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# Review of The Production of Biodiesel from Waste Cooking Oil Using Acid/Alkaline Based Catalysts

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Abstract: Biodiesel has proved to be the greatest substitute for diesel due to its unique characteristics, such as substantial reductions in greenhouse gas emissions, non-sulfur emissions, non-particulate matter pollutants, low toxicity, and biodegradability. In its most basic definition, it is a mono alkyl ester of long-chain fatty acids having characteristics that are similar to those of fossil-fuel-derived diesel. Waste cooking oil (WCO), which has tremendous potential as a raw material, is to produce biodiesel. In the case of waste cooking oil with a high proportion of free fatty acids, the use of alkaline-based catalyst would generate soap through the saponification process, and thus, lowers the biodiesel conversion. Hence, the waste cooking oil would first treat with an acid-based catalyst to undergo an esterification reaction, which also requires high working conditions, in order to minimize the quantity of free fatty acid concentration. This study investigated the benefits and drawbacks of using different catalysts, such as homogeneous or heterogeneous catalysts, that have been used in recent years. Previous researches have shown that the selection of catalyst may be selected based on several characteristics that affecting the biofuel production process, such as reaction rate, catalyst concentration, temperature, catalyst type, alcohol used, alcohol to oil ratio, free fatty acid content, and water content.

**Keywords**: Biodiesel, Homogenous Catalyst, Heterogeneous Catalyst, Esterification, Transesterification, Waste Cooking Oil (WCO)

### 1. Introduction

Biodiesel is a sustainable and biodegradable fuel manufactured from vegetable oil, animal fats, and waste cooking oil (WCO). The biodiesel output in the world is continuously expanding, according to the Organization for Economic Cooperation and Development (OECD) and the United Nations Food and Agriculture Organization (FAO) [1]. Biodiesel could also be produced from a variety of vegetable oils, including palm oil, sunflower oil, soybean oil, rapeseed oil, and castor oil. Waste cooking oil (WCO) is a source of concern in many regions of the world. WCO might translate these concerns into

economic and environmental advantages by effectively using and regulating WCO as a fuel alternative. Many industrialized countries have put in place regulations that make the release of WCO into waste drains prohibited [2]. For this research, waste cooking oil (WCO), including triglycerides and the catalyst, will be used in this process to improve the reaction rate used to produce the biodiesel, which may successfully cut feedstock cost by 60.00-70.00 %. It has been claimed that the cost of raw materials accounts for about 70.00-95.00 % of the entire manufacturing cost. Similarly, the cost of the catalyst has an impact on the entire manufacturing cost [3].

Biodiesel is typically created using a single-step transesterification process catalysed by alkaline catalysts. However, suppose the free fatty acid concentration is more than 2 wt%. In that case, a two-step process may be necessary, with the first step being acid catalysed alcoholysis, also known as esterification, and the second step being transesterification [4]. The first stage of esterification aims to minimize the free fatty acid content of the feedstock oils as much as possible to reduce soap production in the second phase, transesterification. Soap formation prevents the separation of biodiesel and glycerol phases and requires a greater volume of water for washing. It is the main cause of biodiesel loss due to the esterification process

There are some factors that affecting the production of biodiesel from waste cooking oil such as water content, free fatty acid (FFA), type of alcohol, methanol to oil ratio, catalyst type, catalyst concentration, temperature and reaction of time. Water is produced as a byproduct when acid catalysts are used to esterify free fatty acids for masters. Water naturally inhibits acid-catalyzed processes, reducing engine efficiency. Waste cooking oil that has been used contains more free fatty acids than new cooking oils. Consequently, higher quantities of free fatty acids are known to result in the creation of soap and water. Even with a homogenous alkaline-based catalyst, the transesterification process will fail. This problem might be solved by using a heterogeneous catalyst and pretreatment with an acid catalyst.

This study focuses more on the catalyst used in this biodiesel production. Catalyst is a substance that can increase the rate of chemical reaction in the process. In this study, the different ratio from homogenous catalysts such as alkaline and acid will be focused to analyze the most efficient catalysts in biodiesel production from waste cooking oil at ambient temperature (40 to 65°C). When raw materials include a high concentration of free fatty acids, acid catalysts (homogeneous or heterogeneous) are utilized to catalyse the esterification process. An acid catalyst may be used in both esterification and transesterification processes concurrently. However, the recovery of the catalyst from the final product and the generation of soap are two significant drawbacks of using a homogeneous catalyst.

# 2. Literature review

WCO, or vegetable oil, combines long-chain monoalkylic esters produced from green fatty acids utilized in diesel engines. According to ASTM International (American Society for Testing and Materials), it is a short-chain monoalkylic ester produced from fatty acids found in vegetable oil, according to ASTM International (American Society for Testing and Materials). Specifically, pure biodiesel is labelled as B100, while blends of biodiesel are labelled as BX, with X indicating the proportion of biodiesel in the mixture. The most well-known ratio is B20, which is 20.00 % biodiesel and 80.00 % diesel in the fuel mix [5]. Because it is derived from natural fuels and emits less pollution than petroleum diesel, biodiesel, which is made from bio-oil, can be a viable alternative to diesel fuel. It can reduce carbon dioxide ( $CO_2$ ) emissions by 78.00 % and carbon monoxide ( $CO_2$ ) emissions by 50 % compared to petroleum diesel [6]. WCO is becoming more popular in Malaysia for biodiesel production because of the low cost of production and the large quantity of trash produced by each household in the country. According to government statistics, the average Malaysian household generates 2.34 kg of WCO each month [7].

### 2.1 Basic Reaction Mechanism Involved

### i. Transesterification

The triglyceride component of oil combines with the alcohol in the presence of NaOH or any other catalyst to produce ester and glycerol, as illustrated in the Figure 1, depicts the overall scheme of the transesterification process, where R represents a mixture of different fatty acid chains. As seen in Figure 1, complete conversion of the triglyceride requires three reversible interactions involving monoglyceride and diglyceride intermediates [8].

Figure 1: Reaction for transesterification process

# ii. Saponification Reaction

If there are free fatty acids in the vegetable oil, they will react with the homogeneous base catalyst to generate soap and water. The main disadvantages of this reaction are the need for catalyst and the increased difficulty in the separation process, which results in high manufacturing costs. Furthermore, the formation of water in the product will inhibit the reaction. The saponification process is shown in Figure 2.

$$R_1$$
-COOH+NaOH $\longrightarrow$   $R_1$ COONa + $H_2$ O

FFA Sodium hydroxide Soap Water

Figure 2: Saponification process

# iii. Hydrolysis Reaction

Water from vegetable oil or water created during the saponification procedure will hydrolyze triglycerides, creating additional free fatty acids. The hydrolysis process is shown in Figure 3.

Figure 3: Hydrolysis process

### iv. Esterification

Most biodiesel specifications have a maximum FFA level since FFAs may cause deposits and engine damage. Figure 4 shows how esterification may be utilized to convert FFAs to Biodiesel and lower FFAs. Fatty acid combines with alcohol in the presence of a catalyst to form fatty acid alkyl ester (biodiesel) [9].

Figure 4: The esterification reaction

# 2.2 Process of Synthesizing Biodiesel

Biodiesel may be produced in various techniques, including direct usage and mixing, microemulsions, thermal cracking, and the most common process is transesterification. Biodiesel is typically created using a single-step transesterification process catalysed by alkaline catalysts. However, suppose the free fatty acid concentration is more than 2 wt %. In that case, a two-step process may be necessary, with the first step being acid catalysed alcoholysis, also known as esterification, and the second step being transesterification [9]. The first stage of esterification aims to minimize the free fatty acid content of the feedstock oils as much as possible to reduce soap production in the second phase, transesterification. Soap formation prevents the separation of biodiesel and glycerol phases and requires a greater volume of water for washing. It is the main cause of biodiesel loss due to the esterification process [10]. Figure 5 shows a schematic design for single and two-step biodiesel production. The goal of the two-step process is to reduce the manufacturing process's operating costs by decreasing the alcohol ratio, reducing reaction time, and minimizing yield loss.

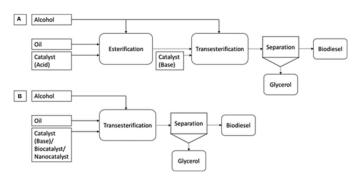


Figure 5: Biodiesel production from feedstock: (A) two-step process;

# (B) single-step process

# 2.3 Factor Affecting Biodiesel Production

# i. Type of Alcohol

Primary and secondary alcohols with carbon lines ranging from 1 to 8 are utilized to make biodiesel [11]. The use of methanol has the advantage of quickly reacting with triglycerides (polar and shortest chain alcohol). It is a relatively inexpensive alcohol, whereas the same reaction using ethanol has the disadvantage of the produced ethyl esters being less stable and leaving a carbon residue after the reaction. Even though ethanol has a more excellent boiling point, flash point, viscosity, and kinematic viscosity, methanol has a greater melting point due to its greater polarity.

### ii. Methanol to Oil Ratio

Three moles of alcohol and one mole of triglyceride are required to produce three moles of alkyl esters. A methanol-to-oil ratio is always beneficial to biofuel conversion. As the

reactant concentration rises, so does the rate of product synthesis. As a result, if the alcohol concentration is automatically raised, the rate of product creation will be enhanced. Increases in the molar ratio of methanol to oil will result in more product production. The recovery of glycerol and unreacted methanol becomes tedious and raises the cost of the finished biodiesel by raising the cost of post-treatment. Methanol was produced utilizing varying molar ratios of alcohol to oil and a NaOH catalyst to transesterify waste sunflower oil. A 6:1 molar ratio of alcohol to oil yielded the best yield of 99.50 % methyl ester. However, when utilized to transesterify waste canola oil at a 1:1 molar ratio of methanol to oil, the output was 49.50 % [12].

# iii. Temperature

The temperature has a significant impact on the transesterification process. When the reaction temperature is raised, the rate of reaction and product yield tend to rise as well. The temperature should not be higher than the alcohol's boiling point. This will prevent alcohol from vaporizing. However, if the reaction temperature is kept below 50 °C, the viscosity of the biodiesel will rise. In some instances, waste cooking oil is heated to 120 °C to eliminate water particles in the raw material before being cooled to 60 °C. Freedman et al. discovered no change in conversion between 45 °C and 60 °C. However, at 32 °C, conversion was substantially lower than at 45 °C and 60 °C. After 4 hours, conversion at 32 °C was slightly greater than conversion at other temperatures. Using the KOH catalyst, waste cooking oil from household and commercial sources produced the greatest yield of 65.00 % for all feedstocks evaluated. To maximize biodiesel output, the reaction time was drastically decreased [12].

# iv. Reaction Time

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# v. Type of Catalyst

In general, three types of catalysts are used in the manufacture of biodiesel: homogeneous, heterogeneous, and enzymes, and it is a chemical that speeds up a process by decreasing its activation energy. A catalyst was retrieved intact and did not appear in the product. Compared to homogeneous and heterogeneous catalysts, biodiesel generation is more widely utilized, and excess catalysts would result in higher manufacturing costs and decreased product yield [6]. This study focuses more on the homogeneous and heterogeneous catalysts.

# 3. Methodology

# 3.1 Project flowchart

This project's method is shown in Figure 6, which includes a list of each stage. Earlier research was carried out to understand the appropriate subject matter for this study's objectives and scope.

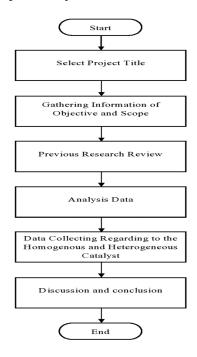


Figure 6: Project flowchart

# 3.2 Research flowchart

Figure 7 shows the overall flow of the research for the production of biodiesel by a two-step transesterification process, which is very important to illustrate the sequence of operations to finish this research. The data will be measured from the yields that the previous research reported.

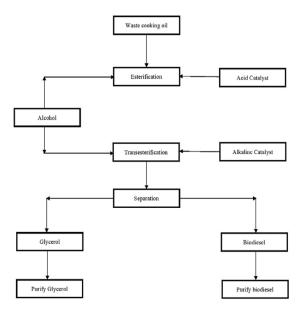


Figure 7: The overall flow of the research for the production of biodiesel.

# 3.3 Research Strategy

The proposed study took the form of new research but on an existing research subject. The research held concerning this dissertation was an applied one, but not new. Instead, numerous pieces of previous academic research exist regarding biodiesel production from WCO using acid and alkaline catalysts.

# 3.4 Data analysis

The main advantage of content analysis is that it helps in data collected being reduced and simplified. Moreover, data analysis gives the ability to researchers to structure the catalyst used in the production of biodiesel. It focuses more on homogeneous and heterogeneous catalyst where it is based on alkaline and acid catalyst that satisfies research objectives.

### 4. Results and Discussion

# 4.1 Method for the production of biodiesel from WCO

If the FFA level exceeds 2 wt %, the first step of esterification is followed by the second transesterification step. The purpose of the first step is to reduce the FFA level as much as possible and transform the oil into a suitable feedstock oil for the second transesterification step. This two-step process has been demonstrated to be the most successful method for producing biodiesel from any feedstock oil [13]. Table 1 summarizes the two-step transesterification process that is most typically used to produce WCO biodiesel.

Table 1: The two-step transesterification process that is most typically used to produce WCO biodiesel

		Reaction conditions				
No.	Catalyst	Temperature, °C	Type of alcohol (alcohol to oil molar	Catalyst concentration	— Biodiesel yield (Waste	
			ratio)	(w/w %)	Cooking	
					Oil,	
					WCO	
					%)	
1.	Ferric sulfate	Acid: 100	Acid: Methanol (9:1)	Acid: 2.0	96.00	
	follow by KOH	Base: 100	Base: Methanol (7.5:1)	Base: 1.0		
2.	Ferric sulfate	Acid: 95	Acid: Methanol (10:1)	Acid: 2.0	97.00	
	follow by KOH	Base: 65	Base: Methanol (6:1)	Base: 1.0		
3.	Ferric sulfate	Acid: 60	Acid: Methanol (7:1)	Acid: 3.0	81.3	
	follow by CaO	Base: 60	Base: Methanol (7:1)	Base: 3.0		

# 4.2 Homogenous catalyst

### 4.2.1 Alkaline-based catalyst

Three alkaline catalysts — sodium hydroxide (NaOH), potassium hydroxide (KOH), and sodium methoxide ( $CH_3ONa$ ) — are often employed in alkaline-based catalyst transesterification, which is more costly and requires the complete absence of water [14]. Figure 8 shows the final product mixtures' states settled overnight after UFO transesterification with various catalysts. After settling overnight with a NaOH or  $CH_3ONa$  catalyst, the resultant mixture split into two layers: a liquid crude ester layer and a glycerol layer (Figure 8 (a)). As solid soaps fell to the bottom, a combination of glycerol and solid soap developed. This meant that the top layer of the separation unit had to be decanted or pulled out

from the top, since it could not flow out straight from the bottom. KOH is frequently utilized in the production of biodiesel from waste recycled oil. After settling overnight, the product mixture split into two liquid layers: a crude ester layer on top and a glycerol layer on the bottom (Figure 8 (b)). Due to the softer nature of potassium soap compared to sodium soap, the residues generated during the transesterification process do not sink to the bottom layer but rather suspend within it.

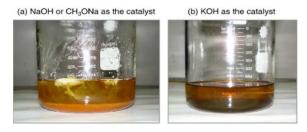


Figure 8: The final product mixtures settled overnight, produced from the transesterification of UFO using NaOH, CH<sub>3</sub>ONa and KOH catalysts

Table 2 list the common homogeneous base catalysts used in the biodiesel production of WCO. By Obtaining 98.26 % conversions with a concentration of 1.16 % KOH in proportion to the volume of oil takes around 62.4 hours, while by using 0.70 % KOH and methanol 9:1 at 50 °C, the yields obtained was 90.70 %. Other than that, a transesterification procedure at 65 °C using 0.60 % NaOH and methanol (4.8:1), yielding 98.00 % biodiesel. Lastly, a yield of biodiesel (97.90 %) is produced by using a 0.75 %  $CH_3$ ONa and molar methanol to oil ratio (6:1).

Table 2: The common homogeneous base catalysts used in the biodiesel production of WCO

		]			
No.	Catalyst	Temperature °C	Type of alcohol (alcohol to oil molar ratio)	Catalyst concentration (w/w %)	- Biodiesel yield (Waste Cooking Oil, WCO %)
1.	Potassium hydroxide (KOH)	50	Methanol (9:1)	0.7	90.7
2.	Potassium hydroxide (KOH)	62.4	Methanol (9.4:1)	1.16	98.26
3.	Sodium hydroxide (NaOH)	70	Methanol (7.5:1)	1.1	85.3
4.	Sodium hydroxide (NaOH)	65	Methanol (4.8:1)	0.6	98
5.	Sodium methoxide (CH <sub>3</sub> ONa)	750 W microwave power	Methanol (6:1)	0.75	97.9

NaOH was selected as the most efficient catalyst since it is the cheapest and gives high yields. This can be explained by the fact that sodium has a lower molecular weight which leads to higher transesterification reaction rates.

# 4.2.1 Acid-based catalyst

The esterification process is catalyzed by Bronsted acids, preferably by sulfonic and sulfuric acids as well as hydrochloric acid [15]. These catalysts produce very high yields in alkyl esters. The common homogeneous acid catalysts used in the synthesis of WCO are listed in Table 3. Obtaining a higher yield, 99.00 % with a concentration of 41.80 % sulfuric acid in proportion to the volume of oil takes around 50 hours at 70 °C by using methanol (245:1). Alcohol consumption is also increased when an acid catalyst is used. Next, high yield of biodiesel (99.00 %) is produced by using a large quantity of sulfuric acid catalyst (41.80 % by weight) and high molar methanol to oil ratio (245:1). Due to its cheap cost, strong reactivity, and high biodiesel output. Table 4 shows the advantages and drawbacks of homogenous alkaline catalysts and homogenous acid catalysts used in the transesterification of WCO.

Table 3: The common homogeneous acid catalysts used in the synthesis of WCO

		]			
No.	Catalyst	Temperature, °C	Type of alcohol (alcohol to oil molar ratio)	Catalyst concentration (w/w %)	- Biodiesel yield (Waste Cooking Oil, WCO %)
1.	Sulfuric acid $(H_2SO_4)$	95	Methanol (20:1)	4.0	90
2.	Sulfuric acid $(H_2SO_4)$	70	Methanol (245:1)	41.8	99
3.	Sulfuric acid $(H_2SO_4)$	95	Methanol (20:1)	4.0	>90

Table 4: The advantages and drawbacks of homogenous alkaline catalysts and homogenous acid catalysts used in the transesterification of WCOs [5]

	Homogenous Alkaline	Homogenous Acid
	Strong catalytic activity	Strong catalytic activity
Advantages	• Inexpensive and widely	• Suitable for feedstock with
	available	high FFA
	<ul> <li>No corrosive</li> </ul>	• Do not form soap
	• Ideal for TGAs with low	
	FFA	
	• Possible formation of soap	• The reaction rate is slower
Disadvantages	Not suitable for feedstock	compared to the base catalyst
	with high FFA	Separation and reuse unusual
	No reusability	Corrosion problem
	• Requires extensive	
	washing	

# 4.2 Heterogeneous catalyst

In general, heterogeneous alkaline catalysts are synthesized in various ways, including impregnation of base metals, precipitation, calcination, and co-precipitation. Calcined waste coral pieces in solid form to transesterify WCO, palm oil, soybean oil, and rice bran with methanol to produce biodiesel and glycerol as a by-product. Coral fragments calcined at 700 °C for 1 hour, a catalyst/oil ratio of 100.00 %, a methanol/oil molar ratio of 15:1, and a reaction temperature of 65 °C with steady stirring may be used to make biodiesel with a FAME yield of over 98.00 % in 2 hours [5]. At 65 °C and 1 atm, the synthesized mixed oxide catalysts comprised of CaO and ZrO2 mixed oxides with varying Ca-to-Zr molar ratios were employed to transesterify WCO with methanol to create biodiesel. The experimental results reveal that when the Ca-to-Zr molar ratio grows, the activity of the produced catalysts increases, but the catalysts' stability diminishes. At 65 °C, with a catalyst loading of 10.00 %, a methanol-to-oil molar ratio of 30:1, and a reaction period of 2 hours, a biodiesel yield of 92.10 % could be attained over the CaO-ZrO2 catalyst with a Ca-to-Zr molar ratio of 0.5.

Heterogeneous acid catalysts made from insoluble carbohydrates in the solvents and liquid reactants tested (water, methanol, n-hexane, t- butanol, oleic acid, triolein and WCO). In this review, a high yield of biodiesel (98.50 %) is produced by using a lower quantity of heterogeneous acid catalyst (2.10 % by weight) and the methanol to oil ratio (20:1). At 80 °C, with a catalyst loading of 10.00 %, a methanol-to-oil molar ratio of 30:1, and a reaction period of 8 hours, a biodiesel yield of 92.00 % could be attained over the carbon-based from starch. Table 5 The common homogeneous acid catalysts used in the synthesis of WCO. Table 6 shows the advantages and drawbacks of heterogeneous alkaline catalysts and heterogeneous acid catalysts used in the transesterification of WCOs.

Table 5: The common homogeneous acid catalysts used in the synthesis of WCO

	Catalyst		Molar ratio			
Catalyst	concentration	Feedstock	(methanol: oil)	T, °C	Time reaction	Yield
	(wt %)					(%)
Carbon-based from	10.0	Waste	30:1	80	8h	92
starch		cooking oil				
$Zr_{0.7}H_{0.2}PW_{12}O_{40}$	2.1	Waste	20:1	65	8h	98.5
		cooking oil				
Ferric-manganese	6.0	Waste	25:1	200	8h	92.3
dopedtungstated/mo		cooking oil				
lybdna						
ZS/Si	3.0	Waste	18:1	200	10h	98
		cooking oil				

Table 6: The advantages and drawbacks of heterogeneous alkaline catalysts and heterogeneous acid catalysts used in the transesterification of WCOs [5]

Heterogeneous Alkaline	Heterogeneous Acid
Heterogeneous Mikanne	Tieter ogeneous Aeiu

### Easily separated from the reaction Advantages Insensitive to FFA and water mixture without the use of water as content in the oil the cleaning agent Preferred-method if low-grade oil High possibility of reusing and is used regenerating the catalyst Esterification and A less corrosive character transesterification occur simultaneously Leans towards being safer, cheaper More environmentally friendly Easy separation of catalyst from product Disadvantages Poisoning of the catalyst when Complicated catalyst synthesis exposed to ambient air procedures lead to higher cost Sensitive to FFA content in the oil Normally, high reaction temperature, high alcohol to oil due to its basic property molar ratio and long reaction time Soap will be formed if the FFA content in the oil is more than 2 wt.% are required Too much soap formation will Energy intensive decrease the biodiesel yield and cause Leaching of catalyst active sites problems during product purification may result in contamination Leaching of catalyst active sites may result in contamination

Both homogeneous and heterogeneous catalysis may be used to catalyze transesterification or alcoholysis. Homogeneously catalyzed processes are often quicker and require less loading than heterogeneously catalyzed ones. A critical disadvantage of homogeneous catalysts is that separating these catalysts from the medium is complicated and generally uneconomical; hence, reuse is often impossible. Furthermore, various washing stages connected with catalyst removal from the product involve the use of water (typically deionized) and the formation of substantial wastewater. Table 7 compares homogeneous and heterogeneous catalysts in conversion rate, processing technique, sensitivity to water and FFAs, reusability, and cost of use.

Table 7: compares homogeneous and heterogeneous catalysts in conversion rate, processing technique, sensitivity to water and FFAs, reusability, and cost of use.

Factor	Homogenous catalyst	Heterogeneous catalyst  Medium conversion	
Conversion rate	Fast and high conversion		
Processing technique	The use of the continuous method	Fixed-bed operation is possible	
	is faced with limitations	continuously	
Sensitivity to water and	Sensitive	Not sensitive	
FFAs			
Reusability	Not possible	Possible	
Cost	Fairly costly	Potentially cheaper	

### 5. Conclusion

Homogeneous catalysts (alkaline or acid) have strong catalytic activity over a short period of time and operate at relatively low temperatures (40 to 65 °C at atmospheric pressure). These catalysts have shown the ability to advance the esterification and transesterification processes simultaneously in the two-step process of biodiesel generation.

From this, it has been learnt that the use of heterogeneous catalysts in biodiesel production via transesterification using WCO as a raw material is attracting more attention and is becoming more attractive. Researchers should concentrate on applying such catalysts in terms of both economic and environmental terms. Some researchers have successfully produced biodiesel using these heterogeneous catalysts. Some of them recommend the best catalysts in biodiesel production. Nonetheless, researchers always strive to improve the biodiesel process economically using environmentally friendly catalysts and selecting the best process technology.

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