

Research Article

# Modelling Gas and Gasoline Production from Cottonseed Oil on Fixed-fluidized Bed Catalytic Cracking over Nbfeapso-5 Molecular Sieve

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## Abstract

The potential for producing hydrocarbons from the conversion of biofuels has been the focus of attention in recent years. In a preliminary study, we observed that it was possible to produce biofuels from cottonseed oil by fixed-fluidized bed catalytic cracking at ambient pressure, using fluid catalytic cracker equilibrium catalyst. In the present work, the production of biofuels from cottonseed oil by fixed-fluidized bed catalytic cracking at ambient pressure, using niobium containing aluminophosphate molecular sieve (NbFeAPSO-5), was studied. The effect of reaction temperature (400-500 °C), catalyst-to-oil ratio (6-10) and residence time (50-90 s) were studied. The response surface methodology was used to determine the optimum values of the operating variables for maximum yield of biofuels in the liquid product obtained. The optimum values of reaction temperature of 420.2 °C, catalyst to oil ratio of 8.8 g/g and the residence time of 51 seconds were obtained for maximum yield of light fuel oil ( $\leq 360^\circ\text{C}$ ) 68.6 wt%, gasoline fraction ( $\leq 205^\circ\text{C}$ ) 37.7 wt% and minimum yield of gas 15.6 wt%. The catalytic cracking of cottonseed oil in a fixed-fluidized bed reactor produced a liquid product rich in gasoline and diesel fraction.

## Keywords

Hydrocarbons, Biofuels, NbFeAPSO-5 Molecular Sieve, Fixed-fluidized Bed Reactor, Response Surface Methodology

## 1. Introduction

Currently, biofuels are being investigated as alternative sources of a variety of hydrocarbons. The production of biofuels such as diesel and gasoline fractions as an alternative fuel obtained from natural oils or fats are environmentally friendly since they are free of nitrogen and sulfur compounds. With biofuel, the greenhouse effect and air pollution can be minimized [1]. Over the years, vegetable oils have been substituted for diesel for use in engines but this has led to

problems such as carbon deposits, oil ring sticking and gelling of the lubricating oil. Because of such problems, many works have been centered on the conversion of these oils to a form that is similar to current fuels. One such fuel, which is currently gaining much attention, is biodiesel. This is a variety of ester based oxygenated fuels made from vegetable oils or animal fats.

There are several methods for the conversion of vegetable

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oils to biodiesel of which the most common is the transesterification process, in which an alcohol is reacted with the oil to form esters and glycerol [2]. Another method for the conversion of vegetable oils to a usable fuel product is by catalytic cracking reactions. Plant oils, animal fats, and recycled cooking greases can be transformed into biofuel using a direct upgrading process such as the catalytic cracking technology [3]. This is currently used in the petroleum and petrochemical industry to convert high molecular weight oil components to lower molecular weight ones which can be used directly or blended for use as fuel [4]. The reaction process usually involves the mixing of catalysts with the oil feed at high temperature in a fluid catalytic cracker (FCC) unit. Here the hydrocarbon product is collected and the spent catalyst is directed to a regenerator which oxidizes the coke that has collected on it to CO, CO<sub>2</sub> and H<sub>2</sub>O. [5].

In the case of catalytic cracking of palm oil, Twaiq et al. [6] have reported extensive work on catalytic cracking of palm oil using micro- and mesoporous materials as cracking catalysts in a microreactor. Problems associated with coking and limited feed/ catalyst contact within the reactor make continuous processing difficult in fixed-bed reactors. Without good contact of feed and catalyst, the formation of large amounts of residue are likely and scale-up to industrial scale is not feasible.

The addition of aluminophosphate-based molecular sieve catalysts to silica-alumina has also been reported [6]. For instance, the addition of SAPO-11 has resulted in gasoline and gasoline-plus-alkylate yields similar to those obtained in the absence of the SAPO additive. However, the paraffins in the gasoline fraction obtained with the SAPO-11 additive showed a higher iso/normal hydrocarbon ratio and therefore higher.

In order to predict the optimum operating conditions in a process to obtain the highest yield of desired product, a statistical approach has been used by several researchers [6-8]. There are classical as well as statistical methodologies available for process optimizations. Statistical methodologies involve the use of mathematical methods for designing and analysing results. Response surface methodology (RSM) is a statistical modelling technique employed for multiple regression analysis using quantitative data obtained from properly designed experiments to solve multivariable equations simultaneously [9-12]. Response surface methodology (RSM), originally described by Box and Wilson [13], is an effective statistical technique for optimizing complex processes. It is widely used in optimizing the process variables. The basic theoretical and fundamental aspects of RSM have been reviewed [14, 15]. It is superior to the traditional approach in which optimization studies are carried out by varying one parameter at a time while keeping others constant.

Recently, response surface methodology was applied for the optimization of catalytic cracking palm oil to biofuels with nitrogen as carrier gas and REY cracking catalyst [8].

The objective of this work is to explore optimum operation

conditions where cottonseed oil is catalytically cracked under realistic conditions using a fixed-fluidized bed reactor in which the catalyst is fluidized and confined in the reaction space [16, 17]. For this purpose, experimental design and response surface methodology were chosen as tools for the optimization of process operating conditions for the production of gasoline fraction ( $\leq 205$  °C) and light fuel oil ( $\leq 360$  °C) in liquid hydrocarbon fuel from cottonseed oil. The light fuel fraction was produced from cottonseed oil using NbFeAPSO-5 molecular sieve as cracking catalyst in a fixed fluidized bed reactor.

## 2. Experimental Section

### 2.1. Material

Refined cottonseed oil (SODECOTON, North Cameroon), is composed predominantly of oleic acid (C18:1 unsaturated fatty acid), 65 wt% unsaturated and 33 wt% saturated fatty acids.

### 2.2. NbFeAPSO-5 Molecular Sieve

In our previous work [18] we reported the synthesis of NbFAPO-5 and NbFeAPSO-5 molecular sieve. The acidic properties of NbFeAPSO-5 molecular sieve are listed in Table 1. Both Brönsted and Lewis acidity are found in the synthesized sample.

Table 1. Lewis and Brönsted acid in NbFAPSO-5.

	TL	TB	SL	SB	WL	WB
NbFAPSO-5	28.646	28.809	0	0	9.512	28.809

TL= total Lewis acid, TB= total Brönsted acid, SL= strong Lewis acid, SB= strong Brönsted acid, WB= weak Brönsted acid, WL = weak Lewis acid

### 2.3. Apparatus

A fixed-fluidized bed reactor was used in the experiments of the catalytic cracking of cottonseed oil, and the schematic diagram is shown in Figure 1. It consists of four sections, (a) sample injection section; (b) temperature controller section; (c) reaction section; and (d) products collection section. The unit is operated manually and automatically. Main operation parameters can be adjusted manually at any time. The catalyst is sucked in the reactor by vacuum system. For each experiment, constant grams of catalyst were loaded into the reactor. An invariable amount of deionized water was pumped into pre-heater to form steam, which was used to fluidize the catalyst and atomize cottonseed oil. Once the reactor param-

eters reached a steady state, Cottonseed oil pumped by another pump mixed with the steam, preheated to approximately 350 °C in a pre-heater, and then entered into the reactor, where cottonseed oil made contact with the fluidized catalyst and reactions took place. The oil gas after reaction was cooled and separated into the liquid sample and the gas sample.

## 2.4. Operating Conditions

The operating conditions for the main catalytic cracking tests are summarized in Table 2.

Table 2. Operating conditions for cracking tests.

Parameter	value
Temperature of reactor, °C	400-500
Temperature of pre-heater, °C	350
Temperature of incubator, °C	70
Catalyst loading, g	80
Residence time of oil gas, s	50-90
Catalyst-to-oil weight ratio	6-10
Water inflow, g/min	3.5

## 2.5. Experimental Design and Mathematical Model

The optimum operating conditions (independent variables)

were determined to maximize the product yield (response). A 5-level 3-factors central composite design (CCD) was employed in this study, requiring 20 experiments. The variables and their respective levels are presented in Table 3.

Table 3. Real and coded independent variables.

Factor	Factor code	Unit	Low (-1)	Central (0)	High (+1)
Reaction temperature (X <sub>1</sub> )	x <sub>1</sub>	°C	400	450	500
Cat/oil ratio (X <sub>2</sub> )	x <sub>2</sub>	g/g	6	8	10
Residence time (X <sub>3</sub> )	x <sub>3</sub>	s	50	70	90

These variables were coded according to equation (1):

$$x_i = (x_i - x_0) / \Delta x_i (i = 1, 2 \dots k) \quad (1)$$

where  $x_i$  is the dimensionless coded value of the variable  $x_i$ ,  $x_0$  is the actual value at the center point,  $\Delta x_i$  is the step change of  $x_i$  corresponding to one unit of  $x_i$ . In order to carry out a comprehensive analysis of the catalytic cracking process in the fixed fluidized bed reactor, three main dependent responses were considered, i.e. yield of gas, yield of gasoline and yield of light fuel oil ( $\leq 360$  °C). Yields of the products are defined as:

$$\text{Yield (wt\%)} = \frac{\text{Product (g)}}{\text{Cottonseed oil Feed (g)}} \times 100\% \quad (2)$$

The data obtained were fitted to a second-order polynomial equation. For three independent variables, the quadratic models were expressed according to equation (3):

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \quad (3)$$

where Y is the predicted response,  $\beta_0$  model constant;  $x_1$ ,  $x_2$  and  $x_3$  are the coded values of the independent variables;  $\beta_1$ ,

$\beta_2$ , and  $\beta_3$  are linear coefficients;  $\beta_{12}$ ,  $\beta_{13}$ ,  $\beta_{23}$  are cross product coefficients; and  $\beta_{11}$ ,  $\beta_{22}$ ,  $\beta_{33}$  are the quadratic coefficients [7]. The coefficients of the equation were determined by non-linear multiple regression analysis using the software Essential Regression. Model terms were selected or rejected based on the P-values with 95% confidence level. Three-dimensional plots were obtained based on the effect of the three factors to study the interaction among different factors and their levels.

## 2.6. Validation of the Model

In order to determine the accuracy of the models, the effects of the three factors (temperature, cat/oil and residence time) on yields of gas, gasoline and light fuel oil were randomly selected within the design space. Six sets of experiments were

carried out [18, 19].

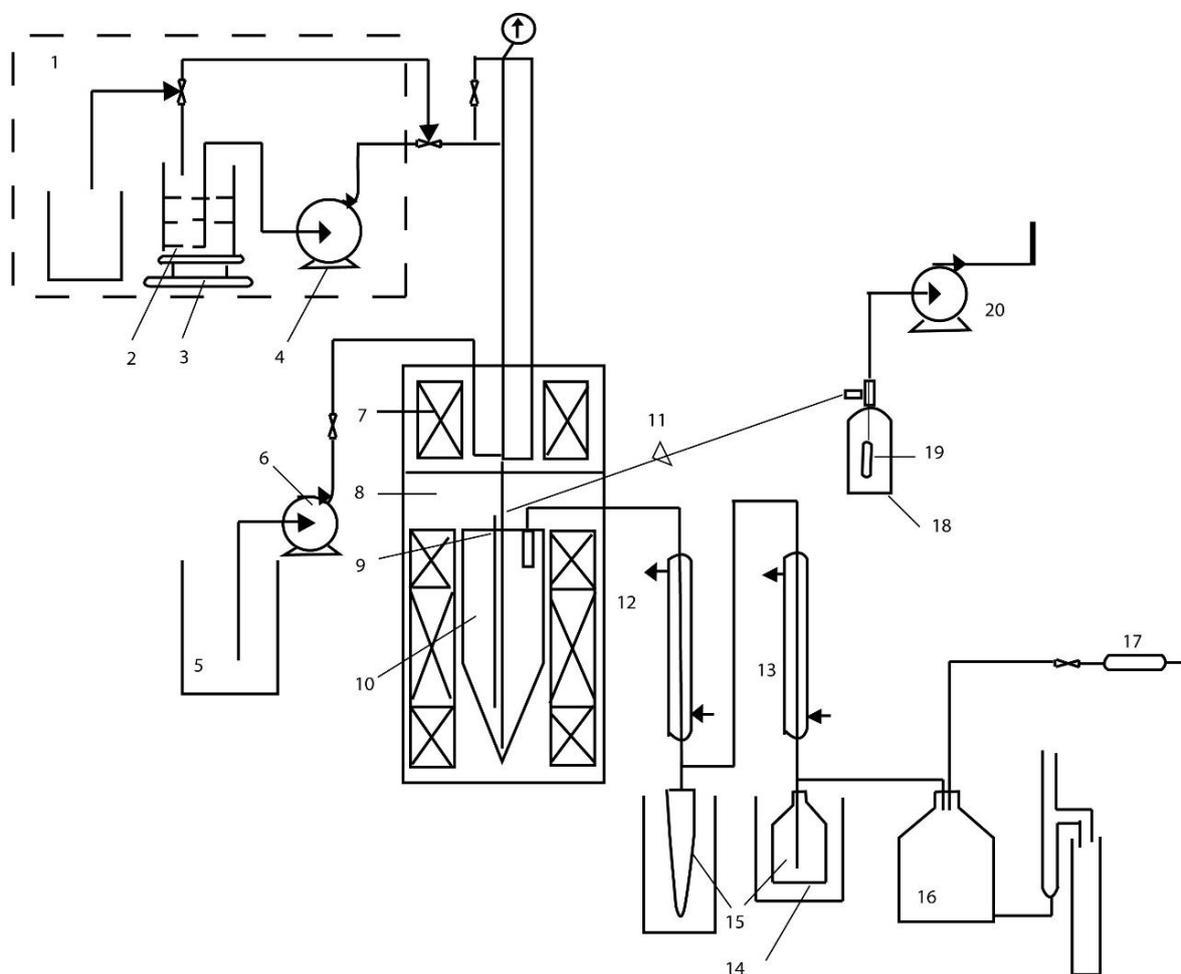
## 2.7. Products Analysis

### 2.7.1. Hydrocarbon Gas Products Composition Analysis

Gas fraction (C1-C5+ hydrocarbons) was analysed by GC1102N gas chromatography. Because C5+ hydrocarbons belong to gasoline fraction, they were analysed as gasoline components.

### 2.7.2. Liquid Product Analysis

Liquid products include gasoline, diesel oil and residue. Liquid fraction was analysed by simulated distillation method using a Shimadzu GC-14A Gas chromatograph.



**Figure 1.** Schematic diagram of the fixed-fluidized bed: 1, incubator; 2, raw oil tank; 3, balance; 4, raw oil pump; 5, water tank; 6, water pump; 7, preheater; 8, furnace; 9, thermocouple; 10, reactor; 11, catalyst exit; 12, filter; 13, condenser; 14, ice bath; 15, liquid collector; 16, brine bottle; 17, sampler; 18, buffer bottle; 19, filter; 20, vacuum pump.

### 3. Results and Discussion

#### 3.1. Design of Experiments (DOE)

The catalytic cracking of cottonseed oil created organic liquid products (OLP), gas, coke and water. Water fraction was not surveyed because it was mixed with condensed water as carrier gas. Furthermore, its presence was not an important parameter to be predicted using the DOE. There was an insignificant amount of coke formation in this study. Gasoline ( $\leq 205\text{ }^{\circ}\text{C}$ ) and light fuel oil ( $\leq 360\text{ }^{\circ}\text{C}$ ) fraction yield were the target functions in optimizing operation parameters. Experimental matrix and results are presented in Table 4.

Table 4. Experimental matrix and results.

Temperature (°C)	Cat/oil ratio (g/g)	Residence time (s)	Y <sub>gas</sub> (wt%)	Y <sub>gasoline</sub> (wt%)
400	6	50	8.5	26.3
400	6	90	8.5	26.1
400	10	50	12.2	28.3
400	10	90	16.1	28.1
425	8	70	13.7	28.2

Temperature (°C)	Cat/oil ratio (g/g)	Residence time (s)	Y <sub>gas</sub> (wt%)	Y <sub>gasoline</sub> (wt%)
450	7	70	16.6	28.9
450	8	60	16.0	32.3
450	8	80	17.0	31.2
450	9	70	18.3	31.6
475	8	70	17.1	28.8
500	6	50	24.1	32.0
500	6	90	24.6	31.4
500	10	50	24.5	33.7
500	10	90	28.8	31.2
450	8	70	16.9	30.3
450	8	70	16.9	30.4
450	8	70	16.8	30.4
450	8	70	16.2	30.1
450	8	70	16.4	30.5
450	8	70	16.3	30.8

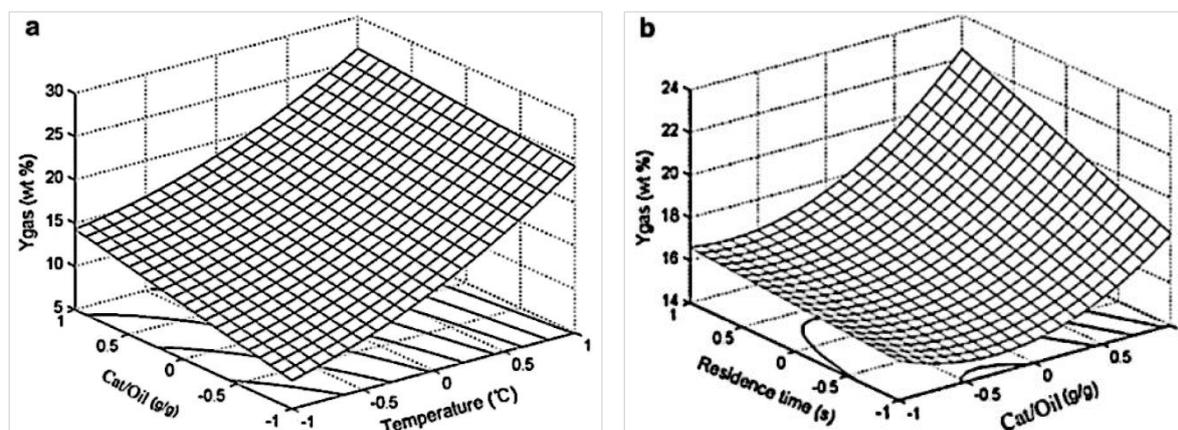


Figure 2. Response surface plot showing the effect of reaction temperature and cat/oil on gasoline yield.

#### 3.2. Statistical Analysis

Since several variables were studied, the conditions which significantly affected the product distribution were identified statistically. In order to quantify the curvature effects, the data from the experimental results were fitted to higher degree polynomial equations. Analysis of variance (ANOVA) in Table 5 showed that experimental data are best fitted into a quadratic equation at 95% level of confidence.

Table 5. ANOVA for response surface quadratic model.

	Y <sub>gas</sub>	Y <sub>gasoline</sub>
R <sup>2</sup>	0.9794	0.9346
Adj.R <sup>2</sup>	0.9698	0.9045
C.V.%	5.230	2.249

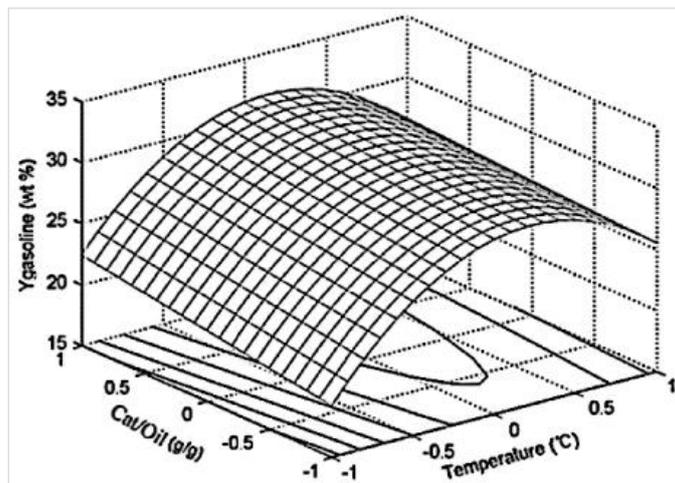
	$Y_{\text{gas}}$	$Y_{\text{gasoline}}$
Model F-value	102.9	30.98
P-value	<0.0001	<0.0001
Lack of fit	0.0511	0.0576

its significance. The quadratic models for yields of gas, gasoline and light fuel oil ( $\leq 360\text{ }^{\circ}\text{C}$ ) are given below.

$$y_{\text{gas}} = 16.26 + 6.988x_1 + 2.053x_2 + 1.204x_3 - 0.9625x_1x_2 + 0.8413x_2x_3 + 2.016x_2^2 \quad (4)$$

$$y_{\text{gasoline}} = 30.34 + 2.565x_1 + 1.041x_2 - 0.4765x_3 - 0.5625x_1x_2 - 6.977x_1^2 + 6.023x_3^2 \quad (5)$$

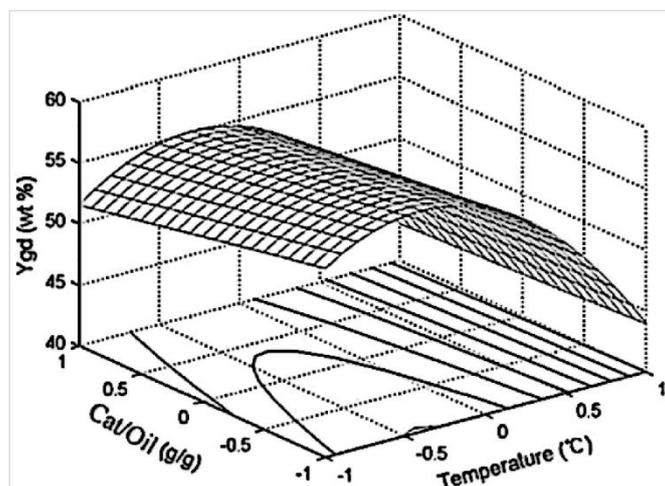
ANOVA for quadratic model was carried out to establish



**Figure 3.** Response surface plot showing the effect of reaction temperature and cat/oil on gasoline yield.

The regression equations indicated that the coefficient of determination ( $R^2$ ) were 0.9794, 0.9346 and 0.9361 respectively. The Prob > F-value was less than 0.0001, therefore, the model  $Y_{\text{gas}}$  terms  $x_1$ ,  $x_2$ ,  $x_3$ ,  $x_1x_2$ ,  $x_2x_3$ ,  $x_2^2$  were considered as significant after elimination of insignificant variables and their interactions. The models  $Y_{\text{gasoline}}$  and  $Y_{\text{gd}}$  were treated with the same means. The adjusted  $R^2$ , which was more suitable for comparing models with different numbers of independent variables, ranged from 0.9698 to 0.9045.

These model F-values were 102.9, 30.98 and 31.77. High F-value and non-significant lack of fit indicated that the models fitted well. Adequate precision value, which measured the 'signal to noise ratio', was 38.57, which indicated an adequate signal. A ratio greater than 4 is desirable [10]. Thus, this model could be used to navigate the design space. Low values of the coefficient of variation indicated good precision and reliability of the experiments [12].



**Figure 4.** Response surface plot showing the effect of reaction temperature and cat/oil on gasoline yield.

The interaction effects were analysed using 3D response surface plots. Figure 2a shows the effect of temperature and cat/oil on gas yield; the gas yield was found to increase with increasing reaction temperature and cat/oil ratio. Figure 2b shows the effect of residence time and cat/oil ratio on the gas yield. The gas yield was found to increase with increasing residence time; however, it first decreases then increases with temperature, leaving an optimum value in a temperature range of 425-450 °C. Figure 3 shows the effect of the reaction temperature and cat/oil ratio on the gasoline yield. Light fuel oil yields were found to decrease with the increase of cat/oil from 6 to 10 g/g. However, it first increased then decreased leaving an optimum value in a temperature range of 420-440 °C. Figure 4 shows that gasoline yield decreases with an increase of cat/oil from 6 to 10 g/g.; however, it first increases then decreases, leaving an optimum value in a temperature range of 430-460 °C.

### 3.3. Process Optimization

In this study, numerical optimization is employed; it is used comprehensively in engineering, science, and business by focusing on the methods that are best suited to practical problems. In the numerical optimization, yield of light fuel oil ( $\leq 360$  °C) and yield of gasoline ( $\leq 205$  °C) is set to a maximum range whereas gas yield is kept to as minimum as possible. The optimization constraints are summarized in Table 6.

Table 6. Optimization Constraints.

Constraints	Lower limit	Upper limit
Name		

Constraints	Lower limit	Upper limit
Temperature	400	500
Cat/oil	6	10
RT	50	90
Y <sub>gas</sub>	7.5	28.8
Y <sub>gasoline</sub>	25.1	33.7

The optimum values of reaction temperature of 426.2 °C, cat/oil of 7.8 g/g and residence time of 50.2 s were obtained for maximum yield of light fuel oil ( $\leq 360$  °C) 65.6 wt%, gasoline fraction ( $\leq 205$  °C) 33.7 wt% and minimum yield of gas 11.6 wt%. The yields of light fuel oil and gasoline fraction are respectively 12.2 wt% and 0.2 wt% more than those achieved by Tamunaidu et al. [8].

### 3.4. Validation of the Models

A second set of experiments was performed to validate the developed equations. The models were validated for the three variables within the design space. The results of a random set of six experiments (Table 7) showed clearly that the experimentally determined production values were in close agreement with the statistically predicted ones. Only gaseous yield relative error is max. 7.7% at temperature 425 °C, cat/oil 10 g/g, residence time 90 s. The good correlation between the experimental and estimated responses verifies the existence of maximum points and the accuracy of the mathematical models.

Table 7. Validation of models using different levels of temperature ( $X_1$ ), cat/oil ( $X_2$ ), residence time ( $X_3$ ).

Sample.	$X_1$	$X_2$	$X_3$	Y <sub>gas</sub>			Y <sub>gasoline</sub>		
				Pred. <sup>a</sup>	Obs. <sup>b</sup>	Err.(%). <sup>c</sup>	Pred. <sup>a</sup>	Obs. <sup>b</sup>	Err.(%). <sup>c</sup>
1	430	8	50	12.3	12.5	1.6	34.6	33.9	-2.0
2	440	8	70	14.9	15.2	2.0	29.5	29.1	-1.4
3	470	9	50	18.8	18.5	-1.6	37.1	37.9	2.2
4	480	7	80	20.6	21.5	4.4	30.3	31.2	3.0
5	425	10	90	19.4	17.9	-7.7	34.1	33.6	-1.5
6	460	6	60	17.6	17.4	-1.1	31.4	31.6	0.6

a Predicted. b Observed. c Error

## 4. Conclusions

The catalytic cracking of cottonseed oil in a fixed-fluidized bed reactor produced a liquid product rich in gasoline and diesel fraction. In this process temperature and cat/oil ratio are important variables. The optimum values of reaction temperature of 426.2 °C, cat/oil of 7.8 g/g and residence time of 50.2 s were obtained for maximum yield of light fuel oil ( $\leq 360$  °C) 65.6 wt%, gasoline fraction ( $\leq 205$  °C) 33.7 wt% and minimum yield of gas 11.6 wt%. The quadratic models proposed were in good agreement with the experimental data within 95% confidence. Catalytic technology can be used for cracking cottonseed oil to produced value added products such as gasoline and diesel. The response surface model can adequately predict the production of gas and gasoline fractions.

## Abbreviations

NbFeAPSO-5	Niobium Containing Aluminophosphate Molecular Sieve FeAPSO-5 Type Structure
FAPSO-5	Aluminophosphate Molecular Sieve FAPSO-5 Type Structure
FCC	Fluid Catalytic Cracker
RSM	Response Surface Methodology
SODECOTON	Cameroon Cotton Development Corporation
CCD	Central Composite Design
Cat	Catalyst
GC	Gas Chromatography
DOE	Design of Experiments
OLP	Organic Liquid Product
ANOVA	Analysis of Variance

## Conflicts of Interest

The authors declare no conflicts of interest.

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