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# A catalytic cracking process on atmospheric residue from the Al-Diwaneyah petroleum refinery

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## ABSTRACT

The Reduced Crude Residue R.C.R it's a product of atmosphere distillation from crude oil in the Al-Diwaneyah refinery, where the product from the lower distillation tower at 300 °C then the atmospheric distillation unit. For Production of Light Petroleum fractions from Reduced Crude Residue R.C.R by Cracking Reaction. Catalytic cracking of reduced crude residue (R.C.R) was joined with a riser pressure continuous reactor was completed in an autoclave with different process selection, temperature about 350–450 °C, catalytic cracking time (60–120) minute, the dosage of catalyst (0.1-0.5) gm and pressure 1–5 bar in the existence of Nitrogen. Statistical program design of testing (RSM) by Box-Behnken (BB) was employed to predict the selection of necessary changes in the cracking by the catalyst of reduced crude residue (R.C.R), and to gain better process conditions. Depended on the (3) plane for the design of the factorial, the cubist system was progressing between the cracking by catalyst conditions to overall production, and the more influential operator for all testing system responses was selected. The prophecy yields and conversion of all upgrade liquid phase, gas phase, coke, and residue were found to agree satisfactory with the testing grade. At a temperature of 400 °C, a pressure of 1 bar, a dosage of 0.3 gm, and a longer residence time (2 h) the best experiment is to get the maximum yield of the liquid phase product. The better run for quality when temperature 400 °C, pressure 5 bar, dosage 0.5 gm and 90 minutes as running time depended on API, sp. gr, density, viscosity, and flash point.

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## 1. Introduction

Crude oil in its natural form isn't can usage in different specials and must be converted to the usable petroleum-based on fractions that produced by refinery in distillation unit such as kerosene, gasoline, and gas oil, also petrochemicals such as polypropylene, polyethylene, and another petroleum-based on polymers [1]–[3].

Because of the rising global requested for fuel and petrochemical, upgrading the highest and heaviest boiling point section of crude oil (i.e., vacuum and atmospheric remains) is becoming more frugality appealing and possible [4]. Approximately 94% of the anticipated increase in crude oil useful is due to raised consuming of light petroleum yield such as

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benzene, diesel, heavy and light naphtha [5]. In last years an increased request of middle and light distillates has product in the raised consumed like factory, building, and power station ...i.e. For distillate of these a section to additional useful lighter yield [6]. middle and Light distillates product from atmospheric crude oil residue is become very necessary for the universal refining manufacture because of the drop in traditional, light petroleum origin [7]. As a byproduct of atmospheric distillation of crude oil, which is widely used in refineries to produce light crude oil upgrades, residual petroleum is left. low boiling point for fractional oil distillate such as light naphtha in an economically justifiable manner [8]. Because of the decline in conventional, light petroleum supplies, the distillation of heavy petroleum remains to more profitable medium and light distillates is becoming more significant for the worldwide refining factory. Catalytic cracking is one of the well-known thermochemical conversion processes that has been widely used in demand of refineries for crude oil [9]. This process has many interest, like as a stronger cracking, lower coke consistence, and is a more environmentally conscious path, when compared with cracking by thermal [10]. Moreover, the cracking by catalytic of bio-oil also gives more selectivity of light olefins, such as ethylene and propylene, which represented a feed in the industry for petrochemical [11]. Numerous researchers have focused on transforming low-cost feedstock to light olefins via the catalytic cracking process. The development of catalytic cracking, one of the most significant technical advances of the last three decades, has effectively revolutionized the petroleum industry (8). The most common operation for upgrading heavy oils are thermal cracking, catalytic cracking and hydrocracking. However, coke formulation through cracking is a significant challenge in these operations [12]. Temperatures above (350–370) °C have identified as critical for crack (C–C), (C–H), and (C-heteroatom) bonds.[13] Coke can be formed from available last molecules or produced in the feed through the reactions for the cracking by thermal. The density of these precursors (for example, asphaltene molecules) increased when their solubility selects, a modern phase known as mesophase separates from the petroleum.[14] Coking, in special, is known not only for the recorded reduction in dense petroleum product but as well as a procedure for yield exceed light products. Coke have about 83% carbon and 6% hydrogen and equilibrium with clearness. The basic useful of crude oil refinery it's coke are as domestic fuel without calcining, producing of anodes, graphite, electrodes and producing of metals [15].

## 2. Experimental work

### 2.1. Materials

#### I- Reduced Crude Residue (R.C.R)

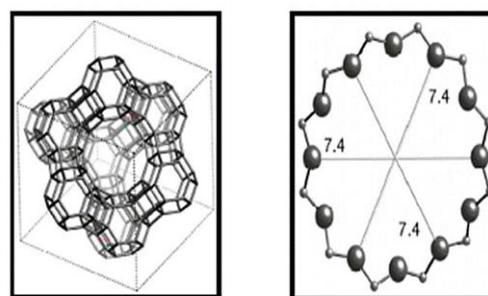
The reduced crude residue (RCR) product is one of the products of the atmospheric distillation tower in the crude oil refining process, as it has a higher molecular weight and a high boiling point and is characterized by containing light and medium petroleum derivatives, so the cracking process in general is capable of converting the heavy hydrocarbon compounds present in RCR into petroleum derivatives.

#### II- Characterization R.C.R

Reduced Crude Residue R.C.R Thermal cracking experiments were conducted on atmospheric residue (R.C.R) from Al-Diwaniya refinery in Iraq with physical properties are given in Tables 1 and Nitrogen as Inert gas used to cooling reactor and to leak check before any run.

**Table 1. Physical properties of Reduced Crude Residue R.C.C in petroleum Al-Divaneyah refinery**

Specification	Value
Specific gravity at 60/60 °F	0.9725
API	14.0
Flash point	90 °C
density	0.9718 gm/cm <sup>3</sup>
Viscosity @ 50 °C	250.1 CST
Sulfur Wt. %	4.62
Pour point °C	-7



**Figure 1. The typical framework structure of zeolite Y**

**Table 2. Specification of Zeolite HY**

Product name	Zeolite
Type	HY
Application	Catalyst
Appearance	Powder. White.
Odor	Odorless
Melting point	1000
SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> mole ratio	5.1
Na <sub>2</sub> O %	2.8
Unit cell size (Å)	24.5
Surface area (m <sup>2</sup> /g)	700
Country of Origin	The Netherlands

### III- Zeolite HY

In order to specify the nature and properties of the prepared zeolite in any method, many tests to be apply. The particle size, molecular structure, chemical composition and activity are possible to be known.

#### SEM analysis of Zeolite

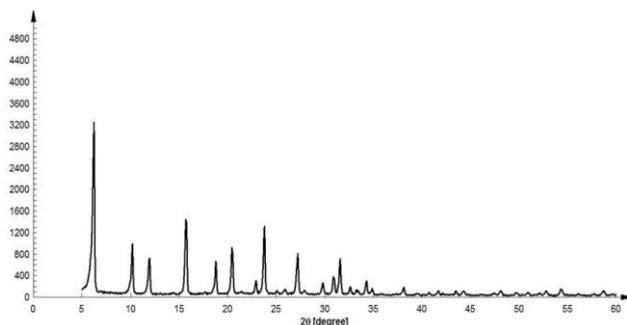
Figure 4 shows SEM study the morphology shape and size of the synthesized crystals. The SEM images of the samples show that the morphology of all the samples as expected for zeolite HY.

### Transmission infrared spectroscopy (FT-IR)

Infrared spectroscopy is commonly used to study the silica aluminate framework, hydroxyl group and also adsorbed molecules in zeolite.

### X-ray diffraction patterns (XRD)

X-ray used to explain the crystallinity, electronic microscopes image is clearing the particles size and shapes, and chemical analysis helps to understand the composition of particles. X-ray Powder Diffraction technique is the best method to define the crystallographic structure of zeolite. Each zeolite has their own specific pattern that can be used as references for the determination of solid crystal phase and it is used as fingerprint for every zeolite. This technique can signify whether the solid



sample is amorphous or crystalline phase, the degree of crystallinity and identification of phase present.

Figure 2. XRD for zeolite HY

## 2.2 Catalytic properties of the zeolites

In 1962 the zeolites were introduced by Mobil Oil Corporation as new cracking catalysts in refinery technology. They were characterized by top activity and selectivity in breaking and breaking with hydrogen. At the end of the 1960s, the concept of shape-selective catalysis with zeolites was introduced to petrochemistry (Electroforming process), and the zeolites became of higher importantly in catalysis research and applied catalysis. Zeolites advantages over conventional catalysts can be summarized as follows:

1. Crystalline and therefore precisely defined arrangement of  $\text{SiO}_4$  and  $\text{AlO}_4$  tetrahedra. This results in good reproducibility in production.
2. Shape selectivity: only molecules that are smaller than the pore diameter of the zeolite undergo reaction.
3. Controlled incorporation of acid centers in the intracrystalline surface is possible during synthesis and/or by subsequent ion exchange.
4. Up 300o C zeolite Y have acidities comparable to those of mineral acids.
5. Catalytically active metal ions can be uniformly applied to the catalyst by ion exchange or impregnation. Subsequent reduction to the metal is also possible.
6. Zeolite catalysts are thermally stable up to 600oC and can be regenerated by combustion of carbon deposits.

## 3. Experimental setup

As shown in fig. 3, the equipment consists the following items:

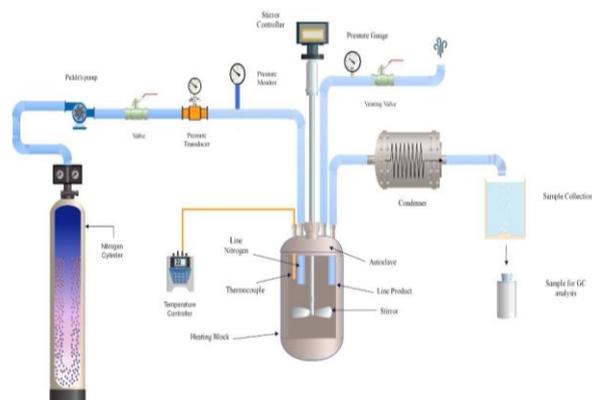


Figure 3. Schematic representation of the experimental procedures unit with controls system

- i. The reactor is made from 316 stainless steel at size approximately (1 liter).
- ii. Nitrogen bottle.
- iii. Vent valve.
- iv. Thermocouple.
- v. Condenser.
- vi. Temperature indicter.
- vii. Power switch.
- viii. Magnetic pump.
- ix. Product container storage (250 mm).
- x. Sensitive balance.
- xi. Dryer

### 3.1 Catalytic cracking test:

The test worked for catalytic cracking of Reduced Crude Residue consisted of a rise-pressure and temperature uncontained reactor. The schematic diagram of the test setup is explained in Fig. 1. The autoclave was used to conduct catalytic cracking tests. To ensure sample homogeneity, the autoclave was placed in a preheated vertically electric furnace with a magnetic stirrer. Wash the reactor with distilled water and then dry it in the dryer for a quarter hour. After placing and securing the reactor in the main apparatus, a pressure leak check was completed by using Nitrogen gas and charging the pressure to the reactor at 150 psi for 5 minutes. Following the leak test, the pressure in the reactor was vented, and the reactor was again charging with Nitrogen for 5 minutes to remove any remaining air via continuously sending out Nitrogen from the reactor.[13] Nitrogen gas was given to the system at a constant pressure via pressure regulators from Nitrogen cylinders.

The reactor in the unit was filled with 300 g of (R.C.R) with the dosage of catalyst its (0.1, 0.3, 0.5) gm, after that increasing the temperature by using electrical heater. The external valve for the reactor was closed before increasing the temperature for Reduced Crude Residue (R.C.R) was heated in an oven for each test. The sample of R.C.R sample and zeolite-HY was filled into a reactor at size (1000 ml) made from 316 stainless steel in electrical heating furnace with cover to maintain temperature were connected to power supply consumed about 1500 kW. The temperature of the autoclave was recorded/selected using by system control, manual

controller and Nitrogen. The pressure of the autoclave was recorded/selected by product valve, rpm for magnetic stirrer and if the pressure high over the set point will check valve working. The pipe line for production with (10 mm. i.d) also manufacture from 316 stainless steel.

The reactor was heated to the desired temperature at a rate of 25 C/ min. When the reactor temperature reached 120 °C, an electromagnetic stirrer began mixing the contents at 500 rpm [13]. The stirrer speed can be selected by a DC change speed selector. Bath water keep on the magnetic stirrer at safe temperature level during experimental test to maintain magnetic in stirrer. Unit temperature in reactor of 300 °C recorded the starting point of the reaction, and the residence time was calculated at 300 °C. [13] The product from the yield valve was undergo to the bath water to condensing the gas product and cooling the liquid product. After completion, the heating jacket was turned off and removed. The reactor was then leave for 5 hours to cool to room temperature for tests without quenching.[13]

Once at room temperature, the uncondensed gases, primarily C1 to C3 .Each test's liquid yield was calculated inter alia the mass of the liquid phase product and the mass of the feed. The liquid production was thoroughly mixed, and the API gravity was determined at ambient temperature by a 250 ml pycnometer. The viscosity measured at 50 °C for heavy liquid or 40 °C for light liquid by a Brookfield digital viscometer equipped with a temperature-controlled water bath for the liquid phase product.[13]

The supernatant was putted in special package, and its mass him were measured. The density and specific gravity of the liquid product were also measured beside API, flash point. The experiment is then repeated several times based on the difference in pressure, temperature, and contact time. Each gas product for any test was collected in a gas pack and analyzed using a gas chromatography (GC). Weighted the remaining coke and liquid product, as well as wash the reactor.

The conversion for all the test were obtained by using the following equations:

$$\% \text{ Conversion} = \frac{\text{Mass}_{\text{Feed}} - \text{Mass}_{\text{Residue}}}{\text{Mass}_{\text{Feed}}} * 100\% \tag{1}$$

Yield is defined as a mass of the cracking in the liquid phase product divided by the mass of crude oil atmosphere residue (R.C.R) feed:

$$\text{Mass}_{\text{Gas Product}} = \text{Mass}_{\text{Feed}} - (\text{Mass}_{\text{Liquid product}} + \text{Mass}_{\text{Coke}} + \text{Mass}_{\text{Residue}}) \tag{2}$$

$$\text{Liquid}_{\text{yield}} = \frac{\text{Mass of Liquid Product}}{\text{Mass}_{\text{Feed}}} * 100\% \tag{3}$$

$$\text{Gas}_{\text{yield}} = \frac{\text{Mass of gas product}}{\text{Mass of feed}} * 100\% \tag{4}$$

$$\text{Coke}_{\text{yield}} = \frac{\text{Mass of Coke Product}}{\text{Mass}_{\text{Feed}}} * 100\% \tag{5}$$

$$\text{Residue}_{\text{yield}} = \frac{\text{Mass of Residue Product}}{\text{Mass}_{\text{Feed}}} * 100\% \tag{6}$$

### 3.2 Experimental Design

The response surface methodology (RSM) was used to assess the effects of process selection on R.C.R thermal cracking and obtain total conversion

and physical property values for the product. RSM is a set of statistical and mathematical techniques for modeling and analyzing problems in which a response of interest is influenced by many variables and the aim is to optimum analysis this response.

The procedure was investigated using Box-Behnken. The response surface model, which is based on Box-Behnken, is the most widely used design method for three variable or more formation in environmental processes. Furthermore, Box-Behnken provides excellent prediction of linear and square interaction effects of variables affecting the chosen mechanism. Box-Behnken requires three level of points: center points, axial points, and cube points derived from factorial nature. The independent variables were the pressure (A), temperature (B) and reaction time (C). The factors named and levels are shown in Table 2.

**Table 3. the three factors: Pressure, Temperature, Time and their level**

Factor	Code	Unit	Low level (-1)	Medium level (0)	High level (+1)
Pressure	A	°C	1	3	5
Temperature	B	Min	350	400	450
Time	C	Bar	60	90	120
Dosage	D	gm	0.1	0.3	0.5

## 4. Results and Discussion

We measured the density from specific gravity (ASTM) table by API standard number. The °API gravity at 60 °F was measured with a 250 mL specific gravity flask.

### 4.1 Specific gravity

A specific gravity scale measured by the American Petroleum Institute (API) for selection the number density of various petroleum liquids phase production. i.e. API = (141.5/sp.gr) -131.5.

### 4.2 Effect of temperature on the experimental

Fig. 4 and table 4 shows the relation between the liquid yield and the variable condition for every run. From this figure the difference in yield between runs increased with increased temperature for the same time, dosage and pressure with constant rotary per minute for magnetic stirrer.

Fig. 5 and table 4 shows the relation between the gas yield and the variable condition for every run. From this figure the difference in yield between runs increased with increased temperature for the same time, dosage and pressure with constant rotary per minute for magnetic stirrer.

Fig. 6 and table 4 shows the relation between the coke yield and the variable condition for every run. From this figure the difference in yield between runs increased with increased temperature for the same time, dosage and pressure with constant rotary per minute for magnetic stirrer. Fig. 7 and table 4 shows the relation between the residue and the variable condition for every run. From this figure the difference in residue between runs decreased with increased temperature for the same time, dosage and pressure with constant rotary per minute for magnetic stirrer.

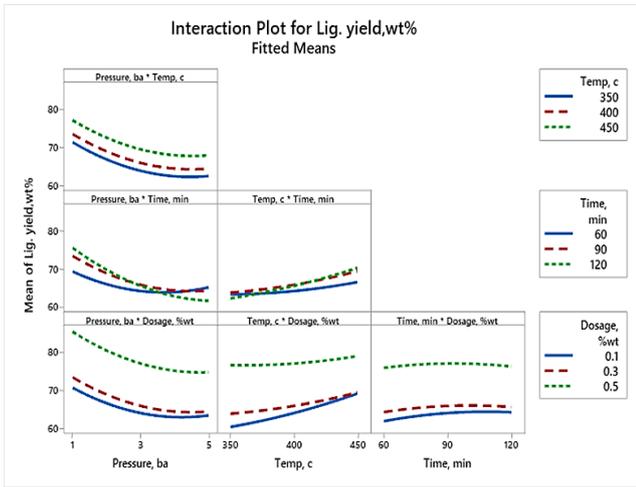


Figure 4. The 2D mean of liquid yield with run condition (Temperature, time, dosage, and pressure)

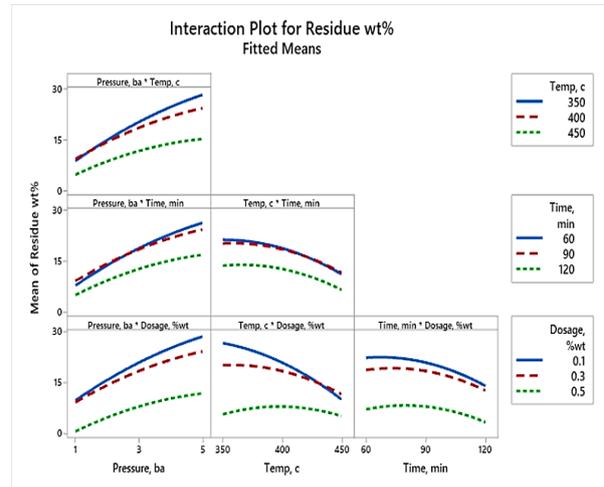


Figure 6. The 2D mean of coke yield with run condition (temperature, time, dosage and pressure)

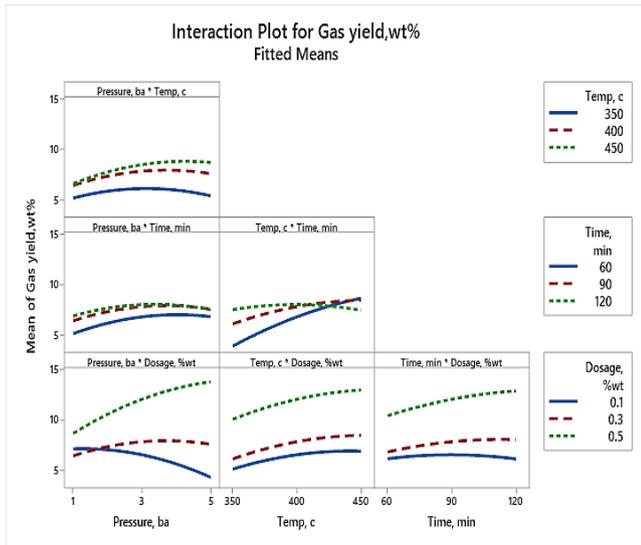


Figure 5. The 2D mean of gas yield with run condition (Temperature, time, dosage, and pressure)

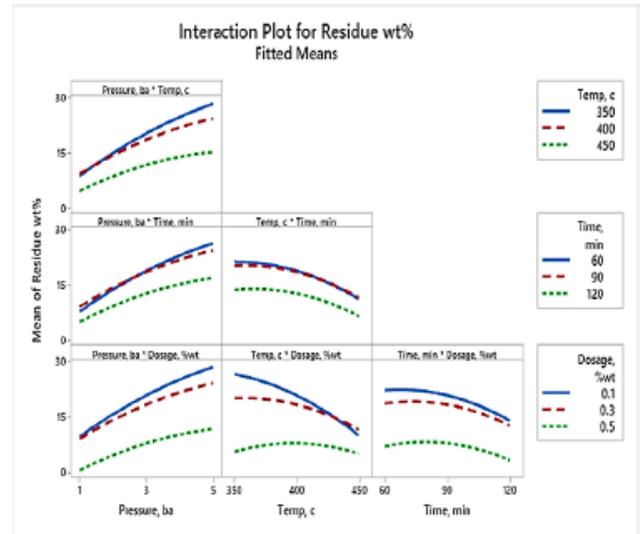


Figure 7. The 2D mean of residue with run condition (temperature, time and pressure)

From table 5 show the relation between the run and physical properties there for we note increased quality with increased temperature with the same condition for pressure, dosage and time depended on API, density and sp.gr.

4.3 Effect of pressure on the experimental

Fig. 4 and table 4 shows the relation between the liquid yield and the variable condition for every run. From this figure the difference in yield between runs the yield decreased with increased pressure for the same time, dosage and temperature with constant rotary per minute for magnetic stirrer. Fig. 5 and table 4 shows the relation between the gas yield and the variable condition for every run.

From this figure the difference in yield between runs the yield decreased with increased pressure for the same time, dosage and temperature with constant rotary per minute for magnetic stirrer. Fig. 6 and table 4 shows the relation between the coke yield and the variable condition for every run. From this figure the difference in yield between runs increased with increased pressure for the same time, dosage and temperature with constant rotary per minute for magnetic stirrer. Fig. 7 and table 4 shows the relation between the residue and the variable condition for every run. From this figure the difference in residue between runs the residue increased with increased pressure for the same time, dosage and temperature with constant rotary per minute for magnetic stirrer. From table 5 show the relation between the run and physical properties there for we note increased quality with increased pressure with the same condition for temperature, dosage and time depended on API, density and sp.gr .

**Table 4. The experimental results obtained by catalytic cracking based on Box-Behnken**

Run	Pressure, bar	Temp, c	Time, min	Dosage, %wt	liquid yield	Gas yield	Coke	Residue
					%wt	%wt	%wt	%wt
1	3	450	120	0.3	68.51	07.32	15.81	08.36
2	3	400	120	0.5	78.81	13.07	04.33	03.79
3	1	400	120	0.3	81.37	06.90	10.43	01.30
4	5	400	060	0.3	63.46	06.02	02.11	28.41
5	3	400	090	0.3	65.92	07.83	07.78	18.47
6	3	350	090	0.1	65.70	04.54	03.92	25.84
7	3	450	090	0.5	77.68	12.75	05.11	04.46
8	3	350	120	0.3	57.78	06.56	24.36	11.30
9	1	400	090	0.5	81.31	08.22	06.67	03.80
10	3	400	060	0.5	80.15	10.12	05.48	04.25
11	5	400	090	0.1	65.50	05.26	02.08	27.16
12	1	350	090	0.3	70.46	06.23	13.49	09.82
13	3	350	060	0.3	63.06	04.57	11.20	21.17
14	1	400	060	0.3	69.22	03.91	21.64	05.23
15	3	450	090	0.1	71.52	05.89	15.13	07.46
16	3	400	090	0.3	65.92	07.83	07.78	18.47
17	1	400	090	0.1	70.97	06.98	09.22	12.83
18	3	350	090	0.5	78.33	10.26	04.76	06.65
19	3	400	090	0.3	65.92	07.83	07.78	18.47
20	1	450	090	0.3	76.63	07.30	12.85	03.22
21	5	350	090	0.3	61.26	04.93	04.43	29.38

**Table 5. Physical properties for liquid product**

Run	Pressure, bar	Temp, c	Time, min	Dosage, %wt	density	Sp.gr	API
1	3	450	120	0.3	0.7888	0.7892	47.8
2	3	400	120	0.5	0.7258	0.7260	63.4
3	1	400	120	0.3	0.8310	0.8314	38.7
4	5	400	060	0.3	0.7823	0.7826	49.3
5	3	400	090	0.3	0.8124	0.8128	42.6
6	3	350	090	0.1	0.8708	0.8713	30.9
7	3	450	090	0.5	0.7225	0.7227	64.3
8	3	350	120	0.3	0.8185	0.8189	41.3
9	1	400	090	0.5	0.7729	0.7732	51.5
10	3	400	060	0.5	0.7322	0.7324	61.7
11	5	400	090	0.1	0.8489	0.8493	35.1
12	1	350	090	0.3	0.8530	0.8534	34.3
13	3	350	060	0.3	0.8319	0.8324	38.5
14	1	400	060	0.3	0.8494	0.8504	34.9
15	3	450	090	0.1	0.8525	0.8529	34.4
16	3	400	090	0.3	0.8124	0.8128	42.6
17	1	400	090	0.1	0.8855	0.8860	28.2
18	3	350	090	0.5	0.7402	0.7405	59.6
19	3	400	090	0.3	0.8124	0.8128	42.6
20	1	450	090	0.3	0.8433	0.8438	36.2
21	5	350	090	0.3	0.7928	0.7932	46.9

## 5. Conclusions

Catalytic cracking of The Reduced Crude Residue R.C.R was carried out in a batch reactor in an autoclave with different process selection, temperature about 350–450 °C, catalytic cracking time 60 -120-minute, dosage of catalyst (0.1-0.5) gm and pressure 1- 5 bar in the existence of Nitrogen. X-ray used to calculate the relative crystallinity of catalysts zeolite HY. The experimental design was utilized to optimize liquid yield, gas yield, Coke and Residue by using the four parameters Pressure, Temperature, time and Dosage of catalyst. A low conversion occurs in the liquid yield, gas yield and Coke yield and increasing in the heavy residue amount of the cracking process when decreased the Time and temperature of the process with constant pressure therefore to prevent the thermal cracking from fouling it necessary to maintain the system at high temperature and residence time as possible. The highest liquid yield was 71.05 at pressure 1 bar and temperature 400C and 120 minutes, and the lowest liquid yield was 44.58 at 5 bar and 350 C and 90 min, therefore the optimum conditions is that keep high liquid yields which mean run number 10. An increasing the pressure decreases (with Keeping the values of the other variables (Temperature and time)), it noticed all the responses of the process will increased that liquid yield will increase from 48.14 to 53.32 and the Gas yield also increased from 4.32 to 5.58, also the Coke yield was increased from 12.0 to 18.1. Run 8 and run 9 show optimum conditions of the cracking process because it gives 0 residue and the highest value of the liquid, gas and Coke yields ranging from 54.87 to 69.21 and 5.60 to 6.0 and 18.2 to 24.79 respectively. The optimum run represents by run number 2 where the of operation conditions were 3 bar Pressure, 400 °C Temp, 120-minute time and 0.5 %wt as catalysts dosage, where the liquid and the gas yield was 78.81 and 13.07 respectively as weight percentage, also the coke and residue percentages were 4.33 %wt and 3.79 %wt. respectively.

### Authors' contribution

All authors contributed equally to the preparation of this article.

### Declaration of competing interest

The authors declare no conflicts of interest.

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This study didn't receive any specific funds.

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