

Production of Petroleum – like Fractions from Waste Cooking Oil

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HYDROCRACKING technique was applied here for producing petroleum- like fractions from waste frying oil. The experiments were performed in a fixed bed flow reactor system, under high pressure using zeolite catalyst. The operating conditions were optimized and found to be 400°C, 2.5 MPa hydrogen pressure and 5 h⁻¹ LHSV.

The obtained biofuel was fractionated to gasoline, kerosene and gas oil- like fractions. All the required analyses were accomplished and the data were evaluated. The results indicate that the properties of the attained products are completely compatible with those of the corresponding distillates ensued from petroleum source.

Keywords: Biofuel, Hydrocracking, Waste cooking oil.

With the progressive exhaustion of petroleum oil reserves, and in serious consideration of environmental issues, a worldwide trend is concentrated on searching for alternative and renewable energy sources [1-3]

Recently, biofuel derived from vegetable oils is receiving a great attention as the most suitable and logical alternative of fossil fuel [4-6]. Considering the problem arising from the intensive use of biofuels come from vegetable oils that could derive in a shortage of raw materials necessary for feed purposes, instead, non-edible plant oils can be employed to fulfill this objective [7-10].

Waste frying oil is visualized as one of the non-edible vegetable oils that regarded to be unsuitable for human consumption. During frying process, the oil is repeatedly subjected to high temperatures in the presence of air and moisture. Under these conditions, degradation reaction proceeds, leading to several changes in the physical and chemical properties of the oil [11-13]. If the frying process is continued, further degradation will occur, and finally the oil will not be appropriate for frying and it has to be discarded.

Disposal of waste cooking oils creates serious environmental problems [14]. This concept motivated researchers not only to tackle the disposal matter of the used oil but also to find a way for producing a renewable liquid biofuel from these low-cost wastes [15-20].

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Currently, transesterification is the primary route for producing biofuels (particularly biodiesel) from plant oils [21-24]. Although this process has many benefits, however, it shows several disadvantages [25,26]. To eliminate or even to minimize such drawbacks, some modern technologies have been developed, such as hydrotreating and hydrocracking processes [27,32]. These technologies have significant potential as the produced oils have better fuel properties than those obtained via transesterification. In addition, they employ the existing infrastructure of petroleum refineries [33].

This study aimed to apply the hydrocracking technique for producing clean biofuel from non-edible waste cooking oil, using a novel zeolite catalyst. A derivation of the optimum operating conditions for attainment the highest biofuel yield with the best quality is an essential target of this work.

Experimental

Feedstock

Waste cooking oil (WCO) collected from some restaurants, fast food and households, was employed in this study as a feedstock. The sample was heated to 60 °C and filtered under vacuum in press filter to get clear and clean oil, then dehydrated using anhydrous sodium sulfate, left over night and finally filtered again under vacuum.

Catalyst

HYDEX-L, a novel zeolite based catalyst, developed by the SUD-CHEMIE GROUP, was used here to catalyze the hydrocracking reaction. It has extrusion shape with 1.5 , 2.5 mm size , surface area of = 290 m²/ gm and total pore volume amount to 0.3331 cm³. The catalyst was dried at 120°C for 3 hrs , calcined at 450°C for 6 hrs in air and then reduced at 450°C under 1 MPa hydrogen pressure .

Catalytic activity testing

Hydrocracking of the waste frying oil over zeolite catalyst was examined at various experimental conditions of temperatures, pressures and liquid hourly space velocities , to investigate the influence of each of these factors on the procession of the reaction, and also to find out the optimum operating circumstances for getting the highest yield with the best quality of the desired product .

The experiments were carried out in a high pressure cata - test unit equipped with a fixed bed reactor. A simplified schematic diagram of the employed setup is illustrated in Fig.1.

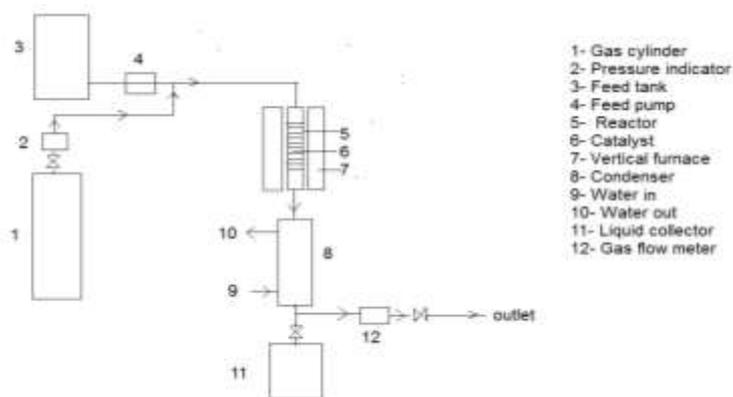


Fig.1. Simplified schematic diagram of cata-Test Unit.

The reactor was loaded with the zeolite catalyst mixed with the same volume of ceramics and placed in a vertical furnace. The unit was flushed with hydrogen gas, and kept constant under H_2 pressure to check any leakage, thereafter; the operating conditions were adjusted as required. Once the steady state had reached, the feed was pumped to the top of the reactor by means of a piston pump having an adjusting knob. The reactor effluents were cooled in a water condenser, the gases were passed through a gas meter to the outlet, whereas the liquid product was collected in a receiver, estimated, analyzed and evaluated.

The obtained biofuel was fractionated to three cuts with boiling points in the range of IBP- $150^\circ C$, $150 - 250^\circ C$ and $250 - < 344^\circ C$, which corresponds to gasoline, kerosene and gas oil – like fractions, respectively. The remainder (that boiled above $344^\circ C$) is considered as the unconverted oil or residuals. Both the hydrocracked products and the individual fractions that attained at the optimum operating conditions were characterized according to the different ASTM methods.

Results and Discussion

Effect of liquid hourly space velocity (LHSV)

The influence of LHSV on hydrocracking the used cooking oil (UCO) over zeolite catalyst was studied at fixed reaction conditions of $450^\circ C$, 2.5 MPa hydrogen pressure and 1600 L/L H_2 /feed ratio. The distribution and properties of the products are declared in Table 1 and Fig. 2.

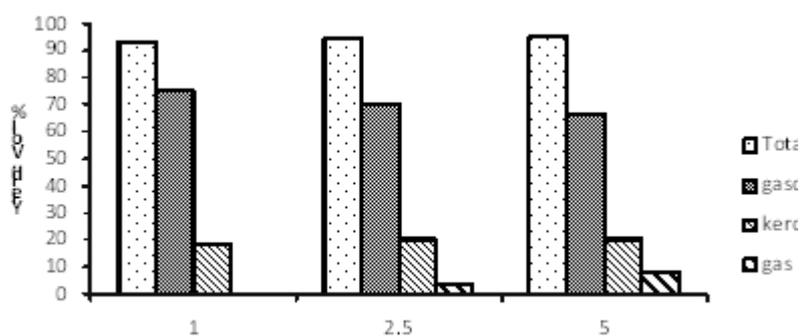
The data reveal that almost complete conversion of the feed to gases and organic liquid (get to 97.22 wt %) has been occurred throughout the investigated range of LHSV. On reducing the velocity of the feed, surplus gases were then evolved, accompanied by an equivalent diminution of the obtained liquid. This resulted from the ascendancy of severe hydrocracking at longer contact time. Accordingly, 13.21, 15.98, 18.81 wt % gases associated with 86.79, 84.02 and 81.19 wt% of OLP were obtained at 5, 2.5 and $1 h^{-1}$, respectively.

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TABLE 1. Distribution of the hydrocracked used cooking oil products obtained at various LHSV.

Hydrocracked Products	1 h ⁻¹	2.5 h ⁻¹	5 h ⁻¹
Hydrocracked products , Wt %			
Gases, Wt %	18.81	15.98	13.21
Liquids , Wt %	81.19	84.02	86.79
Water content, Wt %	-	-	-
Oil content, Wt %	81.19	84.02	86.79
Density at 20 ^o C	0.8086	0.8301	0.8373
Kinematic viscosity, cSt at 40 ^o C	1.214	2.841	3.202
ASTM distillation, Vol. %/ ^o C			
IBP	50	50	50
10	54	60	61
20	60	71	72
30	71	89	83
40	79	90	100
50	88	110	123
60	115	130	140
70	135	150	165
80	165	190	203
90	186	248	264
FBP	195	272	281
Recovered, Vol. %	93	94	95
Gasoline , Vol. %	75	70	67
Kerosene , Vol. %	18	20	20
Gas oil , Vol. %	-	4	8
Residue and losses, Vol. %	7	6	5
Conversion of >344 ^o C fraction, Wt%	97.22	97.22	97.22
Liquid biofuel yield , Wt %	74.165	77.912	81.58

Temperature: 450 °C, Pressure: 2.5 MPa and H₂ / feed: 1600 L/L.

**Fig. 2.** Distribution of the individual fractions of the biofuel obtained from hydrocracked used cooking oil as a function of LHSV.

The obtained oil is characterized by adequate values of densities and viscosities (that varies between 0.8373 to 0.8086) and (from 3.202 to 1.214), respectively, which are completely appropriate for a fuel (taking in consideration that 0.9264 and 42.2 cSt were determined for the feed, consequently).

The ASTM distillation results reflect the abundance of gasoline fraction in the hydrocracked products that represents the principal constituent of the biofuel yields attained at all the examined LHSV. It fluctuates from 67 up to 75 vol% of the acquired biofuels. This evidences that, the used zeolite catalyst is highly selective to gasoline production that enhanced at longer contact time. Consequently, this concomitant with deficiency of the heavier fractions particularly at lower space velocities, so much that the gas oil fraction is quite vanished at 1 h^{-1} .

The highest deal of liquid biofuel equivalent to 81.58 wt% can be realized at 5 h^{-1} , hence it was selected as the optimal LHSV to be applied for the next study.

Effect of reaction temperature

The performance of zeolite catalyst for hydrocracking used cooking oil (UCO), as a function of reaction temperature was examined at constant circumstances (*i.e.* : LHSV of 5 h^{-1} , pressure 2.5 MPa and H_2 /feed ratio 1600L/L).

The data illustrated in Table 2 indicate the significant influence of 400 °C and 450 °C reaction temperature on the conversion, that get to the maximum of 97.22 wt% at both temperatures. Nevertheless, the worst transformation has been occurred at 350 °C, as it gives only 5.55 wt% of the converted product. This signifies that such relatively low temperature is not sufficient (to a certain degree) to promote the hydrocracking reaction to proceed.

Upon raising the reaction temperature from 350°C up to 450°C, gradual increases of gaseous product (from 5.54 to 13.21 wt %), associated with dropping in the liquid yields (from 94.46 to 86.79wt %) can be observed.

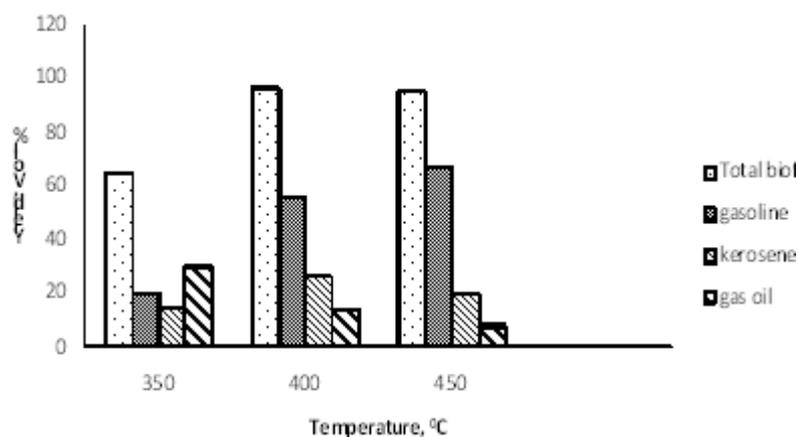
Intensive temperatures have markedly improved both density and viscosity of the oil yield. The organic liquid products (OLPs) exhibit density and viscosity values of 0.8917 and 30,171 cSt at 350°C that progressively ameliorated to become 0.8373 and 3.202 cSt at 450°C, respectively. The high viscosity observed at 350°C may be ascribed to the saturation of the double bonds of the oil molecules due to the predomination of the exothermic hydrogenation reaction (that competed with cracking reaction) at such relatively lower temperature.

The disappearance of water all during the applied temperature range substantiates that the reaction implemented neither via decarbonylation nor hydrodeoxygenation pathway⁽³³⁾. The distillation data demonstrate the domination of gasoline fraction in the hydroconverted products particularly at 400°C and 450°C that constitutes 56 and 67 vol% of the biofuel attained, whereas kerosene amounts to 26 and 20 vol%, added to 14 and 8 vol% gas oil were achieved at both temperatures, respectively (Fig. 3).

TABLE 2. Distribution of the hydrocracked used cooking oil products obtained at various temperatures.

Hydrocracked Products	350 °C	400 °C	450 °C
Hydrocracked products , Wt %			
Gases, Wt %	5.54	12.14	13.21
Liquids , Wt %	94.46	87.86	86.79
Water content, Wt %	-	-	-
Oil content, Wt %	94.46	87.86	86.79
Density at 20 °C	0.8917	0.8318	0.8373
Kinematic viscosity, cSt, at 40 °C	30.171	4.793	3.202
ASTM distillation, Vol. %/ °C			
IBP	95	60	50
10	115	72	61
20	148	80	72
30	180	103	83
40	261	118	100
50	272	130	123
60	295	155	140
70	-	185	165
80	-	249	203
90	-	275	264
FBP	305	283	281
Recovered, Vol. %	65	96	95
Gasoline , Vol. %	20	56	67
Kerosene , Vol. %	15	26	20
Gas oil , Vol. %	30	14	8
Residue and losses , Vol. %	35	4	5
Conversion of >344 °C fraction, Wt%	5.55	97.22	97.22
Liquid biofuel yield , Wt %	62.858	82.997	81.58

Pressure: 2.5 MPa, L.H.S.V.: 5 h⁻¹ and H₂ / feed: 1600L/L.

**Fig. 3. Distribution of the individual fractions of the biofuel obtained from hydrocracked used cooking oil as a function of Temperature.**

The biofuel accomplished at 350°C is characterized by the relative abundance of the heavier gas oil fraction, but the total biofuel liquid yield is not satisfactory as it represents only 62.858 wt%.

Accordingly, the most gratifying temperature that regarded as the best for further investigation is 400°C, since it shows a high conversion and high biofuel yield comprised about 83 wt%, also it reveals proper values for density and viscosity.

Effect of hydrogen pressure

The data in Table 3 shed light upon the distribution of the hydrocracked products of used cooking oil (WCO), using zeolite catalyst. The experiments were carried out at a different H₂ hydrogen pressure in the range of 1 to 5 MPa, and at constant reaction conditions (*i.e.* 400°C, 5h⁻¹ LHSV and 1600L/L, H₂/feed ratio).

TABLE 3. Distribution of the hydrocracked used cooking oil products obtained at various H₂ pressure.

Hydrocracked Products	1.0 MPa	2.5 MPa	5.0Mpa
Hydrocracked products , Wt %			
Gases, Wt %	2.20	12.14	18.98
Liquids , Wt %	97.80	87.86	81.02
Water content, Wt %	-	-	-
Oil content, Wt %	97.80	87.86	81.02
Density at 20 °C	0.8895	0.8318	0.8385
Kinematic viscosity, cSt, at 40 °C	15.672	4.793	3.653
ASTM distillation, Vol. %/ °C			
IBP	80	60	50
10	89	72	68
20	100	80	78
30	138	103	90
40	168	118	110
50	259	130	123
60	281	155	145
70	-	185	165
80	-	249	202
90	-	275	260
FBP	295	283	279
Recovered, Vol. %	69	96	95
Gasoline , Vol. %	30	56	65
Kerosene , Vol. %	19	26	20
Gas oil , Vol. %	20	14	10
Residue and losses , Vol. %	31	4	5
Conversion of >344 °C fraction, Wt%	16.667	97.22	97.22
Liquid biofuel yield , Wt %	69.886	82.997	73.939

Temperature: 400 °C, LHSV. : 5 h⁻¹ and H₂ / feed: 1600L/L.

97.22 wt % conversion of the heavy fraction in the feedstock with a boiling point higher than 344°C was accomplished at pressures beyond 1 MPa. Inhibition of the conversion is clearly obvious at the lowest pressure of 1 MPa, at which the pressure is not sufficient to enhance the hydrocracking reaction, considering only 16.67 wt% of the feed with a boiling point above 344°C, was hydrocracked. The produced liquid is then characterized by high density and viscosity values, comprised 0.8895 and 15.672 cSt respectively.

The data also reflect the significant influence of the hydrogen pressure on the amounts of the emanated gases. A substantial quantity of the undesirable gases in as much as 18.98 wt% was released at the highest pressure of 5 MPa, which led to consequential abatement in the magnitude of the produced liquid biofuel (comprised 73.939 wt %). On the other hand, at the lowest pressure of 1 MPa, only 2.2 wt % of gases was obtained. However, the results of this run are not satisfactory, as it gives the worst yield of liquid biofuel (amount to 69.886 wt %). Therefore, neither the experiment performed at 5 MPa nor that carried out at 1 MPa are accepted to realize our target.

It is apparent from the table, the mild amount of gases (*i.e.* 12.14 wt%) and the good yield of OLP which has a perfect density and viscosity values of 0.8318 and 4.793 cSt respectively, can be acquired at 2.5 MPa. At this pressure, the highest yield of petroleum-like fraction, as far as ≈ 83 wt % was accomplished, and composed mainly from 56 vol% gasoline as well as 26 vol% kerosene and 14 vol% gas oil (Fig. 4). So, 2.5 MPa has been recommended to be the optimal hydrogen pressure value.

Finally, it could be concluded that, the best operating variables for hydrocracking the waste cooking oil on zeolite catalyst are 5h^{-1} LHSV, 400°C and 2.5 MPa hydrogen pressure.

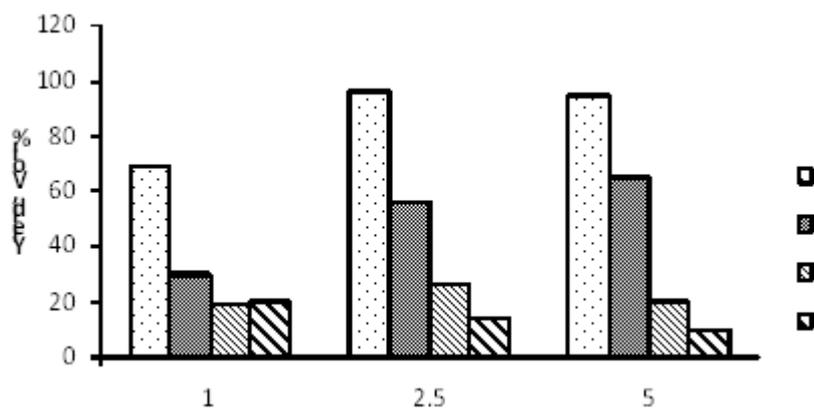


Fig. 4. Distribution of the individual fractions of the biofuel obtained from hydrocracked used cooking oil as a function of pressure.

Evaluation of the prepared petroleum- like fractions

The physicochemical properties of gasoline, kerosene and gas oil – like distillates obtained from fractionating the biofuel, produced from the WCO and hydrocracked over zeolite catalyst at the derived optimum operating conditions, were determined and compared with those of the corresponding commercial petroleum fractions. Their results are declared in Table 4.

The demonstrated data evidenced that, the characteristics of gasoline- like distillate is almost congruent with the properties of petroleum gasoline, with negligible differences. The calorific value of the prepared gasoline is to somewhat lower than that originated from the mineral oil, as it possesses a value of 46889 j/kg compared to 49360 j/kg respectively. This drop in the calorific value is possibly arisen from remaining some of the oxygenated compounds which existed in the molecular structure of the parent oil, whereas petroleum gasoline is constituted principally from saturated hydrocarbons.

TABLE 4. Physicochemical characteristics of petroleum like fractions compared with their corresponding petroleum refinery distillates .

Properties	Petroleum gasoline	Gasoline like fraction	Petroleum kerosene	Kerosene like fraction	Petroleum gas oil	Gas oil like fraction
Yield , wt%	-	41.938	-	22.061	-	12.176
Density /20 °C	0.748	0.7489	0.840	0.8485	0.8543	0.8697
Kinematic viscosity / 40 cSt.	0.5	0.55	1.5	1.74	6	6.25
Ash content , wt%	0.1	Nil	Nil	Nil	Nil	Nil
Molecular weight	105	105	170	178	230	254
Calorific value, j/kg	49360	46889	47220	45152	44300	43999
Sulfur content, wt %	0.05	Nil	0.3	Nil	0.3	Nil
Pour point , °C	- 40	-36	- 40	- 36	Zero	3
Refractive index	1.4100	1.41173	1.4458	1.4494	1.4620	1.4698
Smoke point , mm	-	-	26	25	-	-
Flash point , °C	-	-	58	60	150	125
Cetane number	-	-	-	-	47	45.41
Carbon residue, wt%	Nil	Nil	1.5	Nil	0.3	Nil

The other specifications of the biogasoline, derived from the WCO, such as specific gravity, kinematic viscosity molecular weight, pour point, carbon residue, and ash content are completely compatible with those of mineral gasoline. Furthermore, the obtained biogasoline is distinguished from that ensued from petroleum by the lack of sulfur, which presents a good advantage in reducing the harmful emissions of the exhaust gases and also inhibiting corrosion in engines .

In the same manner, the properties of the 2nd fraction that boiled in kerosene range were also specified, and compared with those of the commercial kerosene. Data in Table 4 indicate that, the properties of the biokerosene sample is approximately very close to that generated from petroleum source, with the exception of calorific value, which shows, to some extent, a lower magnitude, amounting to 45152 j/kg compared to 47220 j/kg for that derived from mineral oil. As mentioned previously, this decrement can be referred also to the oxygenated compounds involved in the produced bio-kerosene, originated from the oxygenated feature of its virgin oil.

As noticed from the data, all the other specifications of the obtained bio-kerosene are highly concordant with the corresponding fraction of petroleum crude oil. Moreover, it is ashless and not contains the undesirable sulfur compounds which cause the hazardous emissions and pollutes the environment.

Table 4 exposes also the properties of the gas oil fractions, which reflects the adequate resemblance of the physicochemical characteristics of both bio- and mineral fuels. Each of the calorific value, ash content viscosity, density, pour point, cetane number, flash point and carbon residue of the bio- gas oil is completely compatible with that of the corresponding fractions issued from petroleum oil. In addition, the gas oil –like distillate is distinguished by the lack of sulfur compounds.

Finally, the obtained data lead us to conclude that, the various distillates (*i.e.* gasoline, kerosene and gas oil – like fractions) generated from hydrocracking the WCO, can be definitely employed as alternates to fossil fuels, as they possess almost similar properties.

Conclusions

High yield with good properties of clean biofuel was obtained from hydrocracking WCO on zeolite catalyst.

83wt% of biofuel yield was accomplished at the optimum operating conditions of 400 °C, 5h⁻¹ and 2.5 MPa hydrogen pressure, which incorporates 56 vol% gasoline, 26% kerosene and 14% gas oil –like fractions.

The physicochemical characteristics of the acquired fractions are completely compatible with those of the corresponding petroleum distillates.

The attained biofuels possess more advantages as they are ashless, contain no sulfure or aromatic compounds, so, they can be utilized deservedly as alternates to fossil fuels.

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إنتاج وقود مماثل للمقطرات البترولية من بقايا زيت الطعام

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لما كانت المعالجة الهيدروجينية هي احدي التقنيات الحديثه الواعده التي يمكن تطبيقها في مجال إنتاج الوقود الحيوي ، مستغله في ذلك الأجهزة المعده سلفا لعمليات تكرير البترول لذا فقد تم استخدام طريقة التكرير الهيدروجينية في تحويل بقايا زيت الطعام الذي لا يصلح للاستهلاك الغذائي الي بدائل شبيهه بالمقطرات البترولية الطبيعيه بكفاءة عاليه وفي هذه الدراسة استخدم نوع جديد من الزيوليت كعامل حفاز ومعروف باسم HYDEX-L ، وأجريت عليه العديد من التجارب بالاستعانة بجهاز الضغط العالي لاختبار نشاطه الحفزي تجاه تفاعل التكرير الهيدروجيني وذلك تحت عوامل تشغيل مختلفه ، مع دراسة تأثير كل عامل منها علي مخرجات التفاعل . وتبين ان درجة الحرارة ٤٠٠ م ، وضغط هيدروجيني ٢.٥ ميجا باسكال ، وسرعة فراغية تساوي ٥ / ساعه ، وهي الظروف المثلي للحصول علي المنتج المطلوب بأكبر كميته وأفضل مواصفات وأكثرت النتائج ان الوقود المحضر عند تقطيره الي قطرات لها مدي غليان يشابه للجازولين والكيروسين والسولار ، قد تميزت بخصائص طبيعيه وكيميائيه مطابقة تماما لمواصفات مثيلاتها المنتجه من مصادر بترولية وقد تفوقها تميزا لعدم احتوائها علي المركبات العطرية والكبريت ، مما يقلل من الإنبعاثات الضارة الناتجة عن احتراق الوقود ولذا فقد ساهمت هذه الدراسة في إنتاج طاقه نظيفة جديدة ومتجددة وصديقه للبيئة من تلك الزيوت الغير صالحه للأكل بكفاءة وجوده عاليه عن طريق التكرير الهيدروجيني .