

# Optimisation of Biodiesel Synthesis Process from Various Edible Oils using KOH and NaOH as Homogeneous Catalysts

Kunal P. Argade<sup>1</sup>, Dhaval G. Shrimali<sup>1</sup>, Jharna Gupta<sup>2,\*</sup>, Nitin Bhate<sup>3</sup>, Nikul K. Patel<sup>4</sup>

## Abstract

*In the present experimental study, an optimization of biodiesel production from different kinds of edible oils (Palm oil, Cotton-seed oil and Sunflower oil) using KOH & NaOH as homogenous catalyst has been investigated by one step transesterification method. The research study was conducted into two parts. In the first experimental study, different types of edible oils (Sunflower oil, Cottonseed oil and Palm oil) have been optimised using KOH and NaOH catalyst for biodiesel synthesis by one step transesterification method. The maximal biodiesel yield (98%) was found by sunflower oil using different reaction parameters such as process temperature of 60°C, Alcohol(methanol) to oil molar ratio [6:1], catalyst (KOH) concentration of 1 gm./100 cc feed and process time of 55 min. In the second experimental study, transesterification reaction parameters were again optimised using optimised feedstock i.e. sunflower oil and optimised catalyst i.e. KOH (homogeneous catalyst) and found the maximal desired biodiesel yield (99%) using a 0.8 gm KOH catalyst, Alcohol (methanol) to oil ratio was 6:1 and 55 minute process time at 55°C. Produced biodiesel properties (Kinematic viscosity, flash point, fire point, cloud point and pour point) were also estimated and confirmed by the biodiesel standard ASTM D6751 and found within the prescribed limit. Gas Chromatography analysis was also performed for checking Fatty Acid Methyl Ester (FAME) purity. These results indicate that KOH as homogeneous catalyst and sunflower oil as edible oil is found to be the best combination of homogeneous catalyst and edible oil in terms of high biodiesel yield with good properties.*

**Keywords:** Biodiesel, Sunflower oil, Homogenous catalyst, Transesterification reaction, Edible oil

### \*Author for Correspondence

Jharna Gupta  
E-mail: [jharnagupta-chemengg@msubaroda.ac.in](mailto:jharnagupta-chemengg@msubaroda.ac.in)

<sup>1</sup>Student, Department of Chemical Engineering, Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, India

<sup>2</sup>Assistant Professor, Department of Chemical Engineering, Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, India

<sup>3</sup>Associate Professor, Department of Chemical Engineering, Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, India

<sup>4</sup>Assistant Professor, Department of Mechanical Engineering, Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, India

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## INTRODUCTION

Energy has a major impact in the welfare progress and survival of any particular nation. [1]. So, the shortage of fuels and energy can be considered as significant problem in commercial and domestic setup. So, this critical scenario can be used to take forward chance for research of making of the sustainable fuels, such as biofuels [2].

Renewable fuels are mainly used to describe the ones which we acquired from the resources like solar waves, winds, and sea or river tides. So, these sources are essentially inexhaustible and naturally available [3]. Biofuels are fuels derived from food crops, energy crops, edible and nonedible oils and waste products [4]. Biofuels offer a more sustainable alternative of fossil fuels as they maintain a cleaner Ienvironment and healthier population [5].

Biodiesel is also named as alkyl (mono) ester of fatty acids produce from vegetable oils and animal fats. The vegetable oils and animal fats convert in to the fatty acid methyl ester or fatty acid ethyl ester (biodiesel) using alcohol and catalyst [6]. It can be used as an alternative to conventional diesel fuel in vehicles, offering environmental benefits as it produces negligible emissions when burned [7]. It is similar to petroleum diesel fuel in structure (straight chain), number of C atoms (10-21) [8], and also have more cetane number by which ignition quality improves [9]. Biodiesel generates minimal amount of harmful gases as compared to diesel [10] and it is also green and clean burning renewable fuel that reduces pollution and greenhouse gas emissions [11].

Biodiesel is Derived by a biochemical process called "Transesterification". In which triglycerides react with alcohol in presence of strong acid/base catalyst. "Triglyceride" is a principal compound which is present in animals and plants [12]. The consumable plants can be categorized into two groups first is oil (Edible) namely coconut oil, soybean, peanut, palm, and rapeseed, and second is oil (non-edible) namely algae, Karanja, sea-mangoes, jatropha, and halophytes. These are used as primary raw material in production of biodiesel [13]. The high FFA content, not available in large quantity, require two step transesterification process and low biodiesel yield are the major concern for using nonedible oil as biodiesel feedstock. Animal fats also contains mainly high saturated fatty acid which is also challenging [14]. Therefore, edible oil based biodiesel production is found to be the best solution to overcome the drawback raised from nonedible oils [15-16]. Apart from this, the fatty acid composition affects the properties of the biodiesel. Non-edible oils may contain higher levels of saturated or poly-saturated fatty acids (FFA %), which can result in biodiesel with less favourable cold flow and oxidative stability properties. Edible oil crops generally have higher yields as compared to non-edible oil crops [17]. This makes edible oil crops more attractive from an economic perspective in biodiesel production.

In synthesis and production of biodiesel generally two types of catalyst are used that is homogeneous and heterogeneous catalyst, which converts triglyceride (fats & oils) into biodiesel (fatty acid methyl or ethyl esters) through transesterification reaction [18]. Many Heterogeneous catalysts like CaO, ZnO, Zeolites, MgO, solid acid or basic catalyst, earth metal oxides etc. are generally used in transesterification reactions for biodiesel synthesis but separation process between heterogeneous catalyst and biodiesel product, long reaction time, highly used catalyst amount and high methanol to oil molar ratio are the major limitations of using it. Homogeneous catalyst i.e. NaOH and KOH are generally used in transesterification reactions. The advantages of using homogeneous catalyst are its soluble nature in alcohol, faster reaction rates, easier to handle and give higher biodiesel yield [19-21]. Therefore the first objective of this study is to make the best combination of the oil and catalyst as biodiesel feedstock in terms of higher biodiesel yield and the second objective is to optimise the reaction conditions by using above combination of feedstock for getting highest biodiesel yield with good biodiesel properties.

## METHODS AND MATERIALS

### Chemicals and Materials

Three types of edible oils (Sunflower, Cotton-seed, Palm) were obtained from the local manufacturer. Catalyst (KOH and NaOH), and Methanol was purchased from Dutta Laboratory Furnishers, Vadodara, India.

### Characterization of Oil

The physical properties like acid value, FFA, density, kinematic viscosity, etc., are calculated by method as stated in the reference literature [22] and illustrated in Table 1.

### Transesterification Reaction Method

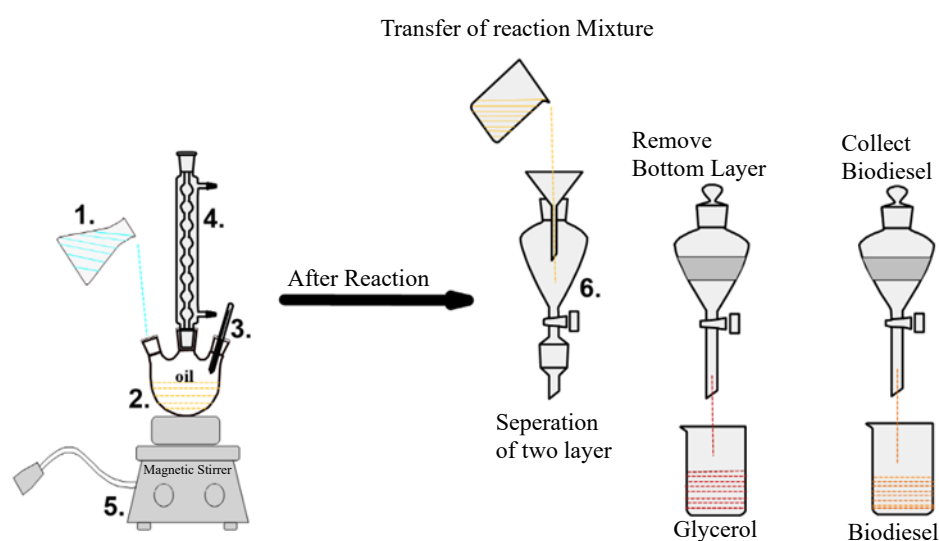
In this present experimental study, oil (Edible) is used for producing biodiesel. In the initial step, an alcohol solution is made, which promote the reaction between the oil feedstock, catalyst, and increase reaction efficiency. In this study, methanol is used in a specific molar ratio. This step is done in a separate vessel like a conical flask. The homogeneous catalyst (KOH or NaOH) is added to the methanol in the conical flask and stir well to dissolve the catalyst in the methanol.

**Table 1.** Different physical properties of Sunflower, Cotton seed, Palm Oil.

Physical Properties of oil	Units	Values		
		Sunflower Oil	Cotton seed Oil	Palm Oil
FFA	-	0.32	0.425	1.681
Acid Value	KOH (mg) / oil (gm)	0.642	0.85	3.36
Density	gm/ml	0.918	0.933	0.912
Kinematic Viscosity at 40 <sup>o</sup> C	Cst	26.68	27.83	42.28
Saponification value	KOH (mg) / oil(gm)	198.00	192.00	205.00
Molecule weight	gm/mol	876.16	861.00	870.00

A three neck round bottom flask is taken for the reaction and its middle neck is attached to the reflux condenser. The reflux condenser allows volatile reactants, such as alcohols (methanol) to evaporate due to high temperature and then condensed liquid again recirculated into the reaction mixture. By which it prevents loss of these valuable reagents, ensuring efficient utilization and maximizing the yield of biodiesel. One flask neck is connected with the temperature indicator controller and another neck was used for the inlet of the reactant in the flask. For biodiesel synthesis, batch experimental system was assembled in which three neck round bottom flask was putted on the magnetic hot plate stirrer as shown in figure 1. Oil (Edible) of 100 ml was first preheated (60°C) and, later (methanol + catalyst) solution was added in to preheated oil. This reaction breaks down the oil to fatty acid methyl esters (FAME) and glycerol. After 60 min, the product mixture was collected in separating vessel and it was allowed to settle down for around 3-4 hr. After 3-4 hr, two discrete phases arose. The lighter or top layer consists biodiesel and bottom or heavier layer consist of glycerol. After that, glycerol was separated from biodiesel layer and biodiesel was stored in any other beaker for next experimental step. The biodiesel was washed with heated distilled water (40°C) to remove any remaining catalyst, soap, and impurities. Multiple washings (3-4 times) are necessary to achieve the desired purity. After water wash biodiesel was heated at 110°C for 30 min which ensure the final product meets quality specifications, enhances stability, and improves process efficiency in biodiesel production. After heating product must have to cool till room temperature and yield was measured by measuring cylinder. The yield of biodiesel was determined as described in the equation 1:

$$\text{Yield (\%)} \text{ of biodiesel} = \frac{\text{Biodiesel produced in ml}}{\text{Feedstock used in ml}} \times 100 \quad (1)$$



**Figure 1.** Biodiesel production procedure by transesterification reaction. 1) MeOH & KOH catalyst mixture. 2) 3-neck bottom (round) flask. 3) Temperature indicator sensor. 4) Condenser column. 5) Magnetic stirrer with heating plate. 6) Separating funnel with stand for separation.

### Biodiesel Characterization

The purity of the Mixture of fatty esters was predicted using a GC (Gas chromatography) analysis technology (Shimadzu, GC-2010 Plus instrument, Japan). In performance of this experiment, 0.2 $\mu$ L sample was mixed with heptane solvent (AR grade) and injected into the inlet port of the GC instrument. After analysis of GC graph, we get the retention time of each and every biodiesel component.

### RESULTS AND DISCUSSION

The comparative study of KOH & NaOH as the homogeneous catalyst for producing biodiesel are shown in Table 2. The Experimental study revealed that sunflower oil biodiesel gives greater biodiesel yield from KOH catalyst as compared to NaOH as homogeneous catalyst and palm oil and cotton seed oil as edible oil feedstock. The optimum condition are 1gm of catalyst, [6:1] Alcohol (methanol) / oil molar ratio, 55 min experimental time and 60°C temperature.

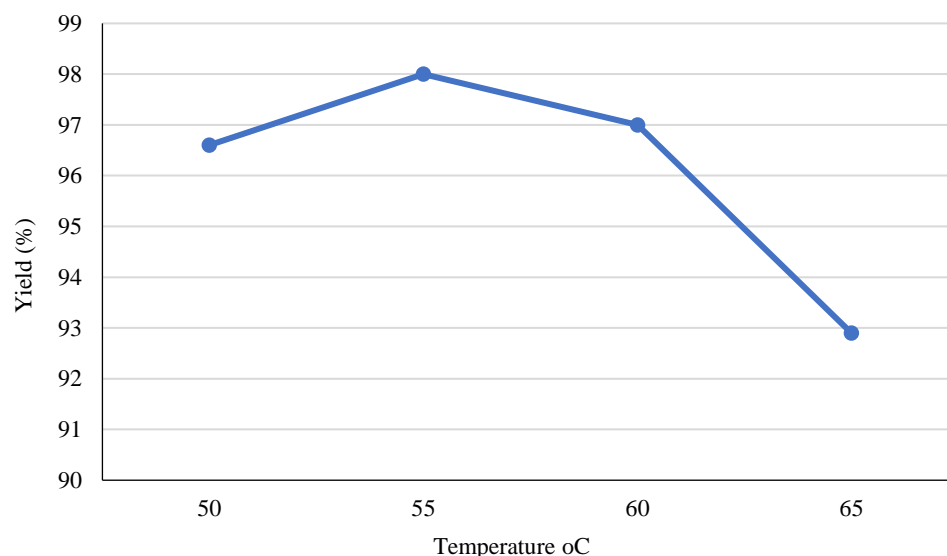
Experimental results confirm that KOH is more effective and basic in nature than NaOH in biodiesel production because KOH has higher ionic size and weaker bond strength between K and OH group. The ionisation of KOH is also stronger than NaOH. Among all edible oils, sunflower oil is found to be best edible oil for gaining higher biodiesel yield. It is because sunflower oil confirm the presence of higher amount of unsaturated fatty acid methyl ester (oleic acid (C18:1), linoleic acid (C18:2) by GC analysis which are mainly responsible for the higher yield of biodiesel.

### Effects on Sunflower oil Biodiesel Yield by Temperature

In this experimental section, Biodiesel production process parameters were optimised for getting maximum biodiesel yields. Therefore, to get the highest product yield, experiments was perform in the temperature range of 50°C-65°C. By performing experiment, the highest yield i.e. 99% for sunflower oil and KOH as a catalyst was obtained at 55°C. After 55°C, the yield was instantaneously because at higher temperature, methanol evaporate and due to the methanol loss, yield decreases as shown in figure 2. To prevent evaporation of alcohol, the transesterification reaction temperature should be less than boiling point of alcohol i.e. 64.7°C (methanol) [23].

**Table 2.** Determination of biodiesel yield of different oils for KOH and NaOH catalyst.

Catalyst	KOH			NaOH		
Oil	Sunflower	Palm	Cotton seed	Sunflower	Palm	Cotton seed
Yield (%)	98	92	97	95	82	94



**Figure 2.** Effects on sunflower oil biodiesel yield by temperature.

### Effects on Sunflower oil Biodiesel Yield by Alcohol (Methanol)/Oil Molar Ratio

In this experiment we have used methanol as alcohol. Three ranges of MeOH/oils molar ratio (3:1-9:1) used in this optimisation experimental study and 98% yield was obtained at the 6:1 MeOH/oils molar. At low MeOH/oils molar ratio, low biodiesel yield i.e. 85% yield was obtained at 3:1 (Figure 3) because when quantity of Methanol is less or lower, the glycerol separation becomes very difficult and results lower biodiesel yield [24]. Excess use of alcohol also effect the biodiesel yield because of the difficulty of the glycerol layer separation. Therefore 6:1 MeOH/oils molar ratio was found to be the optimum value for getting highest biodiesel yield.

### Effect of Catalyst Quantity

Quantity of catalyst plays critical role in biodiesel synthesis. Here KOH catalyst was used in this experiment. Different amounts of catalyst were used from 0.5- 1.1 gm in transesterification reaction. We observed that when we increase the catalyst amount upto 0.5gm-0.8 gm, biodiesel yield increases from 95%-98% (Figure 4) and then decreased from 0.8-1.1 gm of catalyst used. The reason behind the lower yield of biodiesel at higher catalyst concentration is the soap formation during water washing step. Therefore 0.8 gm catalyst was found to be the optimum catalyst amount for getting highest biodiesel yield [25].

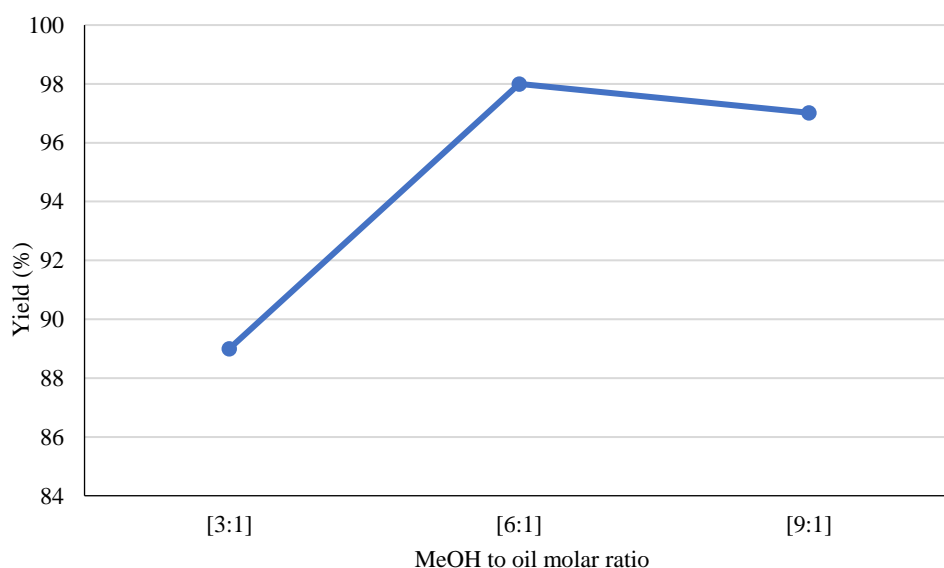
### Effects of Experimental Reaction Time

Three transesterification reactions were performed from 50 min-70 min. The highest yield i.e. 98% was obtained at 55 min reaction time. In graph biodiesel yield is increasing with increasing time but after some time it will decrease (Figure 5) due to reversible nature of the reaction and also some losses of methanol during long reaction time [26]. Therefore 55 min reaction time was found to be the optimum reaction time for getting highest biodiesel yield.

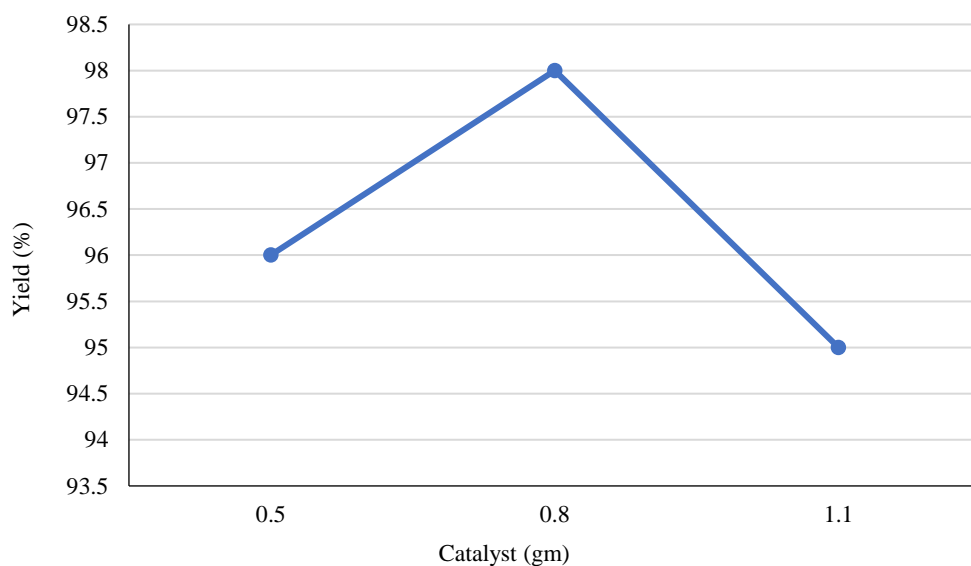
Based on the four different parameters results the best combination for the highest yield was:

1. Temperature: 55°C
2. MeOH to Oils Molar Ratio: [6: 1]
3. Catalyst (KOH): 0.8 gm
4. Experimental Reaction Time: 55 min

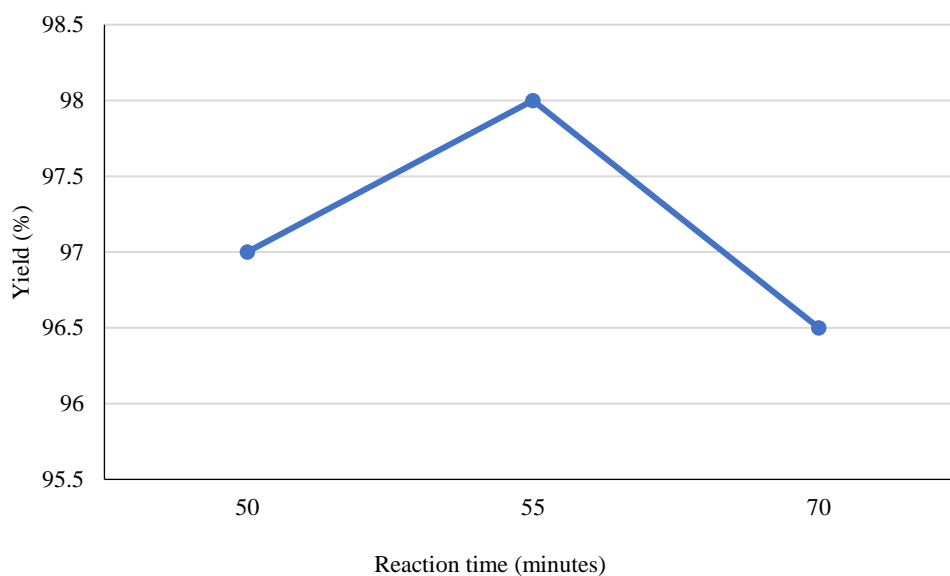
Considering this four Reaction condition one final run was performed by which we get the Maximum highest produced yield as compared with other reaction condition. The biodiesel yield observed was 99%.



**Figure 3.** Effects on sunflower oil biodiesel yield by Alcohol (MeOH)/oil molar ratio.



**Figure 4.** Effect on sunflower biodiesel yield by catalyst quantity.



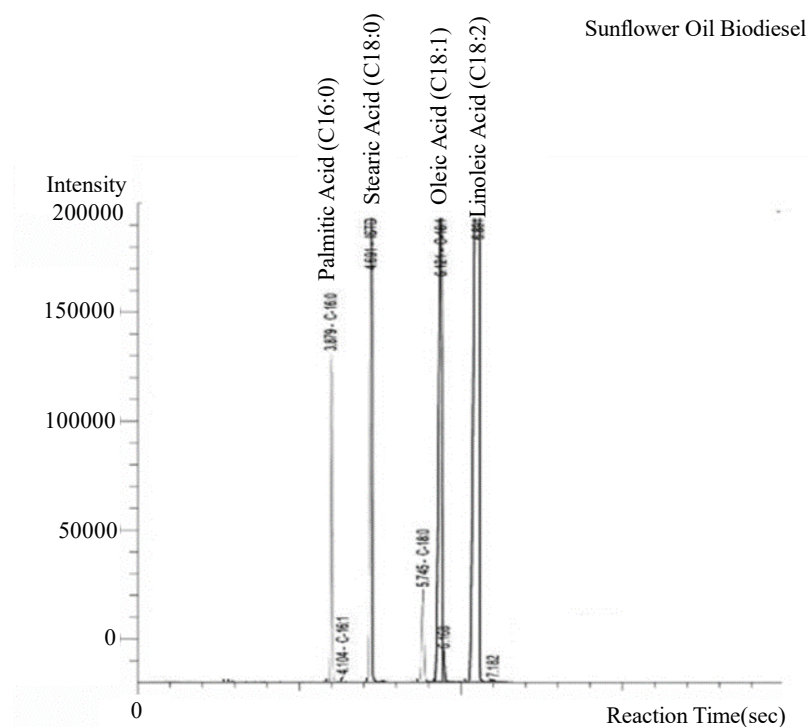
**Figure 5.** Effects on sunflower oil biodiesel by experimental reaction span.

**Table 3.** Measured physical properties of Biodiesel (Sunflower Oil).

Properties	Unit	Biodiesel	ASTM Standard
Density	(gm/ml)	0.869	0.860-0.900
Acid Value	KOH (gm)/Oils(gm)	0.642	0.50
Kinematic viscosity	At 40°C (Cst)	4.67	1.9-6.0
Pour point	°C	7	-15 to 10
Cloud point	°C	12	-3 to 12
Fire point	°C	169	140-215
Flash Point	°C	161	130-170

**Biodiesel Properties Measurement**

The Sunflower oil biodiesel properties were tested manually and were compared with ASTM D6751 Standard as illustrated in Table 3 [27].



**Figure 6.** Produced biodiesel (Sunflower) GC analysis.

### **GC (Gas Chromatography) Analysis Testing**

The GC graph of Sunflower oil biodiesel indicates the presence of FAME components such as palmitic acid, stearic acid, oleic acid and linoleic acid as showed in Figure 6. Graph shows the presence of higher amount of unsaturated fatty acid methyl ester (oleic acid (C18:1), linoleic acid (C18:2) which are mainly responsible for the higher yield of biodiesel. The palmitic acid (C16:0) peak was obtained at standard retention time of 3.879. Similarly different fatty esters like stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2) peak was observed in GC chromatogram at standard time of 5.745, 6.121, 6.891. Comparison of standard graph and GC analysis graph confirmed the purity of FAME from sunflower oils [28].

### **CONCLUSION**

Three different edible oils (Palm oils, cotton-seed oils and sunflower oils) and two homogeneous catalyst (KOH and NaOH) was used to make the best combination of oil and catalyst for biodiesel production. The experimental results was found that sunflower oil and KOH are the best combination for the highest biodiesel yield. Therefore, four different parameters (reaction time, temperature, catalyst quantity and MeOH/Oil ratio) were tested for optimised reaction parameter using sunflower oil and KOH. Optimal reaction parameters for production of biodiesel from sunflower oil were chosen as follows: - 0.8 gm quantity of catalyst (KOH), 55°C temperature, 55 min experimental procedure time, [6:1] MeOH/oils molar ratio. Predicted and observed biodiesel yields were 99.5% and 99%, respectively under optimization conditions. Produced biodiesel properties such as viscosity, flash point, fire point, cloud point etc were also estimated and checked by the biodiesel standard ASTM D6751. It was observed that all biodiesel properties are found within the ASTM limit. These results indicate that KOH serves as a homogeneous catalyst, and sunflower oil, as an edible oil, forms the optimal combination for achieving the highest biodiesel yield and desirable properties under optimized reaction conditions.

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### Author Contributions

Kunal Argade and Dhaval Shrimali has collected the data and written the manuscript. Dr. Jharna Gupta, Dr, Nitin Bhate & Dr. Nikul K. patel have given valuable suggestions for finalizing the manuscript.

### Competing Contributions

The authors declare that they have no conflict of interest regarding this publication.

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