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Determining the Characteristics of Biodiesel from a Mixture of Crop Mustard and Edible Waste Oil using heterogeneous Calcium Oxide Catalyst Prepared from Fish bones and Predicting the Efficiency of Biodiesel using Taguchi Statistical Program Model

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ABSTRACT

In this study, in order to produce biodiesel fuel from mixture of crop mustard and edible waste oil, ester exchange method with heterogeneous calcium oxide catalyst prepared from fish bones was used as a catalyst. Then, the effect of the contribution of important and effective factors on the molar ratio of oil to methanol, catalyst percentage, reaction temperature and reaction time on the amount of biodiesel production was investigated using Taguchi experiment design method. In biodiesel production method the optimum reaction, it can be seen that the effect of this parameter is strongly effective in the production of biodiesel with a ratio of (1:12), that this molar ratio of oil to methanol (1:12) has the best efficiency. According to the results, the amount of (0.3%) calcium oxide catalyst prepared from fish bones can be considered as the maximum value in biodiesel production. It was observed that, with increasing the reaction temperature from (55°C to 65°C), the amount of biodiesel production increases by about 8% and with the continuation of increasing the temperature from (65°C to 75°C), with a sharp decrease in production (about 30%) We face. As a result, temperature for biodiesel production is (65°C), and best efficiency and maximum percentage of biodiesel production occur in (5 h) of reaction time. The thermophysical properties of biodiesel produced from these oils were compared with standard biodiesel and all its properties were within the allowable range of standard ASTM D-6751.

Keywords: ASTM 6751-standard, Biodiesel, Transesterification, Calcium Oxide catalyst, Crop Mustard

1. INTRODUCTION

Nowadays, due to the non-renewability of fossil fuels and their negative environmental impacts, the necessity of finding alternative energy sources is well-established. with this respect, renewable energy sources have been considered as one of the most promising options for the last few decades, as they are cleaner and more environment-friendly and can contribute to the sustainable development

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of societies [1,2]. The maximum economic production of oil resources will be between 2015 and 2030. Consequently, the economic production of petroleum products will decrease in the coming years. In addition to the problem of depleting oil resources, there are many environmental problems in the use of diesel fuel, including the production of unburned hydrocarbons, carbon monoxide, sulfur pollutants and the production of carbon dioxide [3,4]. As these problems intensified, researchers turned their attention to alternative fuels. One of these alternative fuels is biodiesel, which has been introduced as a suitable fuel to replace conventional diesel fuel for use in heating systems and especially for use in diesel engines [5]. Biodiesel, which today Introduced as one of the suitable alternatives to diesel fuel, methyl ester or ethyl ester is vegetable and animal oils and this renewable fuel, of natural origin, contains oxygen, has a high cetane number, boiling point and also viscosity. Suitable for use in diesel engines [6]. Due to the high viscosity of the oil and the need to change the engine fuel system, various methods are used to reduce the viscosity of the oil, including dilution methods by hydrocarbons (combining), emulsions. Pyrolysis (thermal decomposition or breaking of large molecules by heat) and transesterification. It should be noted that the most important of these is the transesterification method [7-11]. In this method, an alcohol reacts with oil in the presence of a catalyst to produce glycerin and an alkyl ester, which is used as a biodiesel and as a fuel in a compression ignition engine [12-14]. In order to produce biodiesel fuel from mixture of crop mustard and edible waste oil, ester exchange method with heterogeneous calcium oxide catalyst prepared from fish bones was used as a catalyst by transesterification reaction. Determining some of the physical and chemical properties of the produced fuel and selecting the most optimal fuel composition with the best properties. Predicting a suitable model for biodiesel efficiency and determining the correlation coefficient. Therefore, in this study, models to predict biodiesel efficiency based on independent parameters (reaction temperature, reaction time, molar ratio of alcohol to oil and oil composition) are presented by Taguchi statistical program and compare.

2. EXPERIMENTAL

2.1. Reagents and materials

Crop Mustard and Edible Waste Oil, Calcium Oxide catalyst prepared from Fish bones were stored at room temperature. Also in this work Methanol, Hydrochloric acid were bought from Merck Company (Merck, Darmstadt, Germany). DW H_2O (double distilled water) was used in preparation of the solutions. The ensuing shows the concentrations of the stock solutions.

2.2. Instrumentation

Gas Chromatography (Varean model cp-3, Germany). Fourier transform infrared (FT-IR) spectra were recorded on a Perkin Elmer (FT-IR spectrum BX, Germany). X-ray diffractometer with CuKα radiation at beam acceleration conditions of 40 kV/35 mA. Scanning electron microscopy (SEM) were taken on a (KYKY-EM 3200, Hitachi Firm, China) under an acceleration voltage of 26kV) used to study the morphology of samples. Transmission electron microscopy (TEM) were taken on a (TEM, JEOL, Hitachi Company, China). For the measurement of pH, the pH/Ion meter (model-728, Metrohm Firm, Switzerland, Swiss) was employed.

2.3. Conversion of oil to biodiesel (production of biodiesel)

According to the design of experiments in Taguchi statistical program, the amounts of oil, catalyst and methanol are weighed and then the oil is poured into a balloon and the catalyst is dissolved in methanol and then added to the oil. At the same time, put a magnet in the balloon and put it on a heat styler (temperature and light mixer), then connect the refrigerant on the balloon and pour the water into the refrigerant. According to the design of experiments for the sample, we control the temperature and time. At the end of the test (production) time, turn off the mixer and temperature and pour the material into the balloon into a separating funnel, allowing the phase to separate completely, and finally the biodiesel produced from the glycerin and soap materials. The catalyst is separated and transferred to other containers for further testing. For example, experiment number one designed by Taguchi program. Weigh 100 g of oil and in the ratio of 1.6 oil to methanol weigh 23.74 g of methanol and weigh 0.3 g of calcium oxide catalyst separately according to the weight of 0.3% catalyst and then pour the oil into the balloon. Heat it until the solid oil is completely liquid. Meanwhile, add the catalyst to the methanol and stir thoroughly to mix. After the oil liquefies, add a mixture of methanol and catalyst to the oil and place a magnet inside it. Then install the refrigerant on the mouth of the balloon and pour the water into the refrigerant so that when the temperature is applied, the evaporated methanol is cooled again by the refrigerant and returns to our mixture. After preparing the biodiesel production system, the temperature of the mixture is raised to 55°C and at this temperature the mixture is stirred for 4 hours to complete the number one operating conditions. After the desired time, the temperature and stirrer are turned off and the material is transferred to the decanter funnel.

2.4. Production of biodiesel

In this study, after preparing the biodiesel for washing, first the glycerol is separated from the methyl ester (biodiesel) phase and then the methyl ester phase is washed several times with double distilled water at C700 to remove the catalyst and methanol in the methyl ester phase. After filtration, the methyl ester phase was poured into plastic bottles made of polyethylene and stored at ambient temperature shown in (Fig. 1), a reactor for biodiesel of crop mustard and edible waste oil in temperature and pressure was required to biodiesel from crop mustard and edible waste oil by the transesterification reaction [15].

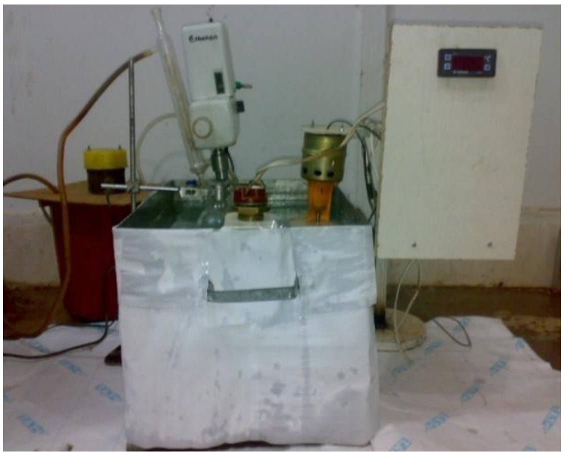

Fig. 1. Production machine biodiesel of crop mustard and edible waste oil.

3. RESULTS AND DISCUSSION

3.2. Sample characterization of adsorbent

As demonstrated in (Fig. 2a), the FTIR spectrum of calcium oxide catalyst prepared from fish bones catalyst presented clear broad. The peaks between cm-1 900-600 are related to the bending vibrations of O-Ca-O, which occurred in 875 cm^{-1}, and the peaks of 900-1500 cm^{-1} are related to the quadrilateral tensile vibrations of Ca-O. Which has appeared in 1415 cm^{-1} and also by examining the peaks of 1500-3500 cm^{-1}, the peaks in this range are related to Ca-O tensile vibrations.

Which occurred at 3427 cm^{-1} [10, 16]. X-ray diffraction (XRD) analysis was performed and the results are presented in Fig. 3b. Considering the fact that cellulose is the main component of calcium oxide catalyst prepared from fish bones catalyst and according to the XRD result in (Fig. 2b), the peak at 2θ around 22º is an evidence of cellulose. As it can be observed in the XRD results of the calcium oxide catalyst prepared from fish bones catalyst (Fig. 3b),the diffraction peaks at 2θ= 38.21°, 44.18°, 64.52°, and 78.12° were assigned to the calcium oxide catalyst prepared from fish bones catalyst [16, 17]. Obviously the perfect crystalline nature of the material was proven after functionalizing with calcium oxide catalyst prepared from fish bones catalyst however the great intensity of signal at 38.21(311) confirmed that there has been a slight amount of material in amorphous state. The perfect synthesis of calcium oxide catalyst prepared from fish bones catalyst is obvious through looking at XRD pattern. The morphological properties of the investigated samples by SEM are exhibited in (Fig.2c). As demonstrated in (Fig.2d), the evenness, homogeneity, orderliness and approximate uniformity of calcium oxide catalyst prepared from fish bones catalyst (even in size distribution) was observed. After surface modification, calcium oxide catalyst prepared from fish bones catalysts came to be uneven, bigger and agglomerate**.** It has been seen that the particles were mostly spherical with a various size distribution. From the particle size distribution. The particle size was also calculated using the Deba Schaer equation of 14-25 nm very close to those determined by XRD analysis [18, 19].

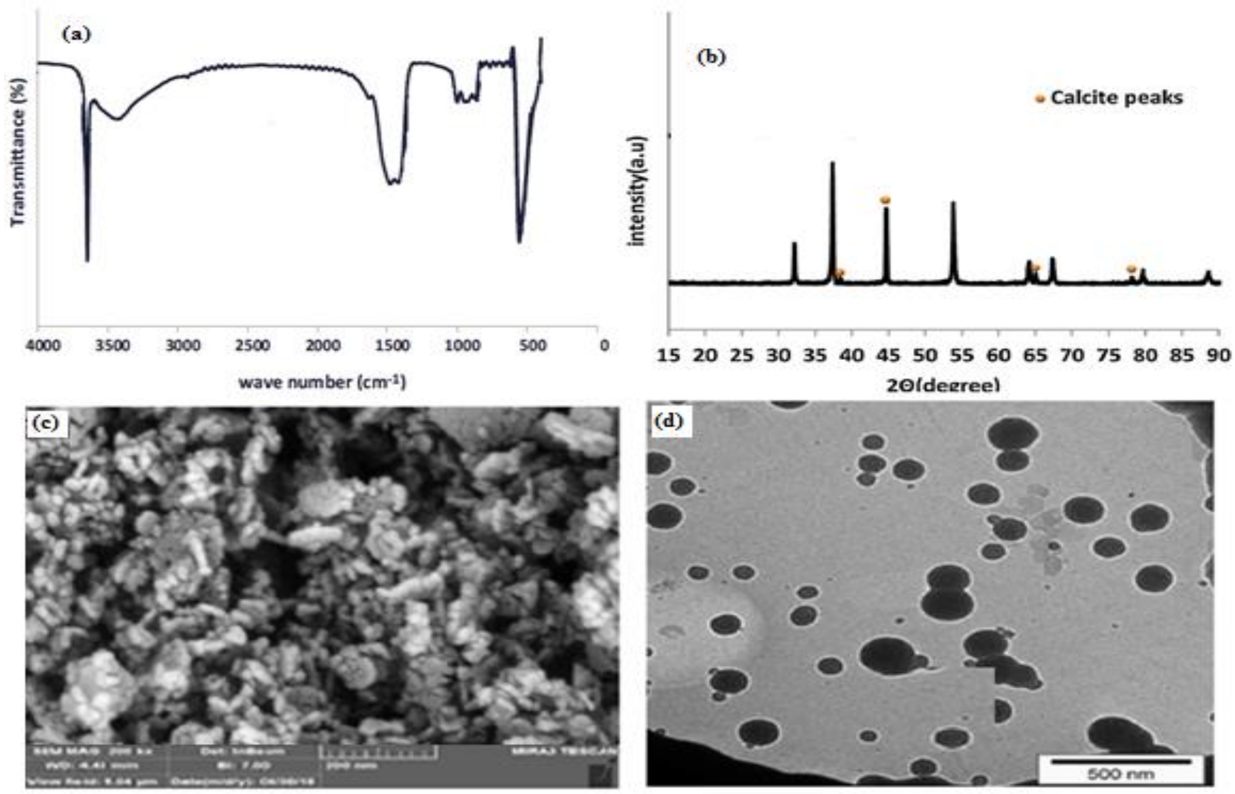

Fig. 2. (a) FTIR spectra, (b) XRD, (c) SEM and (d) TEM of calcium oxide catalyst prepared from fish bones catalyst.

3.2. Determination of heterogeneous calcium oxide catalyst prepared from fish bones composition of mixture of crop mustard and edible waste oil and its analysis

Crop mustard and edible waste oil compositions were determined using gas chromatography. In this study, gas chromatography device model Variancp-3800 which was equipped with FID detector and capillary column with a length of 30 meters with the following program conditions was used to study fatty acids. Helium gas was used as the carrier gas. Nitrogen gas flow was adjusted to compensate for hydrogen and air at 30 and 300 ml / min, respectively. The required standards and the necessary chemicals were purchased from the German company Merck. Initially, the temperature of the column was increased from 150°C to 220°C at a speed of 10°C for 60 s and remained at this temperature for 2 min. The temperature remained. After that, it reached 255 degrees with the same speed and remained for 1 minute, and finally it was brought to 268 degrees with a speed of 10 degrees per minute and kept at this temperature for 30 min. After finishing the work, the fatty acid compositions of the oil were obtained. The fatty acid compositions of crop mustard and edible waste oil were determined using a gas chromatography (GC). Table 1, reports the fatty acid composition of crop mustard and edible waste oil [20].

Table 1. The fatty acid compounds of Crop mustard and edible waste oil

Compounds Fatty acid (%)		**Crop mustard and edible waste oil**
35/71	C 16:0	Palmitic acid
3/3	C 18:0	Stearic acid
41/24	C 18:1	Oleic acid
12/41	C 18:2	Linoleic acid
----	C 18:3	Linolenic acid
0/8	C 14:0	Myristic acid

3.3. Characteristics of produced biodiesel

Some important properties of biodiesel produced in this research such as density, flash point, cloud point, drop point and cetane number were obtained and examined with the international standard ASTM D-6751, which is shown in the table below. Table 2, has been reported [15].

Table 2. Properties of biodiesel produced in accordance with US ASTM standards

Unit	**Biodiesel**	**Limit**	**Standard test method**	**Property**
°C	152	93-160	ASTM D-93	Flash Point
mm^2/s, 40°C	1.89	1.9-6.0	ASTM D-445	Density
°C	7	-	ASTM D-2500	Cloud Point
°C	12	-	ASTM D-97	Drop Point
-	49	47	ASTM D-6890 ASTM D-613	Cetane Number

3.4. The effect optimization of biodiesel

In this study, the effect of effective parameters on biodiesel production such as reaction temperature, reaction time, the amount of catalyst used, the ratio of methanol to oil was investigated and the best result was reported as the optimal condition. The first reaction at 55°C, the reaction time of 65 min and the amount of 1% by weight of potassium hydroxide and sodium hydroxide catalysts (based on oil weight) with a stirrer speed of 500 rpm and different ratios of Methanol to oil (1:3, 1:6, 1:9 and 1:12). 55°C, 1% by weight catalyst and stirring speed 500 rpm at different times (10-150 min). After determining the ratio of methanol to oil and reaction time, the effect of temperature parameters (55°C, 65°C, 75°C), and the amount of catalyst was also determined to be (0.25-1.25) [17,18,21].

3.5. The effect of operational parameters on production rate in Taguchi test design method.

In this section, the effect of the contribution of important and effective factors on the molar ratio of oil to methanol, catalyst percentage, reaction temperature and reaction time on the amount of biodiesel production using Taguchi experiment design method is investigated. As mentioned, in the experimental design method, the purpose of this design must first be specified. In the present study, the purpose of applying Taguchi method is to investigate the effect of operational parameters on the percentage of biodiesel production from mustard oil. To design the experiment, first a series of biodiesel production tests were obtained experimentally, then the number of parameters and levels were defined in the Taguchi program, then the experiments were determined by the program. As can be seen in (Table 3), for each of the effective parameters, three values are specified, for the molar ratio of oil to methanol, the percentage of catalyst, temperature and reaction time, respectively (1:6, 1:9 and 1:12), (0.1, 0.2 and 0.3%), (55°C, 65°C and 75°C) and time reaction (4, 5 and 6 hours) were selected. Based on the molar ratio of oil to methanol, the weight percentage of the catalyst used is the weight of each component, which is reported in (Table 3). Experimental factors, levels and matrix of Taguchi program on 27 L array [16, 22]. According to Table 4, which was planned by Taguchi program, the results of biodiesel production were included in the program.

Table 3. Weight of consumables by ratio and percentage

Weight catalyst (g)	Catalyst (%)	Amount Methanol (g)	Amount Oil (g)	Molar ratio of Oil to Methanol
0.1	0.1	23.74	100	1.6
0.2	0.2	35.49	100	1.6
0.3	0.3	44.92	100	1.12

Table 4. Experimental factors, levels and matrix of Taguchi program

Recovery (%)	Time (h)	Temperature (°C)	Catalyst (%)	Molar ratio of Oil to Methanol	Run
73.79	4	55	0.1	1.6	1
75.78	4	55	0.1	1.6	2
83.77	4	55	0.1	1.6	3
75.29	5	65	0.2	1.6	4
82.23	5	65	0.2	1.6	5
81.22	5	65	0.2	1.6	6
61.56	6	75	0.3	1.6	7
61.59	6	75	0.3	1.6	8
61.53	6	75	0.3	1.6	9
62.58	6	55	0.2	1.9	10
60.57	6	55	0.2	1.9	11
61.53	6	55	0.2	1.9	12
77.71	4	65	0.3	1.9	13
75.72	4	65	0.3	1.9	14
76.71	4	65	0.3	1.9	15
78.11	5	75	0.1	1.9	16
78.11	5	75	0.1	1.9	17
77.22	5	75	0.1	1.9	18
95.67	5	55	0.3	1.12	19
94.63	5	55	0.3	1.12	20
94.65	5	55	0.3	1.12	21
90.16	6	65	0.1	1.12	22
89.05	6	65	0.1	1.12	23
90.08	6	65	0.1	1.12	24
69.04	4	75	0.2	1.12	25
65.74	4	75	0.2	1.12	26
65.89	4	75	0.2	1.12	27

3.6. Investigation of parameters defined in biodiesel production operations

Investigation of the effect of molar ratio of oil to methanol on biodiesel production. The effect of molar ratio of oil to methanol on the percentage of biodiesel production was investigated by Taguchi program. And the resulting curve can be seen in (Fig. 3). Examining the obtained diagram (Table 5), the effect of the molar ratio of oil to methanol on the amount of biodiesel production can be concluded that by changing the oil to methanol ratio from (1:6 to 1:9), no significant effect was observed in production efficiency, but with Changing the molar ratio of oil to methanol from (1:9 to 1:12), we encountered a significant increase in biodiesel production efficiency. In addition, from this diagram, it can be seen that the effect of this parameter is strongly effective in the production of biodiesel with a ratio of (1:12), that this molar ratio of oil to methanol (1:12) has the best efficiency. Finally, using the statistical table obtained from the program shown in (Table 5), it can be concluded that the data obtained are significant for the concentration, because the value of Pi is less than 0.05 (P Value = 0.042) [23, 24].

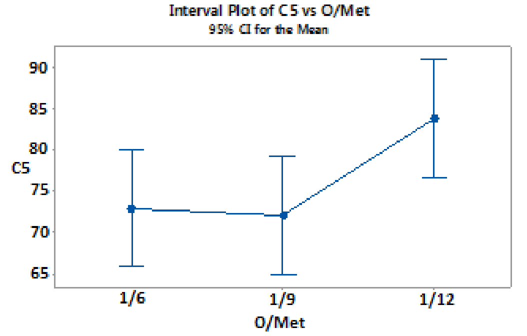

Fig. 3: Investigation of the effect of molar ratio of oil to methanol for biodiesel production.

Table 5. Investigation of the effect of molar ratio of oil to methanol on biodiesel production

P-Value	F-Value	Mean square	Factor
0.042	3.66	770.4	Molar ratio of Oil to Methanol

3.7. Investigation of the effect of catalyst weight percentage on biodiesel production.

By examining the percentage of biodiesel production against the weight percentage of catalyst by Taguchi statistical program, a curve shown in (Fig. 4), was obtained. As can be seen, the percentage of catalyst has a different effect on biodiesel production. From the curve it can be concluded that the value of 0.3% of catalyst can be considered as the maximum value in biodiesel production [15, 25]. Also, from the statistical data reported in (Table 6), it can be concluded that the results of this parameter are significant in biodiesel production (P Value = 0.061).

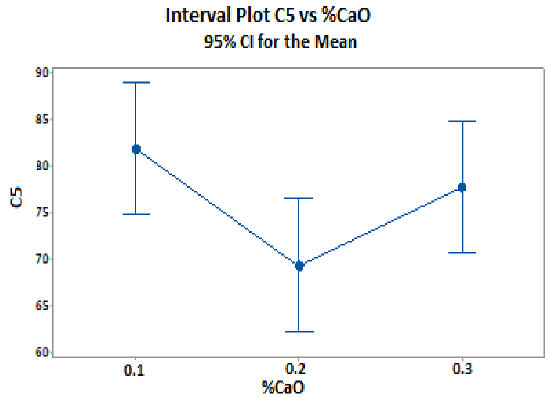

Fig. 4. The effect of catalyst content on biodiesel production.

Table 6. Investigation of the effect of catalyst weight percentage on biodiesel production

P-Value	F-Value	Mean square	Factor
0.061	3.41	766.2	Catalyst (%)

3.8. Investigation of reaction temperature on biodiesel production.

In biodiesel production method, one of the most important influential parameters is the reaction temperature. In this experiment, based on the data and diagram, a curve shown in (Fig. 5), obtained from Taguchi statistical program, it is observed that, by increasing the reaction temperature from 55 to 65°C, the amount of biodiesel production increases by about 8% and with Continued increase in temperature from 65 to 75 degrees, we face a sharp decrease in production (about 30%). As a result, the optimum reaction temperature for biodiesel production is 65°C. Also from the statistical data reported in (Table 7), it can be concluded that the results of this parameter are significant in biodiesel production (P Value = 0.037) [15, 26].

Table 7. Investigation of the effect of temperature on biodiesel production

P-Value	F-Value	Mean square	Factor
0.037	4.09	832.3	Temperature (°C)

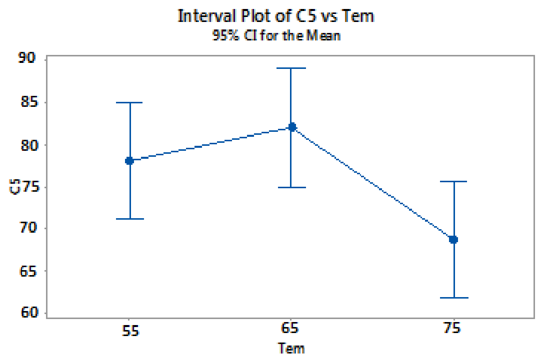

Fig. 5. Effects of temperature on biodiesel production process.

3.9. Investigation of the effect of reaction time on biodiesel production

By examining the graph obtained from the amount of biodiesel production to the reaction time parameter, the reaction time on biodiesel production has led to a significant production trend in production, a curve shown in (Fig. 6), the effect of this parameter on production can be expressed as follows. By increasing the reaction time from 4 hours to 5 hours, the percentage of biodiesel production has increased, but with the continuation of this trend, increasing the reaction time from 5 hours to 6 hours, the percentage of biodiesel production has decreased, resulting in the best efficiency [16, 27, 28]. Also from the statistical data reported in (Table 8). By examining the table obtained from Taguchi program, the P-value obtained is less than 0.05, which indicates the significance of the reaction time chart on biodiesel production and with the highest percentage of biodiesel production in 5 hours, the reaction is about (85%).

Table 8. Investigation of the effect of reaction time on biodiesel production

P-Value	F-Value	Sum of square	Factor
0.031	4.41	878.6	Time (h)

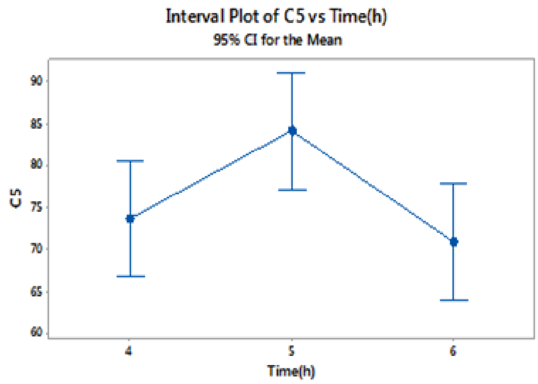

Fig. 6. The effect of reaction time on the percentage of biodiesel production.

4. CONCLUSION

In this study, in order to produce biodiesel fuel from mixture of crop mustard and edible waste oil, ester exchange method with heterogeneous calcium oxide catalyst prepared from fish bones was used as a catalyst. Then, the effect of the contribution of important and effective factors on the molar ratio of oil to methanol, catalyst percentage, reaction temperature and reaction time on the amount of biodiesel production was investigated using Taguchi experiment design method. Accordingly, the parameters considered to produce and evaluate the optimal production conditions, the molar ratio of oil to methanol (1:6, 1:9 and 1:12), weight percentage of inhomogeneous calcium oxide catalyst (0.1% wt, 0.2% wt and 0.3% wt) by weight, reaction temperature (55, 65 and 75°C) and reaction time (4-6-6 hours) were selected and programmed and produced in Taguchi statistical program of L27 array.

The results of this study for the production of biodiesel under optimal conditions with a molar ratio of oil to methanol (1:12), catalyst weight percentage: 0.3%, reaction temperature: 55°C and reaction time of 5 hours with the highest production efficiency of average three times production (94.95%) Percentage was obtained.

From the sample produced under optimal conditions and the highest efficiency produced, tests and experiments were performed, which can be generally obtained with results such as:

1) According to Taguchi statistical program reviews

By increasing the molar ratio from (1:6 to 1:12), there has been a significant increase in biodiesel

By increasing the amount of catalyst from (0.1%) by weight, we witnessed a decrease in biodiesel production to (0.2% wt), and then by increasing the percentage of catalyst from (0.2% wt to 0.3% wt), production was increased.

The increase in temperature of more than (55°C), was initially observed with a gentle slope to (65°C) and then with a sharp decrease in production from (65°C to 75°C), in biodiesel production was observed.

Reaction time had a surprising effect on biodiesel production and the effect of production time can be considered as one of the most tangible reaction conditions.

2) According to flash point studies. It can be concluded that the amount of methanol in the produced biodiesel is so small that the ignition temperature rises and all methanol is consumed in the production process and the flash point is within the ASTM standard.

3) According to cloud point and drop point studies. According to the results obtained from the point of cloud cover and fall of bio diesel produced, their values are relatively high and this indicates that this sample of bio diesel produced can not be used in cold flood areas.

4) According to density studies. The density of biodiesel produced complies with standard conditions.

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تعیین ویژگی‌های بیودیزل از مخلوط خردل و روغن‌های زاید خوراکی با استفاده از کاتالیست ناهمگن اکسید کلسیم تهیه‌شده از استخوان ماهی و پیش‌بینی کارایی بیودیزل با استفاده از مدل برنامه آماری تاگوچی

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چکیده

در این مطالعه به منظور تولید سوخت بیودیزل از مخلوط خردل گیاهی و روغن پسماند خوراکی، از روش تبادل استر با کاتالیست ناهمگن اکسید کلسیم تهیه شده از استخوان ماهی به عنوان کاتالیزور استفاده شد. سپس با استفاده از روش طراحی آزمایش تاگوچی، تأثیر عوامل مهم و مؤثر بر نسبت مولی روغن به متانول، درصد کاتالیست، دمای واکنش و زمان واکنش بر میزان تولید بیودیزل بررسی شد .در روش تولید بیودیزل واکنش بهینه مشاهده می شود که اثر این پارامتر در تولید بیودیزل با نسبت (۱:۱۲) به شدت موثر است که این نسبت مولی روغن به متانول (۱:۱۲) دارد. بهترین کارایی با توجه به نتایج، مقدار (۰/۳ درصد) کاتالیست اکسید کلسیم تهیه شده از استخوان ماهی را می توان به عنوان حداکثر مقدار در تولید بیودیزل در نظر گرفت. مشاهده شد که با افزایش دمای واکنش از (۵۵ درجه سانتی گراد به ٦٥ درجه سانتی‌گراد)، میزان تولید بیودیزل حدود ۸ درصد افزایش می یابد و با ادامه افزایش دما از (٦٥ درجه سانتی گراد به ۷۵ درجه سانتی گراد)، با کاهش شدید تولید (حدود ۳۰ درصد) مواجه هستیم. در نتیجه دمای تولید بیودیزل (٦٥ درجه سانتی گراد) است و بهترین بازده و حداکثر درصد تولید بیودیزل در (۵ ساعت) زمان واکنش رخ می دهد. خواص ترموفیزیکی بیودیزل تولید شده از این روغن ها با بیودیزل استاندارد مقایسه شد و تمامی خواص آن در محدوده مجاز استاندارد ASTM D-۶۷۵۱ قرار داشت.

کلید واژه‌ها: بیودیزل، ترانس استریفیکاسیون، کاتالیست اکسید کلسیم، خردل زراعی، استاندارد.ASTM 6751

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