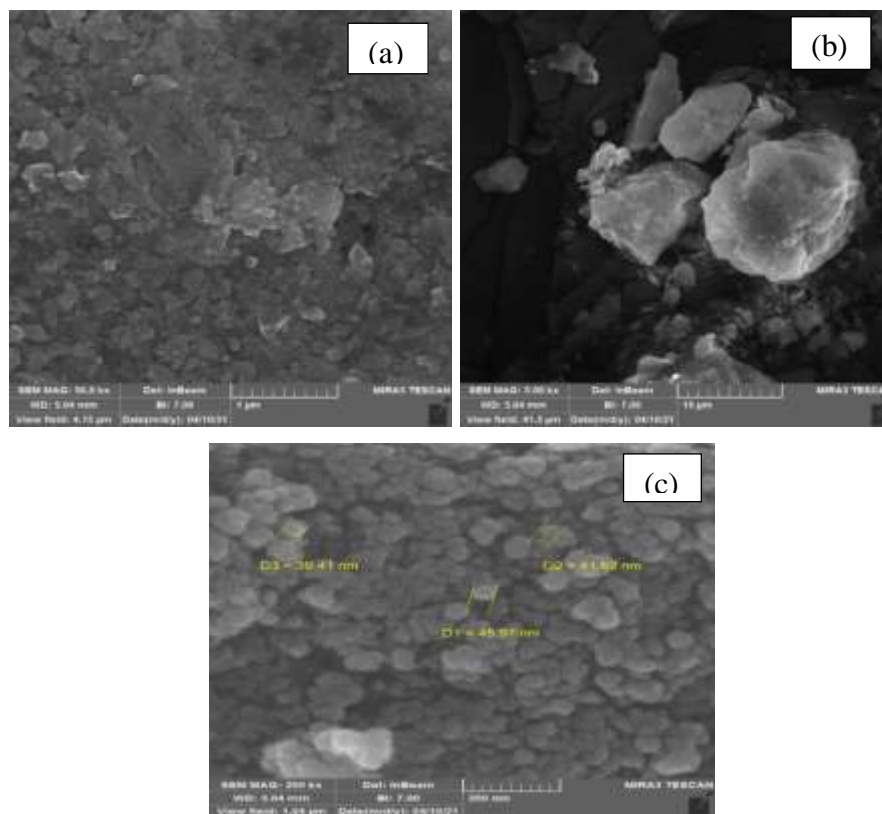


Figure 6: SEM images of spent Platinum RG-412 catalyst.

3.1.3 BET Surface Area

The surface area of the catalysts used in this study was measured using the Brunauer Emmett Teller (BET) method. From Table 7, the test showed that the surface areas of the spent Ketjenfine PR.9 and Platinum RG-412 catalysts were 249.68 and 210.345 m²/g respectively, while the fresh ones as reported in chapter three were 275 and 215 m²/g respectively.

Table 7: BET surface area of fresh and spent Ketjenfine PR.9 and Platinum RG-412 catalysts.

Catalyst Name	Surface Area (m ² /g)
Fresh Ketjenfine PR.9	270 – 280
Spent Ketjenfine PR.9	249.68
Fresh Platinum RG-412	215
Spent Platinum RG-412	210.345

3.2 Physical Properties of Liquid Produced

3.2.1 Density and API Gravity

The density and API gravity of the liquid oil produced from thermal and catalytic pyrolysis were measured and presented in Table 8. It can be observed from the results below that when the temperature increases, the density of liquid oil decreases. It is worth mentioning that the Iraqi fuel gasoline, diesel, kerosene, and gas oil densities are 0.733, 0.902, 0.787, and 0.83 gm/ml respectively. It can be seen that the liquid oil products of plastics approximately have the same values of density as the Iraqi fuel oil. According to the API results of thermal and catalytic cracking, it has been noticed that the API values have increased in the case of catalytic cracking in comparison with thermal cracking for HDPE and PS plastic types, indicating that the catalyst played a key role in improving the quality of the liquid oil produced, Figure 7. However, it decreased for LDPE and stayed the same for PP. Moreover, as a general comparison between the results of API gravity presented below, which ranges from 20° to 70° and the API gravity of crude oil, which for light crude oil is greater than 38°, for heavy crudes is less than 22°, while the API gravity of intermediate crudes ranges from 22° to 38° [21]. This means that almost all of the liquids produced from plastic waste were light products. Furthermore, for a specific comparison with values of the Iraqi fuel API gravity are 25.3°, 39.1°, 48°, and 61.4° for diesel oil, gas oil, kerosene, and gasoline respectively. It can be seen that these values are within the range of the results presented below, which means that the liquid produced from plastics contains mainly diesel oil, gas oil, kerosene, and gasoline.

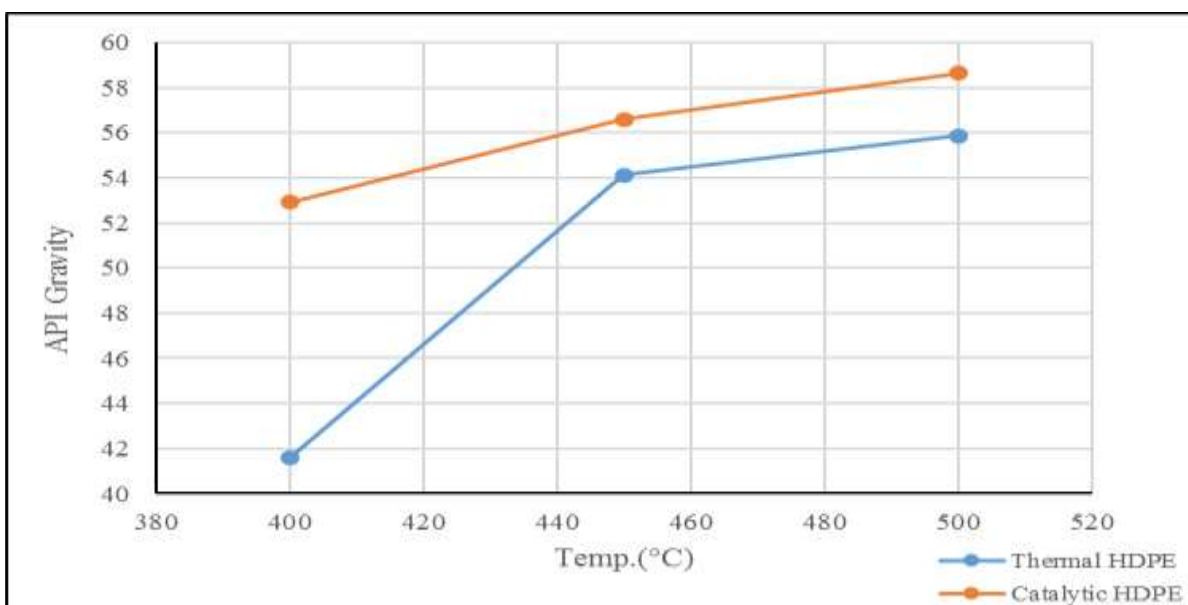


Figure 7: The API gravity of HDPE for both thermal and catalytic cracking.

Table 8: The density and API gravity of the liquid produced through thermal and catalytic pyrolysis.

Plastic Type	Catalyst Type	Temperature (°C)	Density (gm/ml)	API gravity
HDPE	-	400	0.815	41.59
HDPE	-	450	0.76	54.12
HDPE	-	500	0.753	55.85
LDPE	-	400	0.71	67.19
LDPE	-	450	0.75	56.6
LDPE	-	500	0.75	56.6
PS	-	400	0.922	21.51
PS	-	450	0.92	21.84
PS	-	500	0.9	25.25
PP	-	400	0.735	60.43
PP	-	500	0.75	56.6
HDPE	P.R.9	400	0.765	52.91
HDPE	R.G.412	450	0.75	56.6
HDPE	P.R.9	500	0.742	58.63
LDPE	P.R.9	400	0.76	54.12
LDPE	R.G.412	450	0.755	55.35
LDPE	P.R.9	500	0.753	55.85
PS	P.R.9	400	0.91	23.52
PS	R.G.412	450	0.9	25.25
PS	P.R.9	500	0.89	27.01
PP	P.R.9	500	0.75	56.6

3.2.2 Viscosity

The viscosity of liquid oil depends on several factors, such as temperature, feedstock type, operating conditions, *etc.* As long as the viscosity is low, the liquid will be easier to transport and handle. Table 9 summarizes the dynamic and kinematic viscosities of liquid oil products obtained from thermal and catalytic pyrolysis of HDPE, LDPE, PP, and PS. In this work, the viscosity of oil produced from different types of plastics has been determined at a temperature of 43°C and it has been observed that it was in the range between (4 - 10 cSt). In general, the standard kinematic viscosity of fuel oil is in the range between (3.5 - 9.7 cSt), which means that the products of this research were found to be suitable to use as a fuel depending on the similarity between the viscosities. It is important to mention that different additives were employed for strength, color, and as a catalyst in the polymerization process. However, these additives are considered impurities in plastic waste, which are also present in the oil produced. Therefore, further treatment should be taken into consideration to remove these contaminants such as sulfur, chlorine, solid residue, moisture, and acids since it reduces the quality of liquid oil and restricts its commercial usage. Tables 10 and 11 represent products weights of both thermal and catalytic cracking.

Table 9: Dynamic and kinematic viscosities of liquid products.

Plastic Type	Catalyst Type	Temperature (°C)	Dynamic Viscosity (cP)	Kinematic Viscosity (cSt)
HDPE	-	400	6.6	8.1
HDPE	-	450	7.63	10.04
HDPE	-	500	18	23.9
LDPE	-	400	2.78	3.91
LDPE	-	450	5.9	7.86
LDPE	-	500	7.63	10.17
PS	-	400	5.55	6.02

Cont'd Table 9.

PS	-	450	6.94	7.54
PS	-	500	6.59	7.32
PP	-	500	7.98	10.64
HDPE	P.R.9	400	7.28	9.51
HDPE	R.G.412	450	6.59	8.78
HDPE	P.R.9	500	6.94	9.35
LDPE	P.R.9	400	6.59	8.67
LDPE	R.G.412	450	6.24	8.26
LDPE	P.R.9	500	6.59	8.75
PS	P.R.9	400	8.33	9.15
PS	R.G.412	450	7.63	8.5
PS	P.R.9	500	6.24	7.01
PP	P.R.9	500	7.28	9.7

Table 10: Thermal cracking products weights.

Run	Feed type	Feed weight (gm)	Temp. (°C)	Time (min)	Weight of liq. oil (gm)	Weight of wax (gm)	Weight of gases (gm)	Weight of solid residue (gm)
1	HDPE	500	350	60	15.19	0	17.81	467
2		250	400	120	13.63	38.67	21.7	176
3		500	450	90	57.78	7.12	54.76	380.34
4		250	500	120	54.3	73.21	77.5	45
5	PS	250	400	120	186.32	3	45.68	15
6		250	450	120	199.1	37.8	0	13.1
7		300	500	120	277.25	0	20.75	2
8	LDPE	250	400	60	55.14	0	8.86	186
9		250	450	120	190.13	21.3	36.57	2
10		250	500	120	211.75	30	7.25	1
11	PP	250	400	60	0	9	20	221
12		250	500	120	103.4	45	80.6	21

Table 11: Catalytic cracking products weights.

Run	Feed type	Feed weight (gm)	Catalyst type	Catalyst to polymer ratio (wt%)	Temp. (°C)	Time (min)	Weight of liq. oil (gm)	Weight of wax (gm)	Weight of gases (gm)	Weight of solid residue (gm)
1	PET	100	Ca(OH) ₂	200	500	60	10.71	12.87	26.42	250
2	HDPE	250	P.R.9	10	400	120	170.28	0	47.5	32.22
3		250	R.G.412	10	450	120	120	0	44.67	85.33
4		250	P.R.9	5	500	120	83	0	47	120

Cont'd Table 11.

5	PS	250	P.R.9	10	400	120	202.1	10.4	32.5	5
6		250	R.G.412	15	450	120	190.5	11.1	34.42	14
7		250	P.R.9	5	500	90	200.9	3.1	28.96	17.16
8	LDPE	250	P.R.9	15	400	120	212.3	0	29.26	8.46
9		250	R.G.412	10	450	120	205.11	4.2	35.29	5.6
10		250	P.R.9	5	500	90	226.13	2	20.17	1.7
11	PP	150	P.R.9	5	500	60	20.46	18.5	41.54	70

3. Conclusions

In this study, promising and relatively high liquid yields were obtained from the thermal and catalytic cracking of different types of plastics in a laboratory-scale stainless-steel semi-batch reactor. During the catalytic cracking, the platinum RG-412 catalyst didn't have a major impact on the liquid yield. Therefore, Ketjenfine PR.9 was found to be more appropriate as a catalyst for the degradation of plastics. Although it was spent, it reduced the residence time, the reaction temperature, and the amount of wax formed while improving the liquid oil's yield. The viscosity results confirmed that the oil products contained a range of aromatics, aliphatic, and other oxygenated hydrocarbon compounds, which are considered light oils. The liquid oil products of plastics approximately have the same values of density and viscosity as the Iraqi fuel oil. According to the API results of thermal and catalytic cracking, it has been noticed that the API values have increased in the case of catalytic cracking in comparison with thermal cracking for HDPE and PS plastic types, indicating that the catalyst played a key role in improving the quality of the liquid oil produced.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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