Al-Khwarizmi Engineering Journal, Vol. 15, No. 2, June , (2019)
P.P. 100- 107

Al-Khwarizmi
Engineering
Journal

Treatment of Waste Extract Lubricating Oil by Catalytic Cracking Process to Produce Light Fractions

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(Received 22 October 2018; accepted 10 January 2019)
https://doi.org/10.22153/kej.2019.01.003

Abstract

The catalytic cracking of three feeds of extract lubricating oil, that produced as a by-product from the process of furfural extraction of lubricating oil base stock in AL-Dura refinery at different operating condition, were carried out at a fixed bed laboratory reactor. The initial boiling point for these feeds was 140 ºC for sample (1), 86 ºC for sample (2) and 80 ºC for sample (3). The catalytic cracking processes were carried out at temperature range 325-400 ºC and initially at atmospheric pressure after 30 minutes over 9.88 % HY-zeolite catalyst load. The comparison between the conversion at different operating conditions of catalytic cracking processes indicates that a high yield was obtained at 375°C, according to gasoline production. The distillation of cracking liquid products was achieved by general ASTM distillation (ASTM D -86) for separation of gasoline fraction up to 220 ºC from light cycle oil fraction above 220 ºC**.** According to gasoline production, it can be noticed that the condition of the feed with the lowest initial boiling point (80 ºC) (sample 3) made it gives more production of gasoline as compared with the other feeds (sample 1,2). At the best temperature (375 ºC), for the best feed for the production of gasoline (sample (3)), the production of gasoline + kerosene were 19.315, 16.16 and 12.95 wt.% for sample (2, 3 and 1). The RON for the gasoline produced from the catalytic cracking for the feed of the lowest initial boiling point was 92.3.

Keywords: *catalytic cracking, furfural extraction, lubricating oil.*

1. Introduction

Extract lubricating oil is a by-product of the furfural extracting process that applied in refinery process to develop the viscosity index of various kind of lubricating oil fraction. It is a totally rich aromatic content by-product which normally has black color, which identified by high polycondensed aromatics and some lubricating oil fraction and also by high viscosity [1,2]. The tires industry uses large amounts of an aromatic extract. On the other hand, lower amounts of it are utilized in plastic and rubber manufacture and asphalt blends. In spite of that the applications of aromatic extract are relatively limited [3].

These compounds are described as residuum aromatic extracts (RAE) if the oil base stock produces in vacuum process and distillate aromatic extracts (DAE) if it produces in atmospheric distillation [1,2].

The refinery process that is most popularly utilized for yielding gasoline of high octane value from heavy oils is catalytic cracking. This process utilizes to remove the sick effects of thermal cracking, like high carbon producing, the unsaturation of products. The reaction mechanism identifies catalytic decomposition from thermal decomposition and it is named as carbonium ion mechanism. The presence of this mechanism causes the yielding of extra stabilized saturated components having an excess attends for isomeric reactions. The steadiness of these ions are essentially controlled by the nature of molecule submits cracking, then the conditions of decomposition [4].

The kind of the feedstock effect on the octane number of the produced gasoline, gasoline that has high octane number yields from a feed of high aromatic content except, the formation of coke on catalyst's surface is higher and the yielding of gasoline is lower. Depending on the fact that the feedstock of high cycloparaffins content is extra efficient and simply separate yielding olefins, it provides medium quality and yield of gasoline, while the paraffinic feedstock is ready to decomposition and produce a larger gasoline's quantity having lower octane number as compared with those collected from cycloparaffins as well as aromatics including the largest transformation [5,6].

Zeolites are microporous crystalline, hydrated aluminosilicates of alkaline earth metals or alkaline. Its frameworks are consist of $[SiO_4]_4$and $[AlO_4]_5$tetrahedra that produce various open structure by sharing the corner. The negative charge of the lattice is offset by the positive charge of cations that placed at special sites of framework [7]. $M_2/nO\cdot Al_2O_3\cdot ySiO_2\cdot wH_2O$, is the practical formula for zeolite. where n is the cation valence ,y is 2–200, , and w describes the water consisted in the intracrystalline channels of zeolite [8].There are three types of manufactured zeolites are, A, X, Y. For the purpose of adsorption and disconnection of normal alkanes from their mixtures with hydrocarbons of other kinds and for cleaning and drying of hydrocarbon gases from hydrogen sulphide and carbon dioxide , type A zeolites that have a pore diameter of 0.3-0.5 nm are utilized [9]. Zeolite of type X have a pore size diameter of 0.8-1.3 nm and class Y 0.8-0.9 nm. The general used zeolites are X and Y. Zeolite Y remains to present the largest degree of catalytic stability with the largest yielding of gasoline with highest octane. The other molecular sieves and zeolites have lower product selectivity or they have been deficient in instability so they are generally unsuccessful [10,11].

Syamsuddin et al. discussed the catalytic decomposition of atmospheric petroleum residue oil using a batch reactor at 340 ºC for 3 hours residence time at high-pressure. The conversion was 60-65 wt. % and the yield of gasoline was 11.5-13.37 wt. % [12].

Esgair et al. studied the catalytic activity of faujasite type NaY catalysts produced from kaolin (local clay) with various Si/Al ratio in the temperature range of 450-525° C, atmospheric pressure, weight hourly space velocity (WHSV) of 5-20 h^{-1} , particle size ≤75µm. This process is carried out at an experimental laboratory plant scale of fluidized bed reactor. It was found that the cumene conversion rising with lowering WHSV and rising temperature. The highest conversion 42.36 mol.% was at 525° C and WHSV 5 h^{-1}, for catalyst with 3.54 Si/Al. The rapprochement of standard with that of the X-ray diffraction intended NaY zeolite catalyst display that the catalyst is Y-zeolite. At the same operating condition and by utilizing the same clay for preparation , Y-zeolite introduce larger transformation than X-zeolite [13].

Mohammed et al. studied the catalytic cracking carried out at a laboratory plant scale of fluidized bed reactor for vacuum gas oil by utilizing the faujasite type Y- zeolite catalyst which prepared from kaolin that is locally available. The pore volume and surface area for prepared catalyst was 0.39 cm^3 /g and 360 m^2 /g respectively. The cracking process was carried out in the temperature range of 440 to 500 °C, weight hourly space velocity (WHSV) range 10 to 25 h^{-1},and at atmospheric pressure. The conversion at 500 °C and WHSV 10 h^{-1} by utilizing faujasite kind NaHY, $NaNH_4Y$ and NaY zeolite were 69.5 %, 64.1% and 50.2 wt.% respectively. The productivity of gasoline utilizing the same operating conditions were 36.8% ,30.5% and 24.8 wt.% respectively [14].

Rahman et al. studied the catalytic cracking conversion of Iraqi vacuum gas oil on large and medium pore size (HY, HX, ZSM-22 and ZSM-11) of zeolite catalysts by utilizing a continuous fixed-bed laboratory reaction unit. The processes are carried out at the range of temperatures of 673 to 823K, and LHSV range of 0.5-3 h^{-1}. It was found that the catalytic conversion of vacuum gas oil rises with temperature rising and decreasing in LHSV. The activity of catalyst arranged in this order: HY> HX> ZSM-11> ZSM-22. The kind of the catalyst and the temperature effect highly on the product distribution. HY zeolite catalyst produced higher hydrocarbon. The selectivity toward large octane number products for the suggested catalysts are arranged in this order: HY>HX>ZSM-11>ZSM-22 [15].

In the present work, the characterization of three feeds of extract lubricating oil, the performance temperature for catalytic cracking of the feeds using a fixed bed laboratory unit and HY-Zeolite, and the characterization as well as the composition of the produced gasoline were studied.

2. Experimental
2.1 Materials
2.1.1 Feedstock

The feedstock of this work is extract lubricating oil that yield in Al-Dora Refinery by extraction process of lubricating oil stock.

2.1.2 Catalyst

The utilized catalyst for cracking process is HY-zeolite catalyst provided from Pye Unicom Company. The properties of this catalyst are presented in table 1.

Table 1,
The properties of the catalyst

Characteristics	Value
Cylindrical size, mm	3
Bulk density, g./ cm^3	0.8
Si / Al	3.95
Average pore diameter, A	8
Surface area, $m^2\ /g$.	210

2.2 The Experimental Unit

The catalytic cracking experiments were carried out in fixed bed laboratory plant scale. Fig.1 shows the diagram of this unit, that includes: 1. Reactor volume (250 ml), 2. Thermostat couple type K, 3. Temperature controller, 4. Electrical heater, 5. thermal insulator, 6. Valves fitting.

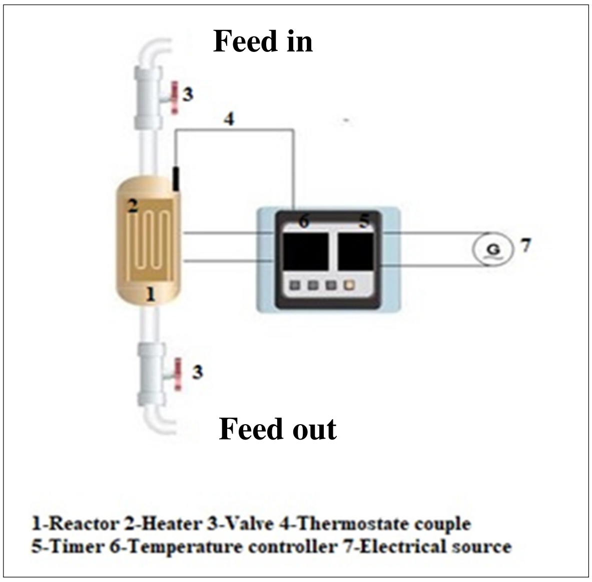

Fig. 1. Diagram of laboratory reactor unit.

The reactor consists of stainless steel tube with 14 cm length and 5 cm inside diameter and 250 ml volume. It was controlled and heated automatically by one heater which insulated by the jacket and carried by the climb. The temperatures inside the reactor were measured by thermocouple fixed inside the reactor at the top.

2.3 The Experimental Procedure

105 ml of extract lubricating oil as a feed (1, 2 and 3) put in the fixed bed reactor unit, that heated at a various temperature (325, 350, 375 and 400 °C) for four runs at residence time (30 minutes) for each run using 10 gm of Y-zeolite catalyst for each run.

Liquid produced as a result of cracking was distillated in ASTM D86.

3. Results and Discussion
3.1 Distillation of the Feeds (Extract lubricating Oil)

The distillation curves for the three feeds of extract lubricating oil were shown in Fig. (2, 3 and 4). The data of these figures are in Table 2.

As shown in table 3, it was noticed that the true boiling point distillation information gives an extra itemized description to extract lubricating oil volatility. The initial boiling point (IBP) for true boiling point distillation test is minimum as well as its end boiling point (EP) is higher as compared with the test of ASTM, the reason behind that is its separation degree is much higher from that of the test of ASTM distillation.

The curve of TBP (sketch for normal boiling point (NBP) against sample distilled percent volume) is used like a principle for description the extract lubricating oil for purpose of analysis and design.

Listak and Oja analyzed the variation between the actual average boiling points and ASTM D86 boiling points for continuous oil mixtures of Shale oils that are “synthetic” crude oils yielded industrially from solid oil shale by pyrolysis, at 500 °C. There was noticed that the average boiling point collected for a cut using atmospheric boiling points was constantly minimum than the one collected from ASTM D86 distillation "as an arithmetic average of the initial and final temperatures of the cut ". This was noticed to be primarily due to an interplay between the residence time in the condenser and the heating rate of the sample. [16].

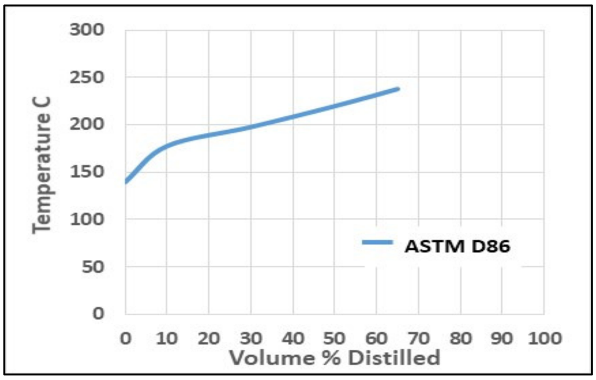

Fig. 2. ASTM D86 of sample 1.

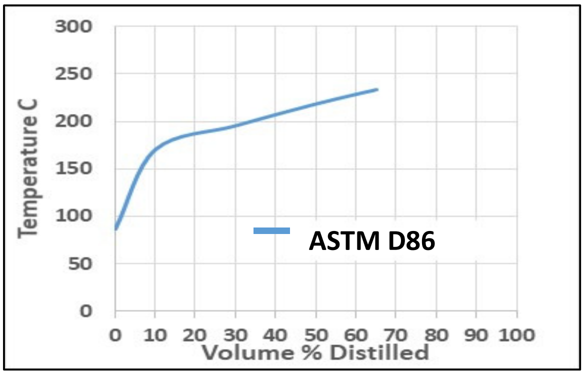

Fig. 3. ASTM D86 of sample 2.

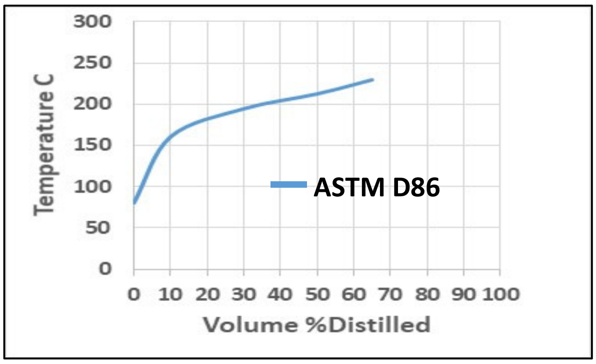

Fig. 4. ASTM D86 of sample 3.

Table 2,
The ASTM distillation of samples.

Vol. %	Number of Extracts Temp. in °C		
	1 **IBP=140**	**2** **IBP =86**	**3** **IBP =80**
5	156	142	146
10	178	170	160
15	180	175	166
20	190	187	169
25	194	191	171
30	198	195	194
35	210	202	197
40	214	211	200
45	216	215	208
50	220	218	212
55	222	221	217
60	234	225	222
65	238	233	229
+65	+238	+233	+229

Table 3,
TBP of three type of feed

Volume %	True boiling point °C		
	Sample (1)	**Sample (2)**	**Sample (3)**
0	110	60	55
10	162	154	143
30	193	190	189
50	223	221	214
70	253	249	248
90	278	278	278
95	288	285	282

The results from atmospheric distillation by ASTM D-86 for feeds and the true boiling point distillation (TBP) are used to calculate some physical properties after calculating the mean average boiling point.

Table 4,
The physical and chemical properties of three samples for extract lubricating oil

Property	Number of Extract		
	1	**2**	**3**
Molecular weight (g/gmole)	144	143.6	141
Specific gravity	0.972	0.965	0.964
API	14.08	15	15.28
Mean average boiling point (T_b) °C	221	218	214
Aniline point °C	78	76	74.5
Diesel index	24.27	25.35	25.36
Cetane index	62.5	60.5	59
Refractive index	1.544	1.546	1.547
Sulfur content %	2.77	1.86	1.59
Aromatic content %	30.6	26.87	24.77
Paraffinic content %	33.125	30.23	28
Hydrogen content %	11.42	11.525	11.54
Heat content MJ/Kg	42. 3	42.3	42.32

3.2 Catalytic Cracking

Liquid products of catalytic cracking which obtained at temperatures (325,350, 375 and 400 ℃) by batch laboratory unit were distilled at continuous wide range distillation unit (ASTM D-86). The atmospheric distillation was continued until the temperature of the cracking product in the distillation flask reached 250 ℃ to separate gasoline + kerosene fraction.

Table 5 displays the production of gasoline by catalytic cracking for the three samples at different temperatures (325,350, 375 and 400 ℃). Tables (6, 7 and 8) display the catalytic decomposition outcomes at 375 °C for each of the three samples (sample 1,2and 3) respectively.

Figure (5) display the comparisons between the yield of gasoline and kerosene for catalytic decomposition of the three samples at 375 ℃.

Table 5,
Production of gasoline by catalytic cracking for the three samples at different temperatures.

Temperature (°C)	Vol. of gasoline fraction at 220 °C		
	Sample (1)	Sample (2)	Sample (3)
325	12	16	19
350	14	18	21
375	16	20	24
400	11	15	18

Table 6,
Material balance of catalytic cracking of sample (1) at 375 °C.

The fraction	Boiling range °C	%yield based on vol.	%yield based on wt.
Gasoline	87-150	6	4.65
Kerosene	150-250	10	8.3
Coke			2

Table 7,
Material balance of catalytic cracking of sample (2) at 375 °C

The fraction	Boiling range °C	%yield based on vol.	%yield based on wt.
Gasoline	80-150	8	6.2
Kerosene	150-250	12	9.96
Coke			3

Table 8,
Material balance of catalytic cracking of sample (3) at 375 °C.

The fraction	Boiling range °C	%yield based on vol.	% yield based on wt.
Gasoline	76-150	11	8.525
Kerosene	150-250	13	10.79
Coke			1.5

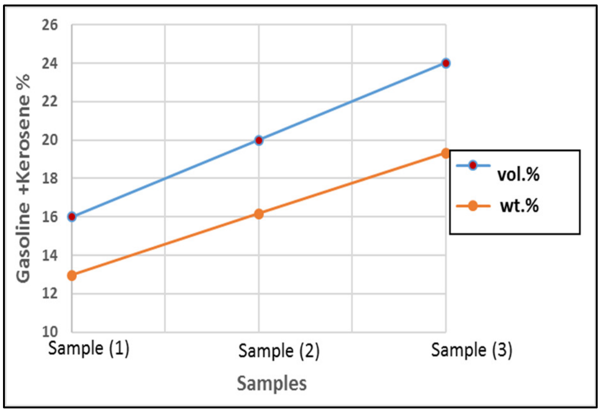

Fig. 5. vol. % and wt.% of Gasoline + Kerosene produced from three samples by catalytic cracking at 375 °C.

For each of the three samples, in this situation, catalytic activation proceeds in synchronism with thermal activation. Addendum to that, rising within the position of active acid which can be utilized for reaction [17,18].

From table 5 it can be noticed that the best temperature in this work was 375°C.

For each of these samples, production of unsaturated gasoline is the elementary step for extract lubricating oil decomposition and this product is commonly the required output. Yet, the produced gasoline perhaps go under furthermore reaction which will be either oligomerization as well as cycloaddition for the compounds into dehydrogenated outputs as well as coke or subjected to side decomposition, which is commonly indicated to over decomposition, into gases component [19].

Precipitated coke, as well as light side outputs, are produced by partly conversion of gasoline that produced like an intermediate output. Each of these compounds minimizes the productivity of gasoline in the case of high amount of transformation for the extract lubricating oil. Commonly, the transformation amount regulates in amount equal to that of the highest gasoline in order to prohibit the incidence of this process of decay that damage the profit of the process [5].

According to that the yield of gasoline increase until the run of (375°C) and reduce by increasing the temperature to 400 °. The mentioned results are agree with those mentioned by Esgair [17] as well as Maadhah et al. [18].

As shown in Fig.5 the gasoline yield reaches the maximum in sample (3).

As shown in table 4, all of the properties of extract lubricating oil samples are changed and there is not one property that changed in one feed and being constant in other feeds, so according to that the study of the effect of the different kind of extract lubricating oil samples on the production of gasoline is complicated.

The structure, as well as the productivity of gasoline, are highly respected for the kind of raw material. And so on, hydrotreated or paraffinic feedstocks yield higher gasoline as compared with non-hydrotreated or aromatic feeds. At high transformation for the feed, the presence of aromatics is favorable for the produced gasoline [20].

Stratiev et al. studied the effect of 35 vacuum gas oils properties on reactivity, conversion, and yields in fluid catalytic cracking. It was found that at the catalytic decomposition of deferent raw materials, the content of saturated compound rises the reactivity of feedstock which also declines with the rising in the content of aromatic. The minimum aromatic content and higher saturated and hydrogen content of the feed cause higher conversion in catalytic decomposition [21] . Depending on that sample (3) in the present work will give higher productivity of gasoline.

The feed with the lower boiling point and lower gravity has higher H/C ratio and that made it produce higher yield of gasoline [22]**.** From table 4, sample (3) has the lower boiling point (214 °C) and lower specific gravity (0.964), depending on that it produces higher yield of gasoline.

3.3 The Possibility of Extract Lubricating Oil Cracking Uses

The chemical composition of the produced gasoline using catalytic cracking from the sample (3) at best temperature (375°C) tested by utilizing PIONA test.

Table 9,
The chemical compositions of cracked gasoline produced from sample (3) at 375 °C

PIONA analysis	Total Mass%
n-Paraffins	6.79
Naphthenes	3.13
Iso paraffins	37.91
Aromatics	33
Olefin	11.76
Oxygenated	7.3

PIONA composition for gasoline that produced is described in table 9 ,which shows that the yielded gasoline has low olefins content in compared with gasoline produced by Esgair [17]**,** who explained that the olefins content in yielded gasoline is 34.1%. The reason for that may be hydrogen transfer reactions growing that in which olefins with naphthenes reaction produce paraffins as well as aromatics. Naphthenic components are able to react with olefins yielding aromatics as well as paraffins because these compounds are hydrogen donors.

Since the extra reactive bonds are the double bonds are more reactive which simply polymerized as well as oxidized causing the production varnishes, as well as gums, injection nozzles and valves of automobiles, will be blocked due to the gasoline high olefin content [23]**.**

It also can be noticed that gasoline which formed from this catalytic decomposition has a lot of aromatics as well as isoparaffins and that cause high research octane number (RON) for this product. Toluene, 1,3-dimethylbenzene, and isobutyl benzene are the aromatic compounds that present in produced gasoline and those have high RON which is 111, 115, and 112 RON, respectively for each of them while isoparaffin compounds have high octane numbers (85-100) [24].

Finally, it is able to utilize the gasoline that yielded by the decomposition of extract lubricating oil as a useful requisite gasoline for regular and premium automobile gasoline yield because the minimum research octane number of regular and premium Iraqi gasoline are 85 and 90, respectively [25].

Table 10 shows the characteristics of produced gasoline by catalytic cracking and required properties of this fraction.

Table 10,
The characteristics of produced gasoline

Characteristics	**Value**	
	Catalytic cracking	**Iraqi commercial value**[25]
Specific gravity, 15.6°C/15.6 °C	0.8	0.775
API gravity	45.375	51.00
Net heat, MJ/ kg.	11000	11000
Octane number	92.3	85

The gasoline has an octane number identical to the required.

4. Conclusion

The use of extract lubricating oil is almost limited but it has the ability to crack because of its high molecular weight so it was able to crack it to produce light fractions more valuable fuels.

The study on the effect of extract lubricating oil catalytic cracking in a fixed bed laboratory reactor shows that the increasing in reaction temperature from 325°C to 375°C gives higher yield of gasoline. After this temperature the yield decrease because the gasoline itself will crack and light cycle oil yield increase in the run of 400 °C.

Based on the maximum yield of gasoline it could be said that the best cracking temperature was 375ºC.

Feed with lowest initial boiling point (sample 3) gives more gasoline yield.
Gasoline yield increase in the run of 375 °C for sample (3) where it is 19.315 wt.%.

From the PIONA analysis, the produced gasoline has high research octane number because it has high isoparaffins and aromatics. This means that it is possible to use gasoline for the automobile.

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معالجة المتبقي من مستخلص زيوت التزييت بالتكسير باستخدام عامل مساعد لإنتاج مقاطع خفيفة

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الخلاصة

يتعامل هذا البحث مع التكسير الحراري والتحفيزي لثلاث عينات من مستخلص زيت التزييت المنتج بوصفها منتجاً ثانوياً من وحدة الاستخلاص باستخدام الفيرفرال لزيوت التزييت في مصفى الدورة في ظروف تشغيل مختلفة في مفاعل مختبري من نوع fixed bed. نقطة الغليان الأولية لهذه المنتجات هي: ١٤٠° س للعينة (١)، ٨٦ ° س للعينة (٢) و ٨ °س للعينة (٣). تم تنفيذ عملية التكسير في نطاق درجة حرارة ٣٢٥-٤٠٠° س في البداية في الضغط الجوي لمدة ٣٠ دقيقة باستخدام ٩,٨٨ ٪ من الزيولايت. لقد بينت المقارنة بين نسب التحويل في الظروف المختلفة للتكسير باستخدام العامل المساعد ان افضل نسبة تحويل كانت عند درجة حرارة ٣٧٥° س اعتماداً على الكازولين الناتج. تم تقطير سائل التكسير الناتج من العملية باستخدام (ASTM D -86) و ذلك لعزل الكازولين عند درجة حرارة تصل الى ٢٢٠°س عن المقاطع الاخرى في درجات حرارة اعلى من ٢٢٠° س . إن ظروف الماده الداخلة للتفاعل و التي تحمل أقل نقطة غليان أولية ٨٠ °س جعلته يعطي إنتاج اعلى للكازولين مقارنة مع بقية المواد الاولية (العينة ٢،١). عند افضل درجة حرارة (٣٧٥ ° س) لأفضل عينة (العينة ٣) كان انتاج الكازولين + الكيروسين عند التكسير ١٩,٣١٥٪ بالوزن و للعينة (٢) ١٦,١٦٪ بالوزن و للعينة (١) ١٢,٩٥% بالوزن. الرقم الاوكتاني للكازولين الناتج من التكسير الحفزي من المادة الاولية التي تحمل أقل نقطة غليان أولية كان ٩٢,٣.