


<p><b>KLM Technology Group</b></p> <p>Practical Engineering Guidelines for Processing Plant Solutions</p>	 <p><b>Engineering Solutions</b></p> <p><b>Consulting, Guidelines and Training</b></p> <p><a href="http://www.klmtechgroup.com">www.klmtechgroup.com</a></p>	<p>Page : 1 of 85</p> <hr/> <p>Rev: 01</p> <hr/> <p>Rev 01 February 2025</p>
<p>KLM Technology Group #033, Jalan Bayu 8/1, Taman Nusa Bayu, 79200 Iskandar Puteri, Johor, Malaysia</p>	<p><b>Kolmetz Handbook of Process Equipment Design</b></p> <p><b>BIODIESEL MANUFACTURING</b></p> <p><b>(ENGINEERING DESIGN GUIDELINES)</b></p>	<p>Co Authors Rev 01 – Riska Ristiyanti</p> <hr/> <p>Editor / Author Karl Kolmetz</p>

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		Rev: 01
		February 2025

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		Rev: 01
		February 2025

## TABLE OF CONTENT

INTRODUCTION .....	6
Scope.....	8
General Design Consideration.....	9
Biodiesel Production Techniques .....	12
Biodiesel Properties and Specification .....	20
Property Requirements and Specified Methods for B100.....	22
DEFINITION .....	28
REFERENCES .....	30
THEORY.....	31
FEEDSTOCKS USED IN BIODIESEL PRODUCTION.....	31
Fats and Oils .....	31
Alcohol.....	32
Catalysts.....	33
BIODIESEL PRODUCTION PLANT.....	35
Feedstock Preparation.....	35
Biodiesel Production Process Option .....	45
Basic Plant Equipment and Operation.....	53
Chemical Plant Controls .....	61
Biodiesel Recovery Process .....	68
Biodiesel Transportation and Storage .....	82

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		Rev: 01
		February 2025

## LIST OF FIGURES

Figure 1. Different types of vegetable Oils used for Biodiesel Production.....	9
Figure 2. Process of the Biodiesel Production Process .....	17
Figure 3. Flow Diagram for Soybean Oil Deguming.....	40
Figure 5. Reduction of Fatty Acids by Glycerolysis .....	43
Figure 6. Batch Reaction Process.....	47
Figure 7. Plug Flow Reaction System.....	48
Figure 8. Acid Catalyzed Direct Esterification Process .....	50
Figure 9. Preparation of Soapstock from a High FFA Feed .....	51
Figure 10. Biox Co-Solvent Process .....	52
Figure 11. Supercritical Esterification Process.....	53
Figure 12. Centrifugal Pump .....	57
Figure 13. Centrifuge .....	58
Figure 14. Thermocouple.....	62
Figure 15. Manometer.....	64
Figure 16. Displacer Transmitter.....	65
Figure 17. Differential Pressure Transmitter .....	66
Figure 18. Flowmeters .....	67
Figure 19. Flash Evaporator .....	70
Figure 20. Thin Film Evaporator.....	71
Figure 21. Falling Film Evaporator.....	72
Figure 22. Distillation Set-Up .....	73

These design guidelines are believed to be as accurate as possible, but are very general and not for specific design cases. They were designed for engineers to do preliminary designs and process specification sheets. The final design must always be guaranteed for the service selected by the manufacturing vendor, but these guidelines will greatly reduce the amount of up front engineering hours that are required to develop the final design. The guidelines are a training tool for young engineers or a resource for engineers with experience.

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		Rev: 01
		February 2025

## LIST OF TABLES

Table 1. Chemical Structure of Common Fatty Acid and Their Methyl Esters .....	11
Table 2. Characteristic of Common Fatty Acids and Their Methyl Esters .....	12
Table 3. Composition of Various Oils and Fats .....	14
Table 5. ASTM D 6751 – 02 Requirements .....	21
Table 4. Average Composition for Crude and Refined Soybean Oil .....	36

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		Rev: 01
		February 2025

## INTRODUCTION

Energy demand is rising due to population growth, industrial development, and transportation needs. Consumption rates are high, rapidly depleting supplies. Additionally, burning fossil fuels causes significant air pollution.

Governments, research communities, and private organizations worldwide are seeking alternative and renewable energy sources. Energy can be generated from non-conventional sources such as agricultural waste, plants, solar energy, wind, geothermal, animal, and human waste. Renewable energy resources have the potential to meet high energy demands without causing severe environmental impact.

Biodiesel is a renewable energy source that can be produced at any time and place. It does not require a large storage system, and there are multiple sources for its production. Therefore, biodiesel provides a practical and sustainable option for addressing energy needs.

Biodiesel is a chemically modified eco-friendly fuel mainly used as a fuel in compression ignition engines. It is prepared from vegetable oil or animal fat. It can also be produced from algae oil. It is gaining worldwide popularity because it is a renewable, non-toxic, biodegradable, and non-flammable fuel.

Technically, biodiesel is defined as mono-alkyl esters of long fatty acids obtained from plant oil or animal fat and meets the requirements of ASTM D6751. ASTM method provided detailed specifications for biodiesel. It includes standard test methods for determining the properties of biodiesel and the range/limits of the properties. Biodiesel is not raw fuel.

It is obtained from vegetable oil or animal fat through the process known as transesterification or esterification. The transformation of feedstock into ester brings about a severe change in their characteristics. The properties of biodiesel vary depending on the source. This is essentially related to the chemical structure of the source used.

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		Rev: 01
		February 2025

Compared to fossil diesel, the average composition of the fuel has a considerable amount of oxygen (11%) and a reduced amount of carbon (77%), while the hydrogen content is similar. For this reason the LHV (Low Heating Value) is typically 10% lower, the stoichiometric air-to-fuel ratio has a typical value of 12.5 and the combustion is generally improved.

Biodiesel is completely miscible with petroleum diesel fuel, and is generally used as a blend, including ASTM International Standard D975 grades No. 1 and No. 2 diesel fuel or ASTM International Standard D396 heating oil, as well as other distillate and residual fuel oils. The mixtures of biodiesel and diesel are by and large alluded to as B5, or B20, etc., where the number denotes the percentage of biodiesel in the mixture. B5 means a blend containing 5% (by vol.) biodiesel and 95% (by vol.) diesel in 1 gallon of fuel the remainder of the fuel can be No. 1 or No. 2 diesel, heating oil, or any other distillate or residual fuel. Pure (or neat) biodiesel is called B100.

Biodiesel is made by reacting a triglyceride (the component of oil or fat) or a fatty acid with an alcohol to produce a fatty acid alcohol ester, usually with the aid of a catalyst. It can be produced from many different alcohols but methanol is the simplest, most reactive and least expensive of all the alcohols, which makes it the product of choice for biodiesel producers.

Most of the biodiesel production methods require an excess of methanol over the stoichiometric ratio required for the reaction. Typically an excess of twice the required amount is used. Because of the nature of the equilibrium and reversible reaction for biodiesel production, the excess methanol is employed to favor a more complete reaction to achieve a higher quality product.

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		Rev: 01
		February 2025

## Scope

The purpose of this Guideline is to describe and explain the process and issues involved in producing biodiesel. Biodiesel is a prominent alternative of petroleum diesel used worldwide. It is a clean-burning renewable fuel, which has similar properties to petroleum diesel. Biodiesel is mainly composed of fatty acid mono-alkyl esters, which are produced by the transesterification reaction of alcohol and free fatty acids (FFA), derived from renewable resources such as vegetable oils, waste cooking oils or animal fats.

However, the byproduct 'water' formed in transesterification reaction makes the reaction rate sluggish, which results in high residence time, low biodiesel yield and purity in a conventional process. Moreover, separation and purification of biodiesel further add up to the cost process economics.

This engineering design guideline provides an overview of the steps in the production of from preparation of the feedstock to the recovery and purification of the fatty acid esters (biodiesel) and the co product glycerol (also called glycerin) and will review several chemistries used for esterification and different approaches to product preparation and purification. This guideline describes some of the methods used to recover methanol as part of the biodiesel production process, as well as how to refine it for reuse.

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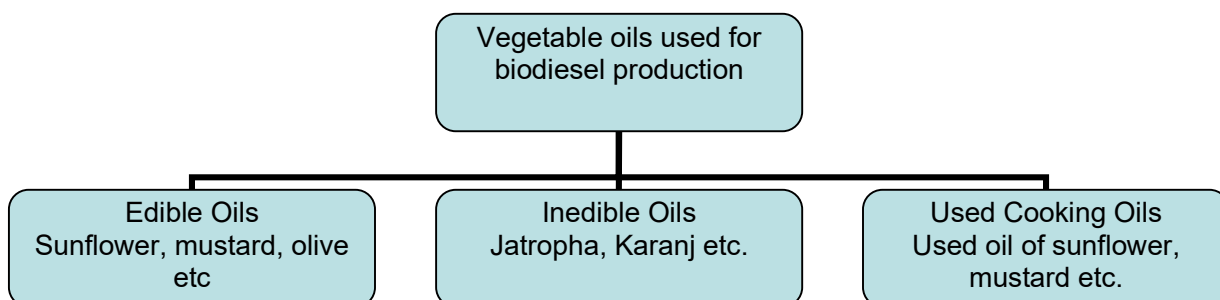
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	<b>BIODIESEL MANUFACTURING</b>	<b>Rev: 01</b>
	<b>ENGINEERING DESIGN GUIDELINES</b>	<b>February 2025</b>

## General Design Consideration

The properties of biodiesel vary depending on the source. This is essentially related to the chemical structure of the source used. Biodiesel can be obtained from edible, inedible, or used cooking oils as depicted in Figure 1.



**Figure 1. Different types of vegetable Oils used for Biodiesel Production**

Edible oil includes sunflower oil, mustard, olive, coconut, etc. Jatropha and karanja oils come under the category of inedible oils. Different edible oils are used for cooking. When cooking oils are heated for a long time, they go through oxidation and produce oxides and polymeric substances. These oxides are harmful to humans. Instead of discarding or disposing of, this oil can be reused for different applications. One such application is biodiesel production.

Waste Frying oil (WFO) or cooking oils have the potentiality to be used as the feedstock of biodiesel production as it is 2–3 times cheaper than virgin vegetable oils. The total manufacturing cost of biodiesel can be considerably reduced if WFO is used instead of fresh oil.

The conversion of this large amount of WFO into fuel can eliminate the environmental impacts caused by the harmful disposal of these waste oils to some extent. The necessity for finding an appropriate approach for converting the large amount of WFO into biodiesel, not only to reduce the harmful effects on environment but also to meet the present energy crisis.

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		Rev: 01
		February 2025

The use of non-edible and used oils is significantly more imperative than edible oils because of their expensiveness and demand as a food item. Biodiesel is a clean-burning fuel obtained from renewable sources. It is non-toxic and produces no pollution. It is sustainable and easy to obtain. The term biodiesel is usually wrongly used.

Biodiesel is the product obtained through the transesterification or esterification reaction of vegetable oil (or animal fat) but it is different from the source. In any case, the term biodiesel is frequently used for neat vegetable oil or a mixture of biodiesel and diesel. Neat vegetable oils that have not undergone conversion reaction, are not biodiesel and contain a higher amount of fatty acids content.

The major components of vegetable oils are triglycerides. The term triacylglycerols is being used more and more. Triglycerides are esters of glycerol with long-chain acids, commonly called fatty acids. Tables 1 and 2 list the most common fatty acids and their corresponding methyl ester.

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		Rev: 01
		February 2025

**Table 1. Chemical Structure of Common Fatty Acid and Their Methyl Esters**

Fatty Acid trivial name / rational name)	Structure	Common Acronym	Methyl Ester (trivial name / rational name)
Palmitic acid / Hexadecanoic acid;	$R-(CH_2)_{14}-CH_3$	C16:0	Methyl Palmitate / Methyl Hexadecanoate
Stearic acid / Octadecanoic acid	$R-(CH_2)_{16}-CH_3$	C18:0	Methyl Stearate / Methyl Octadecanoate
Oleic Acid / 9(Z) Octadecanoic acid	$R-(CH_2)_7-CH=CH-(CH_2)_7-CH_3$	CH18:1	Methyl Oleate / Methyl 9(Z) - Octadecenoate
Linoleic acid / 9(Z),12(Z)-Octadecadienoic acid	$R-(CH_2)_7-CH=CH-CH_2-CH=CH-(CH_2)_4-CH_3$	CH18:2	Methyl linoleate / Methyl 9(Z),12(Z)-Octadecadienoate
inolenic acid / 9(Z),12(Z),15(Z)-octadecatrienoic acid;	$R-(CH_2)_7-(CH=CH-CH_2)_3-CH_3$	C18:3	Methyl linolenate / Methyl 9(Z),12(Z),15(Z)-octadecadienoate

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		Rev: 01
		February 2025

**Table 2. Characteristic of Common Fatty Acids and Their Methyl Esters**

Fatty Acid Methyl Ester	Formula	Molecular weight	Melting point
Palmitic acid	C16H32O2	256.423	63 – 64
Methyl Palmitate	C12H14O2	270.457	30.5
Stearic Acid	C18H36O2	284.481	70
Methyl Stearate	C19H38O2	298.511	39
Oleic Acid	C18H34O2	282.465	16
Methyl Oleate	C19H36O2	296.495	-20
Linoleic Acid	C18H32O2	280.450	-5
Methyl Linoleate	C19H34O2	294.479	-35
Linoleic Acid	C18H30O2	278.434	-11
Methyl Linolenate	C19H32O2	292.463	-52/ -57

### Biodiesel Production Techniques

The process for biodiesel production is known as transesterification which involves chemical reaction between oil (triglycerides) and short chain alcohol such as methanol and ethanol in the presence of a suitable catalyst to yield fatty acid alkyl esters i.e. biodiesel and glycerin as by-product. The most cursory look at the literature relating to biodiesel will soon reveal the following relationship for prediction of biodiesel from fats and oils.

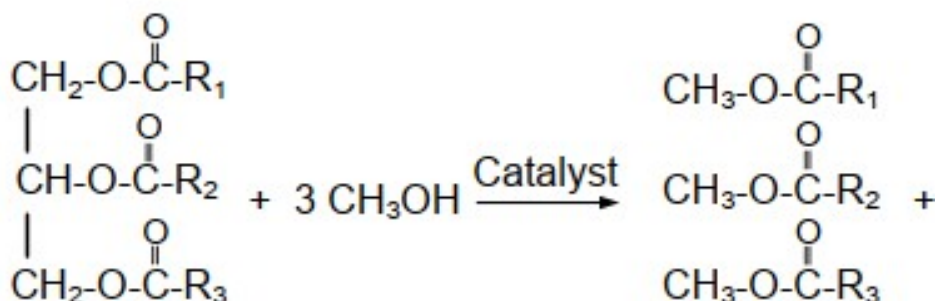
100 lbs of Oil + 10 lbs of methanol → 100 lbs of Biodiesel + 10 lbs of Glycerol

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	<b>BIODIESEL MANUFACTURING</b>	Rev: 01
	<b>ENGINEERING DESIGN GUIDELINES</b>	February 2025

Methanol is mostly used for the transesterification due to its low cost and advantageous physical and chemical properties. Moreover, catalysts can be dissolved faster in methanol than other alcohols to react easily with triglycerides. According to stoichiometry, three moles of methanol react with one mole triglyceride to produce fatty acid methyl ester (FAME) as shown in the following reaction.



where R<sub>1</sub>, R<sub>2</sub>, and R<sub>3</sub> are long chains of carbons and hydrogen atoms, sometimes called fatty acid chains. There are five types of chains that are common in soybean oil and animal fats (others are present in small amounts):

- |  |   |
|--|---|
| Palmitic: R = - (CH <sub>2</sub> ) <sub>14</sub> - CH <sub>3</sub>   | 16 carbons, (including the one that R is attached to.) (16:0) |
| Stearic: R = - (CH <sub>2</sub> ) <sub>16</sub> - CH <sub>3</sub>  | 18 carbons, 0 double bonds (18:0)                             |
| Oleic: R = - (CH <sub>2</sub> ) <sub>7</sub> CH=CH(CH <sub>2</sub> ) <sub>7</sub> CH <sub>3</sub>                                  | 18 carbons, 1 double bond (18:1)                              |
| Linoleic: R=-(CH <sub>2</sub> ) <sub>7</sub> CH=CH-CH <sub>2</sub> -CH=CH(CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub>           | 18 carbons, 2 double bonds (18:2)                             |
| Linolenic: R=-(CH <sub>2</sub> ) <sub>7</sub> CH=CH-CH <sub>2</sub> -CH=CH-CH <sub>2</sub> -CH=CH-CH <sub>2</sub> -CH <sub>3</sub> | 18 carbons, 3 double bonds (18:3)                             |

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	<b>BIODIESEL MANUFACTURING</b>	Rev: 01
	<b>ENGINEERING DESIGN GUIDELINES</b>	February 2025

**Table 3. Composition of Various Oils and Fats**

Oil or fat	14:0	16:0	18:0	18:1	18:2	18:3	20:0	22:1
Soybean	6-10	2-5	20-30	50-60	5-11			
Corn	1-2	8-12	2-5	19-49	34-62	trace		
Peanut	8-9	2-3	50-65	20-30				
Olive	9-10	2-3	73-84	10-12	trace			
Cottonseed	0-2	20-25	1-2	23-35	40-50	trace		
Hi linoleic Safflower	5.9	1.5	8.8	83.8				
Hi Oleic Safflower	4.8	1.4	74.1	19.7				
Hi Oleic Rapeseed	4.3	1.3	59.9	21.1	13.2			
Hi Erucic Rapeseed	3.0	0.8	13.1	14.1	9.7	7.4	50.7	
Butter	7-10	24-26	10-13	28-31	1-2.5	.2-.5		
Lard	1-2	28-30	12-18	40-50	7-13	0-1		
Tallow	3-6	24-32	20-25	37-43	2-3			
Linseed Oil	4-7	2-4	25-40	35-40	25-60			
Yellow grease (Typical)	2.43	23.24 16:1=3.79	12.96	44.32	6.97	0.67		

These chains are designated by two numbers separated by a colon. The first number designates the number of carbon atoms in the chain and the second number designates the number of double bonds. Note that the number of carbon atoms includes the carbon that is double bonded to the oxygen atom at one end of the fatty acid (called the carboxylic carbon). This is the end that the methanol attaches to when methyl esters are produced. Table 3 shows the percentages of each fatty acid chain present in common oils and fats.

Transesterification is the reaction of a fat or oil with an alcohol to form esters and glycerol. Alcohol combines with the triglycerides to form glycerol and esters. A catalyst is usually used to improve the reaction rate and yield. Transesterification consists of a sequence of three consecutive reversible reactions. The first step is the conversion of triglycerides to diglycerides, followed by the conversion of diglycerides to monoglycerides, and finally monoglycerides into glycerol, yielding one ester molecule from each glyceride at each step.

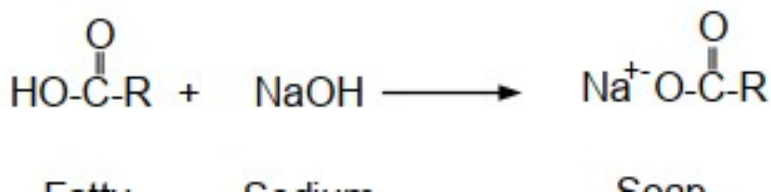
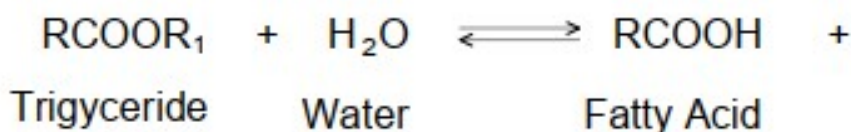
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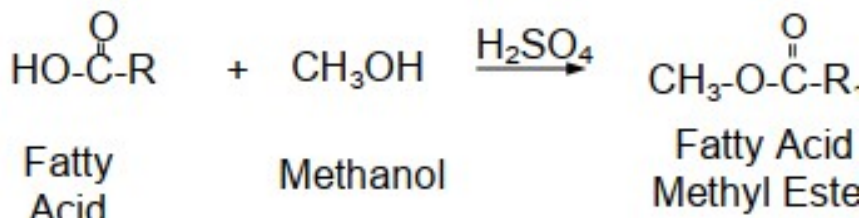
<b>KLM Technology Group</b>  Practical Engineering Guidelines for Processing Plant Solutions  www.klmtechgroup.com	<b>Kolmetz Handbook          of Process Equipment Design</b>	Page 15 of 85
	<b>BIODIESEL MANUFACTURING</b>	Rev: 01
	<b>ENGINEERING DESIGN GUIDELINES</b>	February 2025

Transesterification reactions are basically of three types: alkali-catalyzed, acid-catalyzed or enzyme-catalyzed. The third one is expensive and relatively slow compared to the first two. Sodium or Potassium hydroxide (KOH) are the commonly used alkali catalysts due to rapid reaction rate and high yields.

Water can originate from the oils and form during the saponification reaction and can hydrolyze the triglycerides to diglycerides resulting in the formation of Free Fatty Acid (FFA). The FFAs react with the catalyst to form soap and water. The reactions are given below:



Additional catalysts must be added to compensate for the catalyst lost to soap. The soap formed should be removed to meet the standard such as European Union standards for alternative diesel fuel which restricted minimum biodiesel purity to 96.5%. Removal of soap and purification of biodiesel can be done by washing with hot distilled water, citric, 5% phosphoric acid and with silica gel. When the FFA level is above 5%, an acid catalyst, typically sulfuric acid, is more suitable for esterification of FFAs to methyl esters, as shown in the following reaction:



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		Rev: 01
		February 2025

Acid catalyzed process is non-practical for biodiesel production commercially because of its slow reaction rate, high molar ratio of alcohol to oil condition, requirement of high temperature and problem related to separation of the catalyst.

The process of biodiesel production can be grouped as catalytic, non-catalytic, and enzymatic.

### 1. Base Catalysis

Base catalysis is the most popular method of biodiesel production. Vegetable oils consist of 98% triglycerides. The conversion reaction of vegetable oil produces biodiesel and glycerin. The reaction requires at least three moles of alcohol for every mole of vegetable oil to give three moles of biodiesel and one mole of glycerol.

Methanol is commonly used in this reaction due to its physical and chemical advantages. Also, its reaction with vegetable oil is rapid and it can be easily dissolved in the catalyst. An alkaline catalyst is used to enhance the reaction rate and yield. NaOH, KOH, and CH<sub>3</sub>ONa are the widely used catalysts. KOH is preferred more because of its low cost and high activity.

The transformation of waste cooking oil using NaOH was found to be 86%. Increasing the concentration of the catalyst increases the biodiesel yield up to 0.80% NaOH and then it starts to decrease. The alkaline catalyst concentration in the range of 0.5 – 1 % by weight yield 94-99% biodiesel. The conversion reaction takes place at 60-80°C temperature.

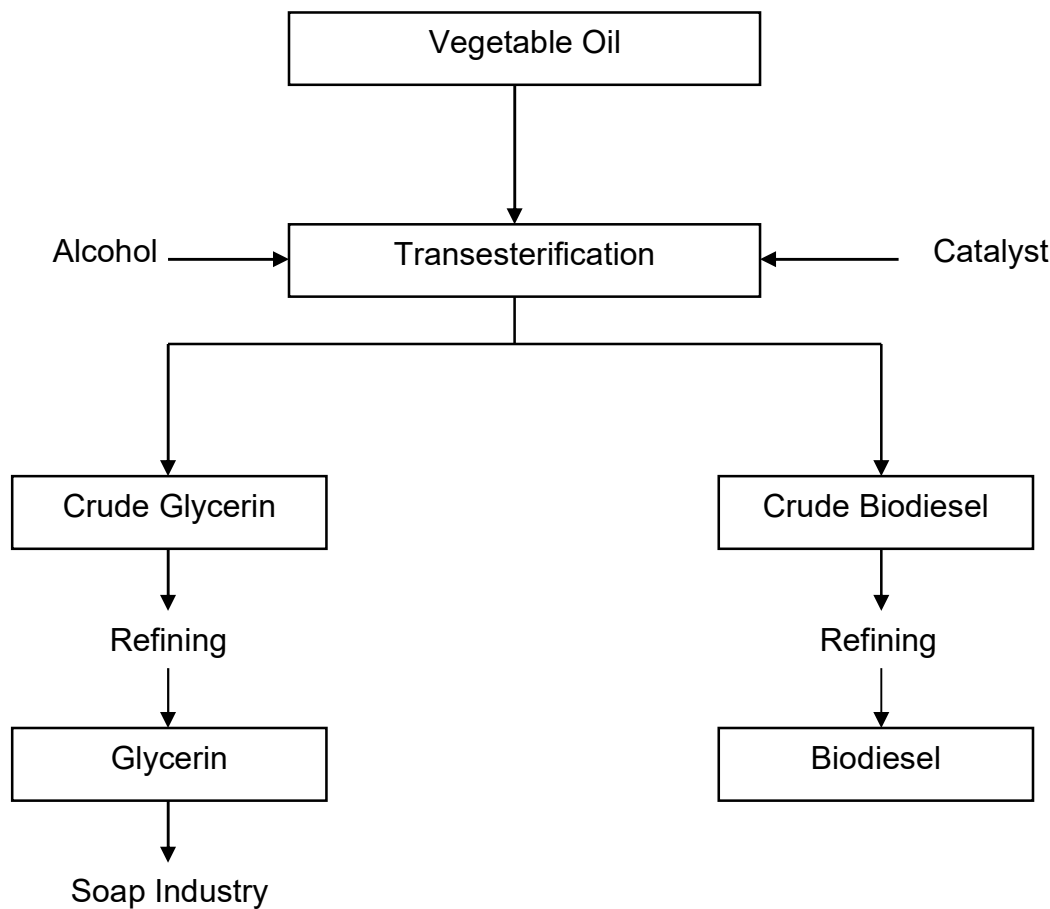
The stepwise procedure for biodiesel production is represented in Figure 2. It can be depicted that the transesterification of vegetable oil takes place in presence of a suitable catalyst. Biodiesel is obtained after proper refining. Glycerin is obtained as a by-product of the reaction and is used for soap manufacturing. Excess alcohol is recovered and re-used.

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	<b>BIODIESEL MANUFACTURING</b>	Rev: 01
	<b>ENGINEERING DESIGN GUIDELINES</b>	February 2025



**Figure 2. Process of the Biodiesel Production Process**

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		Rev: 01
		February 2025

## 2. Acid Catalysis

Acid catalysis is suitable for feedstock with exceptionally high fatty acid content. In this method, strong acid is used. Acid catalysts can be used for the triglycerides, but the reaction will become very slow. Acid catalysis can also be used for direct esterification of oils or for making esters from soap stock, which is a byproduct of edible oil refining.

Acid catalysis requires high alcohol to the fatty acid ratio (20:1 or 40:1 mol ratio) and a large amount of catalyst (5–25%)  $H_2SO_4$ ,  $HCl$ , and  $H_3PO_4$  are the most used catalysts. The acid-catalyzed transesterification is to be carried out in the absence of water, to keep away the formation of carboxylic acids which decrease the yields of biodiesel. Methanol is the most widely used alcohol in this process.

## 3. Enzymatic Conversion

The enzymes are utilized in solutions or immobilized onto the substance. The process is carried out at 35–35 C. In any case, the reaction is slow and takes 4–40 h for completion. Also, the cost of enzymes is too high. Biodiesel was produced from used oil using a biocatalyst of lipase enzyme immobilized magnetic nanocomposite and a maximum yield of 96% was achieved.

## 4. Non-Catalytic Conversion

Biodiesel production using a non-catalytic approach is a slow process because of the inferior miscibility of methanol and oil. Using a co-solvent that is soluble in both methanol and oil can enhance the rate of reaction. Tetrahydrofuran or methyl tert-butyl ether is used for this purpose. In the presence of a co-solvent, the conversion process is 95% complete in 10 min under ambient conditions.

In the second approach supercritical conditions are used for carrying out conversion reaction. The process entails extremely high temperature and pressure. Also, high alcohol to oil ratio (42:1 mol ratio) is required. The reaction is completed in 3 to 5 min. This method is very expensive.

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		Rev: 01
		February 2025

Base catalysis is preferred over other methods as it possesses certain advantages: the ability to complete the transesterification quickly, less reaction temperature and pressure, methanol requirement is less, and better conversion efficiency. Base catalysts give good results when the free fatty acid content of the source is less. The non-catalytic reaction takes place under supercritical conditions. Non-catalytic and enzymatic conversion is not economically viable options.

### 5. Other Catalyst Used for Transesterification

Apart from the mentioned category of catalyst, there are important types of catalyst which include nano catalyst and ionic liquid catalyst. Nanocatalyst involves the use of nanomaterial as the catalyst. Nanoscale catalysts have a high specific surface area and surface energy which results in high catalytic activity. Several researchers used nanocatalyst for biodiesel production:

Used heterogeneous Ni-doped ZnO nanocatalyst, used CaO nanocatalyst, used CaO/Ag nanocatalyst used MgO/MgSO<sub>4</sub> nanocatalyst. Ionic liquids are organic salts consisting of anions and cations that are liquid at room temperature. Their unique advantage is that while synthesized, they can be moderated to suit required reaction conditions.

A low-cost quaternary ammonium salt-glycerin-based ionic liquid as a solvent for extracting glycerin from the transesterified biodiesel. Among the different possible types of ionic liquids for catalysis of transesterification, ionic liquids composed of the 1-n-butyl-3 methylimidazolium cation are the most widely researched compounds.

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		Rev: 01
		February 2025

## Biodiesel Properties and Specifications

The best current measure for biodiesel quality is the ASTM standard, ASTM D 6751: Standard Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels. This standard specifies the properties required for a biodiesel fuel to be used in an engine without problems. Even in blends with conventional diesel fuel, most people in the industry expect that the biodiesel blending stock will meet the standard before being blended.

While some properties in the standard, such as cetane number and density, reflect the properties of the chemical compounds that make up biodiesel, other properties provide indications of the quality of the production process. Generally, the parameters given in the standard ASTM D6751 are defined by other ASTM standards.

However, other test methods, such as those developed for the American Oil Chemists' Society, (AOCS) may also be suitable (or even more suitable as they were developed for fats and oils and not for petroleum-derived materials addressed in the ASTM standards). The standard for biodiesel is ASTM 6751-02.

ASTM D 6751 – 02 sets forth the specifications that must be met for a fatty acid ester product to carry the designation “biodiesel fuel” or “B100” or for use in blends with any petroleum-derived diesel fuel defined by ASTM D 975, Grades 1-D, 2-D, and low sulfur 1-D and 2-D.

The values of the various biodiesel properties specified by ASTM D 6751 are listed in Table 4. Each of these properties and the test method used to measure it are described below.

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		Rev: 01
		February 2025

**Table 4. ASTM D 6751 – 02 Requirements**

Property	Method	Limits	Units
Flash Point, Closed Cup	D93	130 min	°C
Water and Sediment	D2709	0.050 max	%volume
Kinematic viscosity, 40°C	D 445	1.9 – 6.0	mm <sup>2</sup> /s
Sulfated Ash	D 874	0.020 max	wt.%
Total Sulfur	D 5453	0.05 max	wt.%
Copper strip corrosion	D 130	No. 3 max	
Cetane number	D 613	47 min	
Cloud point	D 2500	Report to customer	°C
Carbon residue	D 4530	0.050 max	wt.%
Acid Number	D 664	0.8 max	Mg KOH/g
Free Glycerin	D 6584	0.020	wt.%
Total Glycerin	D 6584	0.240	wt.%
Phosphorus	D 4951	0.0010	wt.%
Vacuum distillation end point	D 1160	360°C max, at 90% distilled	°C
Storage Stability	To be determined	To be determined	To be determined

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		Rev: 01
		February 2025

## Property Requirements and Specified Methods for B100

### 1. Flash Point

The flash point is defined as the “lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen to ignite under specified conditions of test.” This test, in part, is a measure of residual alcohol in the B100.

The flash point is a determinant for flammability classification of materials. The typical flash point of pure methyl esters is  $> 200^{\circ}\text{C}$ , classifying them as “non-flammable”. However, during production and purification of biodiesel, not all the methanol may be removed, making the fuel flammable and more dangerous to handle and store if the flash point falls below  $130^{\circ}\text{C}$ . Excess methanol in the fuel may also affect engine seals and elastomers and corrode metal components. Generally, a production Quality Control (QC) laboratory should include a flash point apparatus for quality control and as a means of detecting excess alcohol levels.

### 2. Water and Sediment

Water and sediment is a test that determines the volume of free water and sediment in middle distillate fuels having viscosities at  $40^{\circ}\text{C}$  in the range 1.0 to  $4.1\text{ mm}^2/\text{s}$  and densities in the range of 700 to  $900\text{ kg}/\text{m}^3$ .

This test is a measure of cleanliness of the fuel. For B100 it is particularly important because water can react with the esters, making free fatty acids, and can support microbial growth in storage tanks. Water is usually kept out of the production process by removing it from the feedstocks.

However, some water may be formed during the process by the reaction of the sodium or potassium hydroxide catalyst with alcohol. If free fatty acids are present, water will be formed when they react to either biodiesel or soap. Finally, water is deliberately added during the washing process to remove contaminants from the biodiesel. This washing process should be followed by a drying process to ensure the final product will meet ASTM D 2709.

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		Rev: 01
		February 2025

Sediments may plug fuel filters and may contribute to the formation of deposits on fuel injectors and other engine damage. Sediment levels in biodiesel may increase over time as the fuel degrades during extended storage. The production QC lab should be equipped to perform this test on a routine basis.

### 3. Kinematic Viscosity

Kinematic viscosity: the resistance to flow of a fluid under gravity. The kinematic viscosity is a basic design specification for the fuel injectors used in diesel engines. Too high a viscosity, and the injectors do not perform properly.

The viscosity of biodiesel can be predicted  $\pm 15\%$  using the esters composition determined using ASTM D 6584. The viscosity apparatus to run D 445 is not critical to the QC lab, but it is valuable as a quick assay method for estimating the degree of completion for a reaction batch.

### 4. Sulfated Ash

Sulfated ash is the residue remaining after a sample has been carbonized, and the residue subsequently treated with sulfuric acid and heated to a constant weight. This test monitors the mineral ash residual when a fuel is burned.

For biodiesel, this test is an important indicator of the quantity of residual metals in the fuel that came from the catalyst used in the esterification process. Producers that use a base catalyzed process may wish to run this test regularly. Many of these spent sodium or potassium salts have low melting temperatures and may cause engine damage in combustion chambers.

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		Rev: 01
		February 2025

## 5. Total sulfur

This method covers the determination of total sulfur in liquid hydrocarbons, boiling in the range from approximately 25 to 400°C, with viscosities between approximately 0.2 and 20 cSt (mm<sup>2</sup>/s) at room temperature.

Biodiesel feedstocks typically have very little sulfur, but this test is an indicator of contamination of protein material and/or carryover catalyst material or neutralization material from the production process. Producers using rendered or waste feedstocks should have access to this measurement for their feedstocks. Some biodiesels from rendered fats and greases have been found to have sulfur levels of 40-50 ppm. The sulfur limits for on-highway diesel fuel will be reduced to 0.0015% (15 ppm) in 2006, so producers that do not meet this future specification on a regular basis may need to consider sulfur removal technology in their facility.

## 6. Copper Strip Corrosion

The copper strip corrosion is used for the detection of the corrosiveness to copper of fuels and solvents.” This test monitors the presence of acids in the fuel.

For B 100, the most likely source of a test failure would be excessive free fatty acids, which are determined in accordance with an additional specification. The producer may choose to run this test periodically, but the acid number (D 664) determination is the more important acid content QC measurement.

## 7. Cetane Number

The cetane number is “a measure of the ignition performance of a diesel fuel obtained by comparing it to reference fuels in a standardized engine test.” Cetane for diesel engines is analogous to the octane rating in a spark ignition engine – it is a measure of how easily the fuel will ignite in the engine.

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		Rev: 01
		February 2025

For B100, the cetane number is seldom an issue because all of the common fatty acid esters have cetane numbers near or above 47. The cetane number can be predicted  $\pm 10\%$  using the esters composition. It is unlikely that an individual producer will ever run cetane tests on-site because the equipment is extremely expensive.

### 8. Cloud Point

The cloud point is “The temperature at which a cloud of wax crystals first appears in a liquid when it is cooled down under conditions prescribed in this test method.” The cloud point is critical factor in cold weather performance for all diesel fuels.

The chemical composition of some biodiesel feedstocks leads to a B100 that may have higher cloud points than customers desire. The cloud point, however, is another parameter that can be predicted  $\pm 5\%$  with knowledge of the esters composition, but producers are advised to be equipped to perform this test. Since the saturated methyl esters are the first to precipitate, the amounts of these esters, methyl palmitate and methyl stearate, are the determining factors for the cloud point.

### 9. Carbon Residue

In petroleum products, the part remaining after a sample has been subjected to thermal decomposition is the carbon residue. The carbon residue is a measure of how much residual carbon remains after combustion. The test basically involves heating the fuel to a high temperature absence of oxygen. Most of the fuel will vaporize and be driven off, but a portion may decompose and pyrolyze to hard carbonaceous deposits. This is particularly important in diesel engines because of the possibility of carbon residues clogging the fuel injectors.

The most common cause of excess carbon residues in B100 is an excessive level of total glycerin, total glycerin is also measured directly using ASTM D 6584, so this measurement is generally not critical to the producer.

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		Rev: 01
		February 2025

## 10. Acid Number

The acid number is the quantity of base expressed as milligrams of potassium hydroxide per grams of sample, required to titrate a sample to a specified end point. The acid number is a direct measure of the free fatty acids can lead to corrosion and may be a symptom of the water in the fuel. Usually, for a base catalyzed process, the acid value after production will be low since the base catalyst will strip the available free fatty acid. However, the free fatty acid value may increase with time as the fuel degrades due to contact with air or water. This test should be performed regularly as a part of the producer QC program.

## 11. Free Glycerin

Free glycerol is the glycerol present as molecular glycerol in the fuel. Free glycerol results from incomplete separation of the ester and glycerol products after the transesterification reaction. This can be a result of imperfect water washing or other approaches that do not effectively separate the glycerol from the biodiesel. The free glycerol can be a source of carbon deposits in the engine because of incomplete combustion.

## 12. The Total Glycerin

Total glycerol “is the sum of free and bonded glycerol.” Bonded glycerol “is the glycerol portion of the mono-, di-, and triglyceride molecules.” Elevated total glycerol values are indicators of incomplete esterification reactions and predictors of excessive carbon deposits in the engine.

## 13. Phosphorus

This test covers the quantitative determination of barium, calcium, copper, magnesium, phosphorus, sulfur, and zinc in unused lubricating oils and additive packages.” In the case of B100, phosphorus can come from incomplete refining of the phospholipids (or gums) from the vegetable oil and from bone and proteins encountered in the rendering process. The producer should have access to this method for periodic measurements.

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		Rev: 01
		February 2025

#### 14. Vacuum Distillation End Point

The vacuum distillation end-point test “covers the determination, at reduced pressures, of the range of boiling points for petroleum products that can be partially or completely vaporized at a maximum liquid temperature of 400° C.”

Petroleum fractions have tens to hundreds of individual compounds mixed together. The distillation curves are used to characterize the broad chemistry of a given crude oil source in terms of the boiling temperatures of its constituent compounds.

In B100 there are, at most, ten different esters present, and they can be identified using gas or liquid chromatography. The same chromatograph that determines free and total glycerin can determine the esters composition in the B100. This composition allows calculation of the T-90 point for the fuel, without having to perform the testing for every batch of product. It is unlikely that the producer would have a reason to run this test except to certify compliance with the ASTM standard.

#### 15. Storage Stability

All fuels are subject to degradation over time when they are stored. This degradation may be due to microbial action, water intrusion, air oxidation, etc. The standard and the test methods for determining storage stability for B100 are still in the development stage within the ASTM process. In general, the following parameters will change and can be used to determine if the fuel should not be used: acid number, water and sediment, and viscosity. Should an aged fuel fail any of these three standards, it should not be used.

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		Rev: 01
		February 2025

## DEFINITION

**Biodiesel** – Refers to the fuel commonly called an ester, or fatty acid methyl esters (FAME) produced from renewable fats, oils, and/or greases that meets the most current ASTM International Standard D6751.

**Biocatalyst** - A type of catalyst that offers regio- and stereo-selectivity in chemical processing, often serving as a more environmentally.

**Bonded glycerol** – Is the glycerol portion of the mono-, di-, and triglyceride molecules

**Catalyst** – a substance that increase the yield of biodiesel while simultaneously boosting the rate of reaction.

**Cetane number** – A measure of the ignition performance of a diesel fuel obtained by comparing it to reference fuels in a standardized engine test.

**Cloud point** – The temperature at which a cloud of wax crystals first appears in a liquid when it is cooled down under conditions prescribed in this test method

**Density** – The mass per unit volume of a substance at a given temperature

**Dynamic viscosity** – Ratio between applied shear stress and rate of shear of a liquid

**Free glycerol** – The glycerol present as molecular glycerol in the fuel

**Fatty Acid** – a carboxylic acid with an aliphatic chain, which is either saturated or unsaturated.

**Free Fatty Acid** – A carboxylic acid with an aliphatic chain, which is either saturated or unsaturated.

**Flash point** - lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen to ignite under specified conditions of test.

These design guidelines are believed to be as accurate as possible, but are very general and not for specific design cases. They were designed for engineers to do preliminary designs and process specification sheets. The final design must always be guaranteed for the service selected by the manufacturing vendor, but these guidelines will greatly reduce the amount of up front engineering hours that are required to develop the final design. The guidelines are a training tool for young engineers or a resource for engineers with experience.

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		Rev: 01
		February 2025

**Hazard** – Anything with the potential for producing an accident.

**Nanocatalyst** - The use of nanomaterial as the catalyst.

**Refining** – A Process to remove the phospholipids and free fatty acids from the crude oil.

**Risk** – The probability of a hazard resulting in an accident.

**Safety** – The prevention of accidents by the use of appropriate technologies to identify the hazards of a chemical plant and to eliminate them before the accident occurs.

**Total glycerol** – The sum of free and bonded glycerol.

**Transesterification** – the reaction of a fat or oil with an alcohol to form esters and glycerol

**Triglyceride** – An ester derived from glycerol and three fatty acids.

**Water and sediment** – A test that determines the volume of free water and sediment in middle distillate fuels having viscosities at 40 °C in the range 1.0 to 4.1 mm<sup>2</sup>/s and densities in the range of 700 to 900 kg/m<sup>3</sup>.

ASTM – American Standard Testing and Materials

CSTR – Continuous Stirrer Tank Reactors

FAME - Fatty Acid Methyl Ester

FFA – Free Fatty Acid

LHV – Low Heating Value

PFR – Plug Flow Reactor

WFO – Waste Frying Oil

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		Rev: 01
		February 2025

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