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Process parameters optimization of carbon nano tube based catalytic transesterification of algal oil

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ABSTRACT

The requirement for different types of natural fuel and the peak in the price are big issues for mankind. Researchers are more concerned about using environmentally friendly fuel than limited energy sources now that public awareness has been raised. Different recyclable energy sources like nuclear, wind, solar, and biofuels are the best resources, whereas biofuel in different phases has been experimentally performed as one of the best representative candidates in terms of usage and the production process. The energy of the biomass is recyclable and also has properties similar to those fossil fuels. To develop the biodiesel with the help of the transesterification methodology, the catalyst used must be more effective to ensure faster catalytic activity. In various research journals, different varieties of homogenous type catalyst are being experimented with, but the reusability of the catalyst is impossible as compared with heterogeneous catalysts. The biodiesel yield is calculated using response surface methodology and Chlorella vulgaris algae oil as a feedstock and carbon nanotubes as a heterogeneous nano catalyst. The present work is performed under 15 experiments by considering 3 parameters: time of reaction, catalyst concentration, methanol to oil volume, and temperature, which is kept constant at 50 °C.

1. Introduction

Energy extraction from renewable sources like solar energy, wind energy, hydro energy, and biofuel-based energy has always center of attraction for energy researchers because of its endless benefits [1–4]. Biofuel has a wide area in the development of energy that can easily accomplish the requirements of daily life. With the use of organic materials, the bioenergy produced is biomass. The synthesis of biofuel from algae has been performed in a wide area that has applications in the automobile to reduce carbon pollutants in the atmosphere [5,6]. At the global level, almost 10 % of the energy is being used as thermal energy [7]. To produce such energy, different varieties of feedstock are obtained from plants, agricultural waste, animals, and the industrial area. The amount of energy produced always depends on the type of methodology followed in the synthesis of the biofuel [8]. The lignocellulose that comes from the second generation provides a better synthesis of biofuel. The *trans*-esterification of oil results in the smooth operation of diesel engines [9,10]. We can analyse the biodegradability of biodiesel using the design in free fatty acids. The fuels have a large variety of applications and make the environment free from harmful pollutants. They can degrade as compared to natural diesel fuel [11–15]. With the help of transesterification, using waste vegetable oil and mixing alcohol with catalyst results in alkyl easter, the separate layer of biodiesel rises to the top and glycerol settles at the bottom [16,17].

According to a study, the amount of biodiesel produced across the globe is estimated at 2,616 thousand barrels. The fastest rate of synthesis of biodiesel is nearly 87 % in the US and Brazil [18]. The production of biogas with biofuel is between 1890 and 702 thousand barrels a day in the United States, which is more than that of Brazil (approximately 693.2 barrels) a day, making it the second leading country in the synthesis of biofuel [19]. Algae can be easily grown, which requires water and an adequate amount of sunlight. The surrounding air temperature can be regulated with the application of solar air heaters [2,20–23]. The filtration and drying procedure is done to harvest the microalgae. With the help of chemical and mechanical methodologies, biodiesel is extracted

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from algae [24–26]. India stands in the fourth position in the synthesis of edible oil products across the world [27,28].

By-products vary along with the different generations of biofuel types, in the first generation, the ethanol is synthesized using corn. Waste products and biomass, as well as crops, are classified as second-generation biofuels [29]. The production of biodiesel is more concerned with the third generation type, in which natural algae and waste materials from sewage are used in the production of biodiesel [30]. The use of catalysts in biofuel production speeds up transesterification depending on the variety used as a catalyst [31]. Application of catalysts on the nanoscale is the new technology that increases the transesterification process' speed, provides recyclability in the catalyst, and avoids the overuse of a suitable catalyst. Several researchers have conducted experiments on biofuel using a homogenous type catalyst [32]. Because the catalyst used mostly sodium and potassium hydroxides as a homogeneous catalyst, sodium hydroxide is preferred more due to its ease of availability[33–37].

C. H. Su [38] produces biodiesel by using various categories of homogenous catalysts of the acid type. The peak yield of nearly 98.19 % resulted in an optimum for variable such as the reaction time of 10.57 min, the molar ratio of 7.92, and the temperature of 76.67°Celsius. Maleki et al. [39] accomplished the transesterification method by operating the waste cooking oil and mixing it with CH_3OH at a reaction temperature of 62 °C, time at 120 s, and a methanol to oil ratio of 9:4, which resulted in a maximum yield of 98.26 % of biodiesel. Shariati et al. [40] transfigure triglycerides by converting alcohol into biofuel by using the methodology of transesterification. The maximum yield of 98.18 % was achieved at optimum conditions for the temperature of 48 °C followed by time (120 min) and methanol/oil concentration at 6.825. Garg et al. [41] used an artificial neutral network and response surface method to produce 94.17 % of biodiesel at an optimum value while keeping the temperature constant (50 °C), time at 77 min, and catalyst concentration at 1.32 %.

The use of the various catalyst under the homogenous category provided fair results but in some experimental research, the homogenous catalyst resulted in issues regarding the purity of the biofuel as the concentration of the catalyst increases [42]. The homogenous catalyst increases the yield but it is toxic. The separation of the catalyst seems challenging and also very expensive [43]. To overcome the situation, the researchers started the use of heterogeneous catalysts in the synthesis of biodiesel [44].

Souza et al. [45] produce biofuel from calcium oxide and soya bean. 600 °C, 4 h, and 7:1 M ratio yielded 99 % biodiesel. Awad et al. [46] maximize biodiesel output from waste oil. Pyrolysis created a catalyst that maximises biodiesel output at 90.37 % and maintains methanol concentration at 20 for 6 h at 70°Celsius. Biodiesel yielded cleaner results than diesel fuel. Singh et al. [47] synthesis biodiesel using potassium hydroxide and alumina catalysts. The muffle furnace produced low-temperature alumina. Optimization yielded 98 % biodiesel at 9 methanol for 60 min at 70°Celsius. Sharma et al. [48] heterogeneous catalyst magnesium zirconate integrated biodiesel. Transesterification with Kusum oil shows the catalyst's advantages.

When compared to a homogeneous type catalyst, the heterogeneous catalyst provided exceptional yield at high temperatures. It resulted in a yield of 97.98 % at an optimum value of 18:1 for methanol to oil quantity for 150 min at 65°Celsius. With such a catalyst, the biofuel properties seem preferable for biofuel production. Chung et al. [49] synthesize biofuel with the help of jatropha curcas; which is used as feedstock, and a mixing catalyst, which comes under the heterogeneous category. The catalyst can be easily separated and also provides biodiesel properties similar to those of natural diesel fuel. A catalyst concentration of 2–3 % at 50°Celsius for 67.9 min resulted in a 97.17 % biofuel yield. Aiswarya et al. [50] created a biofuel by using castor oil as a feedstock, which was then mixed with zinc oxides doped with nickel. Heterogeneous nano-catalyst resulted in 97.17 % of biofuel yield at an optimum temperature of 55 °C for 60 min with a methanol concentration of 8 %. The nano-catalyst proved reliable at various ranges of temperature during the experimentation, and the catalyst can be reused up to five times. The catalyst showed a decent outcome with economical castor oil. The work towards the nano-catalyst is very minimal, and still more research analysis is needed to provide efficient and environment-friendly biofuel for various applications all over the globe and to cut down the peak rate of natural fuel [51].

According to the findings of the following study, synthesis of biodiesel using a heterogeneous catalyst yields better results. The biodiesel can be easily developed and also separated, which is quite expensive in the case of a homogenous catalyst [52]. The homogeneous catalyst is being used in various journals, but the use of nano catalysts has limited biodiesel yield research. The researchers are tracking new methodologies to develop biofuel without exploitation of natural resources, which are limited. Due to the large surface area, the nanoparticles can increase the catalytic activity faster and provide more enhancement in the transesterification process [53]. The nano-catalyst which comes the category of heterogeneous catalyst, can also be reused again, which can overcome the problem related to overuse of the homogeneous type catalyst while performing transesterification. The study's goal is to determine what percentage of biodiesel yield can be achieved by using nanocatalysts such as carbon nanotubes.

2. Materials used in transesterification

2.1. Catalyst and feedstock

For performing the transesterification process, Chlorella vulgaris oil is used as feedstock, which is bought 10 L from the IIT Roorkee. The single-wall carbon nanotube is used as the catalyst because it has a large surface area with a range of 400–900 m^2/g. The greater outside size of carbon nanoparticles leads to intensifying transesterification activity; almost 100 gm of carbon nanoparticles are used in the experimental work. The temperature in the electric heater is set at 50 °C for 15 experiments. The details of the properties of Carbon Nanotubes for single and multi CNTs are shown in Table 1.

3. Design setup for experiment

The design experiments are carried out in a 1 L glass-type reactor that is connected to a condenser component, a magnetic stirrer, and an electric heater. The temperature of the electric heater is fixed at 50 °C for all the experiments to be performed. The 2D design of the layout is presented in Fig. 1. The condenser is connected with the inlet and outlet water supplies to maintain the temperature for better transesterification of Chlorella vulgaris oil. The magnetic stirrer is placed inside the conical flask to ensure homogeneous mixing of the catalyst with the oil for better enhancement of the biodiesel yield. The experiments were per-

Table 1
Properties of Carbon Nanotubes [54].

Characteristics with unit	Single wall CNTs	Multi wall CNTs
Specific Gravity (g/cm^3)	0.8	1.8
Elastic Modulus (P)	1	0.3–1
Strength (GP)	50–500	10–60
Resistivity (Ω-m)	5–50	5–50
Thermal Conductivity (W/m K)	3000	3000
Thermal Stability (°C)	>700	>700
Specific Surface Area (m^2/g)	400–900	200–400

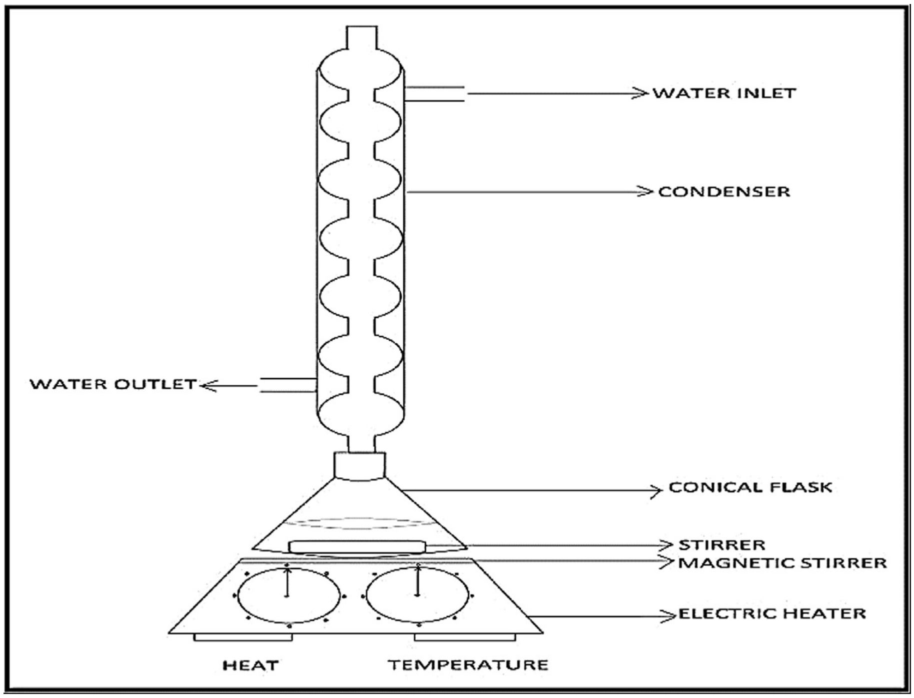

Fig. 1. Transesterification process setup diagram.

formed as provided by the design expert software with fixed ranges for all three variables except the temperature, which is kept fixed; the details are shown in Table 2**.**

4. Design of experiments

4.1. Use of response surface method

The Response Surface Methodology (RSM) is very beneficial for designing the experiments; we used Design of Expert (DoE) version 11.1.2.0. By setting the minimum and maximum ranges, 15 experiments were conducted. The process of transesterification is analysed on three independent variables, which are the concentration of catalyst, the temperature during the reaction, and the time for completion of the reaction process, according to the study of various research papers. The present work is performed under 15 experiments by considering 3 parameters: time of reaction, catalyst concentration, methanol to oil volume, and the temperature, which is kept constant at 50 °C. Various literature reviews showed different ranges for the three variables in the synthesis of the biofuel. With the use of the range, the significance of the design of the model will be analyzed. The experiments designed with the help of Box-Behenken are Table 3 and Table 4 for three variables.

5. Results and discussion

5.1. 5.1Transestrification process

The designed experiments were designed by using the design technique from the box- Behenken 15 experiments have been conducted, which are given in Table 3. The experiments performed will help to achieve the optimum ranges for all three variables (A: Time, B: volume of methanol/oil concentration, C: the amount of catalyst in grams), and the responses achieved are represented in Table 4. On the behalf of 15 experimental data, with the help of the RSM model is designed which is represented by equation (1):

$$BiodieselYield(\%) = -384.78375 + 2.61477 * A + 56.90458 * B + 44.54400 * C - 0.107236 * A * B + 0.121433 * A * C - 2.80533 * B * C - 0.006927 * A^2 - 2.27542 * B^2 - 5.50420 * C^2 \quad (1)$$

Equation (1) was applied to analyse the predicted values for the biodiesel yield, which resulted in Table 4. The model can be analyzed for the optimized ranges to achieve maximum yield. The val-

Table 2
High and low range of Variables.

Variables	unit	High range	Low Range
Time of reaction (A)	Minutes	180	60
Methanol/oil percentage (B)	% (v/v)	12	6
Catalyst concentration(C)	% (w/w)	5	0

Table 3
List of experiments for transesterification.

run	Time of reaction (min)	Methanol/oil ratio (%v/v)	Concentration of catalyst (%w/w)
1.	180	12	2.5
2.	120	6	0
3.	60	9	0
4.	120	9	2.5
5.	120	9	2.5
6.	180	6	2.5
7.	60	12	2.5
8.	120	9	2.5
9.	120	12	5
10.	60	6	2.5
11.	180	9	0
12.	120	12	0
13.	60	9	5
14.	180	9	5
15.	120	6	5

Table 4
Responses of variables.

S. No.	Time	Methanol	Catalyst	Experimental Value %	Predicted value	Error %
1.	180	12	2.5	29.43	32.4487	−10.2572
2.	120	6	0	6.75	11.54229	−70.9968
3.	60	9	0	18.87	17.09001	9.4329
4.	120	9	2.5	89.57	91.525895	−2.1835
5.	120	9	2.5	90.34	91.525895	−1.3127
6.	180	6	2.5	97.14	94.66118	2.5518
7.	60	12	2.5	33.67	36.16361	−7.4060
8.	120	9	2.5	94.65	91.525895	3.3006
9.	120	12	5	24.45	19.66949	19.5521
10.	60	6	2.5	24.17	21.1664	12.4269
11.	180	9	0	17.83	15.54993	12.7878
12.	120	12	0	30.72	30.01449	2.2965
13.	60	9	5	10.1	12.39506	−22.7233
14.	180	9	5	81.92	83.71478	−2.1908
15.	120	6	5	84.64	85.35719	−0.8473

ues for the error percentage are evaluated from the values of the predicted and the experimental, which are developed with the use of the response surface method.

5.2. Study of ANOVA

ANOVA helps in obtaining the best results in the experimental area for biofuel synthesis, along with the design of the model. The results obtained with the use of ANOVA for the response surface methodology are represented in Table 5. For statistical hypothesis testing, the value of P helps in understanding the significance of the design model. If the P-value of P is<0.05, the particular value is considered significant. In Table 5, the model has P-value < 0.05 which shows it is significant. In the following case, all the sources from A to C^2 are represented as significant for the model. The value of F for the model is 88.29, which signifies that the model is remarkable. The increased value in F can be explained by noise. Some values seem to be>0.1000; such terms have been marked as significant. Due to the cause of the error, it leads to an insignificant lack of fit, which is 4.19. Due to this high value, the chances are nearly 19.87 % that it has happened because of noise. In various journals, it is studied that the least value for R^2 is 0.80 which signifies that the fitness of the model is good [3155]. R^2 value for the response surface model was 0.9937.

5.3. Outcome from process variables in yield of biodiesel

The role of process variables in the yield of biodiesel is analyzed by the three variables time (A), the volume of methanol/ oil (B), and the amount of catalyst (C) used during the activity of the transesterification process. The perturbation graph provides a clear analysis of the change in the biodiesel yield, as shown in Fig. 2. It is clear that in curve C after reaching the maximum desirability the yield starts decreasing on further increasing the catalyst concentration. The carbon nanoparticles have a greater surface area per volume due to which the catalytic activity is faster, but on further increasing the concentration, the yield decreases. As time increased, Curve A showed a positive slope, but as it reached the maximum point, it showed a declination in the slope. This declination is due to the increase in the amount of catalyst in the process of transesterification. Curve B denotes that on mixing the methanol the response yield showed declination in slope on increasing the methanol to oil value.

5.4. Effect reaction parameters

The role of reaction parameters in the maximum biodiesel yield can be analyzed with the help of 3D curves. With the help of elliptical contours, curves can be created. The use of the Design-Expert application software helps in obtaining output data through contour designs. The 3D curves are designed by keeping two variables constant. By using such variables with the help of a design expert, we obtain response type curves that are from AB to BC. After the analysis is made, the study for maximum yield in biodiesel is studied between two independent parameters rather than all three variables. These curves are shown in Fig. 3, Fig. 4 and Fig. 5 respectively.

The 3D design in Fig. 3 explains the yield between time and methanol in the form of curve AB. Biodiesel synthesis increases with increasing time value up to maximum yield. When the methanol content was increased, the yield of biodiesel decreased. In the AB curve, time played a valuable role in increasing the yield

Table 5
Analysis of variance of biodiesel.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	17350.07	9	1927.79	88.29	< 0.0001	(significant)
A-Time	2432.88	1	2432.88	111.42	0.0001	
B-Methanol	1114.63	1	1114.63	51.05	0.0008	
C-Catalyst	2014.22	1	2014.22	92.25	0.0002	
AB	1490.35	1	1490.35	68.26	0.0004	
AC	1327.14	1	1327.14	60.78	0.0006	
BC	1770.73	1	1770.73	81.10	0.0003	
A^2	2296.40	1	2296.40	105.17	0.0002	
B^2	1548.48	1	1548.48	70.92	0.0004	
C^2	4369.65	1	4369.65	200.12	< 0.0001	
Residual	109.17	5	21.83			
Lack of Fit	94.18	3	31.39	4.19	0.1987	(not significant)
Pure Error	14.99	2	7.50			

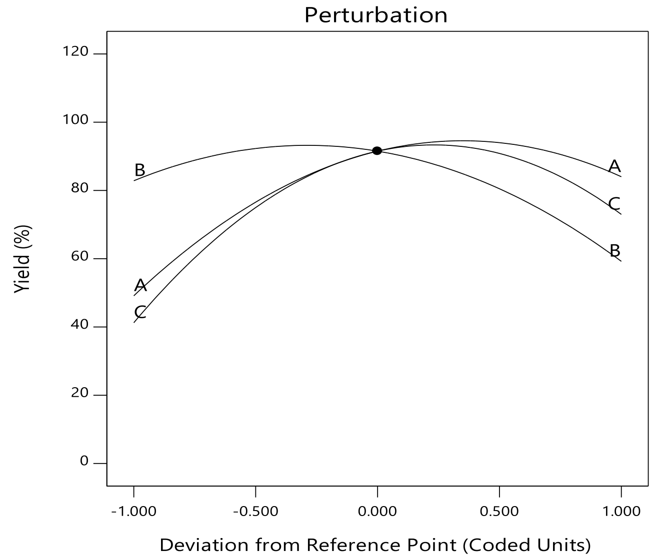

Fig. 2. Role of three variables regarding yield percentage.

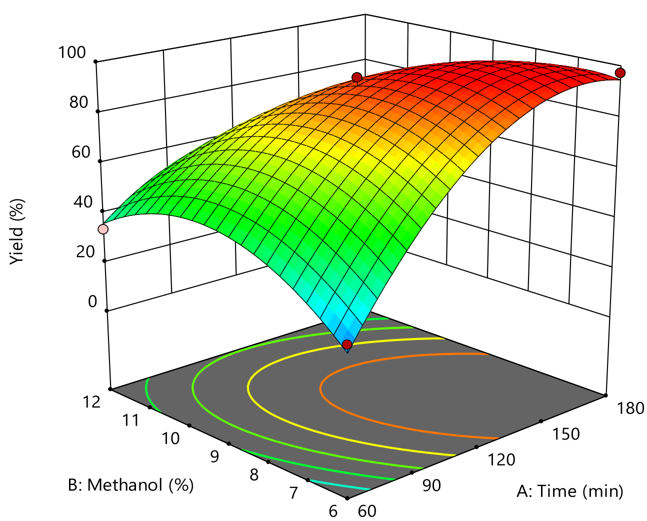

Fig. 3. Analysis of yield between B and A.

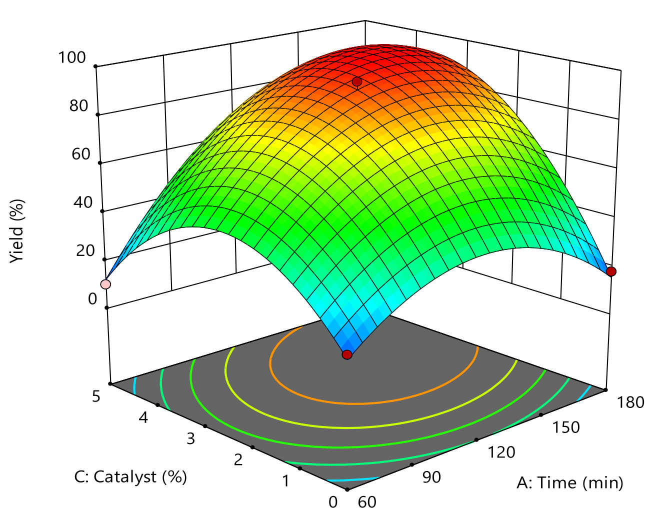

Fig. 4. Analysis of yield between C and A.

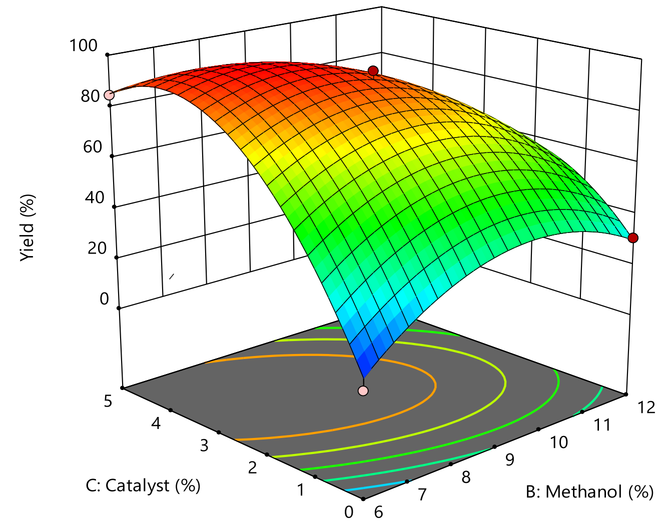

Fig. 5. Analysis of Yield between C and B.

Table 6
The optimum range for maximum biodiesel output.

Variables	Target	Optimum range
Time (minutes)	In range	144.443
Methanol/ oil ratio %(v/v)	In range	8.620
Catalyst concentration %(w/w)	In range	2.766
Biodiesel yield %	Maximize	99.023

percentage. Fig. 4 depicts an analysis of the AC curve toward the yield percentage. The concentration of catalyst, when increased with an increase in time, the yield to a maximum range; on further increasing the time, the yield was reduced to 12 % along with the increase in catalyst concentration. The biodiesel yield has been significantly increased due to the large surface area. In Fig. 5, yield is the function of two variables, the catalyst and methanol. The increase in methanol raised the biodiesel yield to the maximum, but increasing the concentration of catalyst to the maximum goal gave rise to a decline in the yield percentage.

5.5. Selection of optimum parameter for maximum yield

The optimization of all three independent variables used in the methodology of the transesterification process is shown in Table 6, where the output value of the yield percentage is at its maximum.

6. Conclusion

In the following research work, the transesterification process is carried out between Chlorella vulgaris oil as feedstock and carbon nanotubes as a catalyst. With the use of three independent variables: the concentration of catalyst in grams, the volume of methanol, and the time for the reaction. The experiment design has been made using Box-Behnken in the design expert software, keeping the temperature at 50℃. The minimum and maximum ranges for the time 60–180 min, Methanol/oil 6–12 % (v/v) and catalyst concentration 0–5 % (w/w). The optimized range for maximum yield of 99.023 % at 144.443 min, 8.620 % of methanol, and 2.766 % catalyst concentration, is obtained. The catalyst accelerated the transesterification process, increase the biodiesel yield to the optimum point, but increasing time along with the catalyst led to the decrease in yield. The over-usage of homogenous catalysts makes the yield higher, which is not seen in the case of heterogeneous catalysts like carbon nanoparticles, due to their large surface area, more enhancement has been analyzed in the process of transesterification. The recycling and reuse of catalysts is cheaper and more convenient than that of homogenous catalysts. It is being established that biofuel can be applied to various industrial areas to upgrade effectiveness and to avoid overuse of homogenous catalysts, which will save time and money.

CRediT authorship contribution statement

Anna Raj Singh: Conceptualization, Software. **Sudhir Kumar Singh:** Software, Supervision. **Varun Pratap Singh:** Supervision, Writing – review & editing.

Data availability

Data will be made available on request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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