LECTUTE-13
(BIODIESEL)

➢ INTRODUCTION

- Biodiesel is a liquid biofuel obtained by chemical processes from vegetable oils or animal fats and an alcohol that can be used in diesel engines, alone or blended with diesel oil.
- ASTM (American Society for Testing and Materials) defines biodiesel as a mixture of long-chain monoalkylic esters from fatty acids obtained from renewable resources, to be used in diesel engines.
- Blends with diesel fuel are indicated as ‘‘Bx’’, where ‘‘x’’ is the percentage of biodiesel in the blend.
- For instance, ‘‘B5’’ indicates a blend with 5% biodiesel and 95% diesel fuel; in consequence, B100 indicates pure biodiesel.
- Biodiesel is an alternative energy source and could be a substitute for petroleum-based diesel fuel.
- The biggest challenge modern industrial society is facing today is the decline and exhaustion of the fossil energy resources.
- The primary sources of energy that power our civilization are those fossil fuels.
- The International Energy Agency has forecasted that the world's total energy requirements will rise by half in the next 25 years.
- On the other hand, global oil product demand has been revised to 84.3 million barrels a day (mb/d) in 2006 and 85.8 mb/d in 2007
- Therefore continued use of petroleum-sourced fuels is now widely recognized as unsustainable because of depleting supplies and increasing demand.
- Oil prices have been rising steadily over the past 3 years and surged to a record high above $60 a barrel in June 2005, sustaining a rally built on strong demand for gasoline and diesel and on concerns about refiners' ability to keep up.
- In addition, the combustion of the fossil fuels used is considered as the major factor responsible for global warming due to large-scale carbon dioxide emissions.
- One of the most prominent alternative energy resources, attracting more and more interest in recent years with the price for crude oil reaching record heights, is **biodiesel**, which is a possible substitute for petroleum-based diesel fuel.
- Many studies have shown that the properties of biodiesel are very close to diesel fuel.
- Therefore, biodiesel fuel can be used in diesel engines with little or no modification.
- Biodiesel has a higher cetane number than diesel fuel, no aromatics, no sulfur, and contains 10-11% oxygen by weight.
- These characteristics of biodiesel are responsible for a reduction in the emissions of carbon monoxide (CO), hydrocarbon (HC) and particulate matter (PM) in the exhaust gas compared to diesel fuel.
- *However, to be a viable alternative, a biodiesel should provide a net energy gain, be economically competitive, and be producible in large quantities without reducing food supplies.*
- Biodiesel cylcle has been reflected in the **Figure-2** and a biodiesel plant in **Figure-3**.

- Figure-1 showing vegetable oil and animal fats being converted to biodiesel.

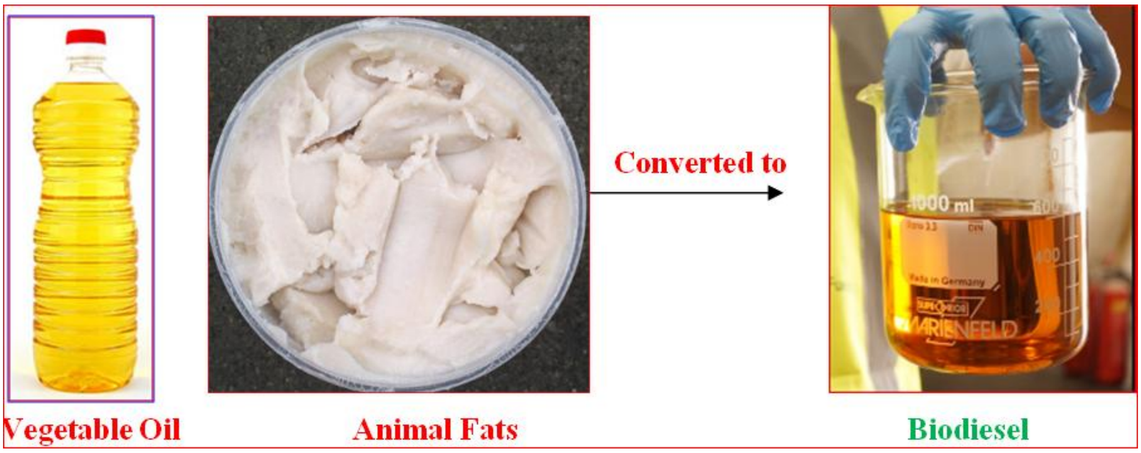
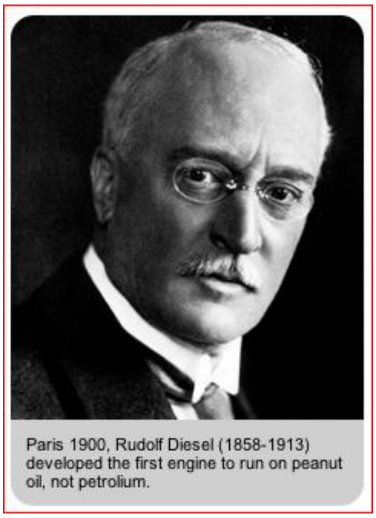
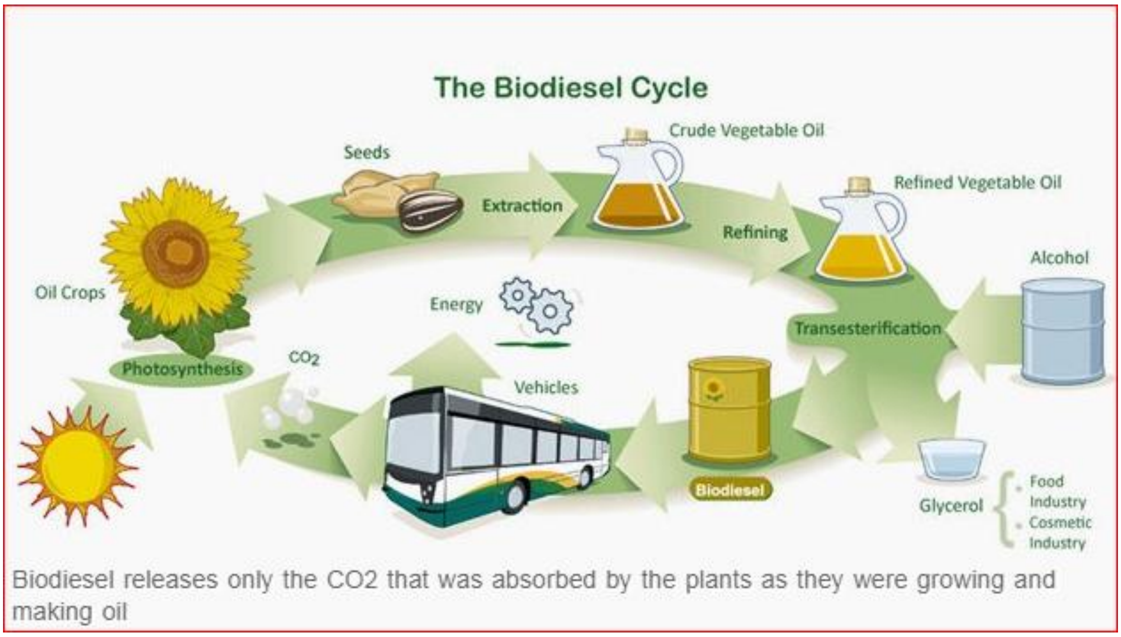


Figure-1: Vegetable oil and animal fats to biofuel (biodiesel).

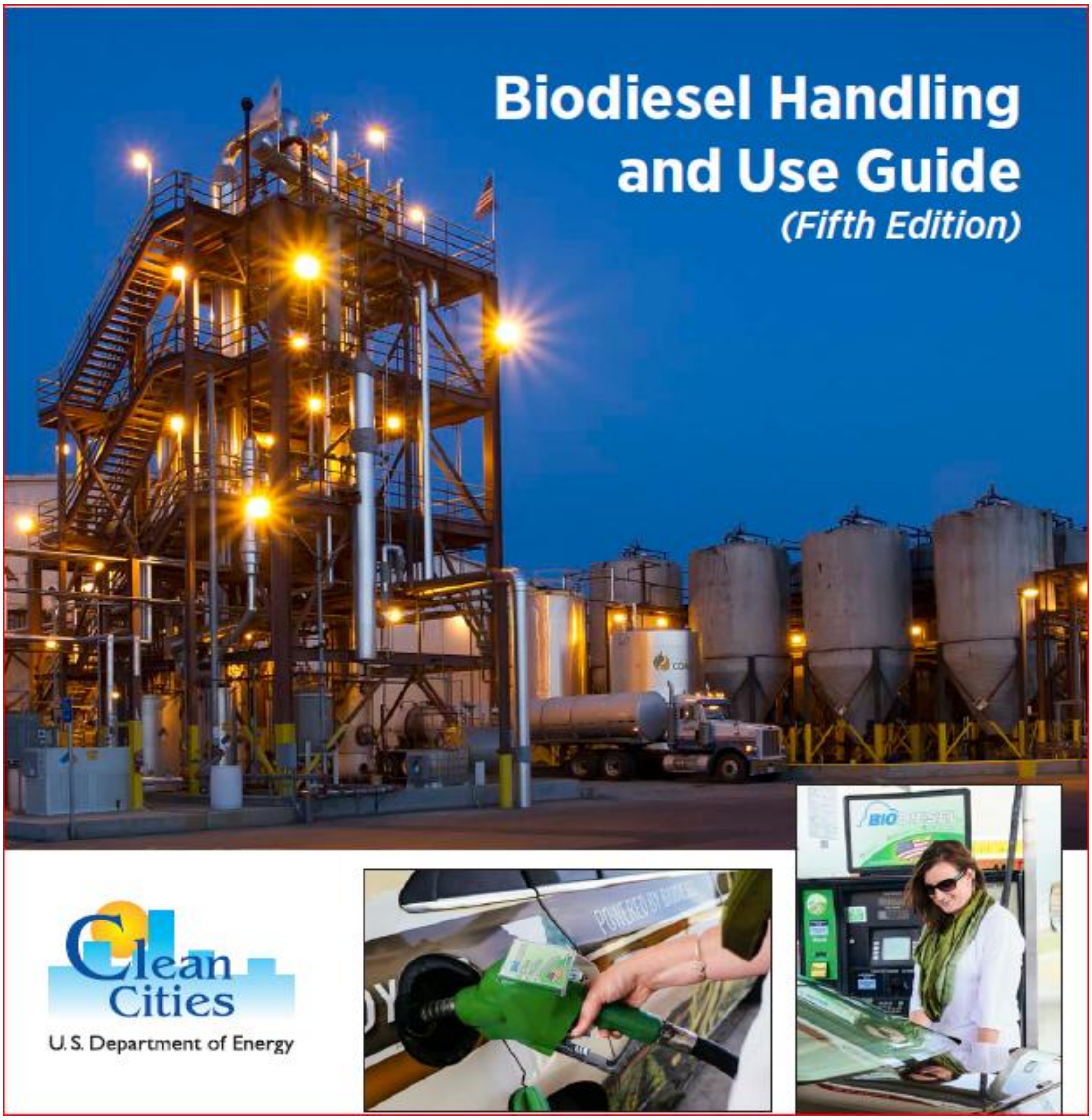
- **History**
  - ✓ Before petroleum diesel fuel became popular, Rudolf Diesel (**Rudolf Christian Karl Diesel**, a German mechanical engineer, 18 March 1858 – 29 September 1913) the inventor of the diesel engine in 1897, experimented with using vegetable oil (biodiesel) as fuel.
  - ✓ Until 2001, the United States consumed only small amounts of biodiesel.
  - ✓ Since then, U.S. biodiesel production and consumption have increased substantially, largely because of the availability of various government incentives and requirements to produce, sell, and use biodiesel.


Paris 1900, Rudolf Diesel (1858-1913) developed the first engine to run on peanut oil, not petrolium.



Biodiesel releases only the CO2 that was absorbed by the plants as they were growing and making oil

Figure-2: The biodiesel cycle.



Biodiesel Handling and Use Guide

(Fifth Edition)

Clean Cities

U.S. Department of Energy

Figure-3: Biodiesel production plant in the United States

➢ ADVANTAGES AND DISADVANTAGES OF USING BIODIESEL

Advantages and disadvantages of using biodiesel are summarized below:

- **Advantages of the Use of Biodiesel**

  Some of the advantages of using biodiesel as a replacement for diesel fuel are:

  - ✓ Renewable fuel, obtained from vegetable oils or animal fats.
  - ✓ Low toxicity, in comparison with diesel fuel.
  - ✓ Degrades more rapidly than diesel fuel, minimizing the environmental consequences of biofuel spills.
  - ✓ Lower emissions of contaminants: carbon monoxide, particulate matter, polycyclic aromatic hydrocarbons, aldehydes.
  - ✓ Lower health risk, due to reduced emissions of carcinogenic substances.
  - ✓ No sulfur dioxide (SO_2) emissions.
  - ✓ Higher flash point (10^0C minimum).
  - ✓ May be blended with diesel fuel at any proportion; both fuels may be mixed during the fuel supply to vehicles.
  - ✓ Excellent properties as a lubricant.
  - ✓ It is the only alternative fuel that can be used in a conventional diesel engine, without modifications.

  - ✓ Used cooking oils and fat residues from meat processing may be used as raw materials.

- **Disadvantages of the Use of Biodiesel**

  There are certain disadvantages of using biodiesel as a replacement for diesel fuel that must be taken into consideration:

  - ✓ Slightly higher fuel consumption due to the lower calorific value of biodiesel.
  - ✓ Slightly higher nitrous oxide (NOx) emissions than diesel fuel.
  - ✓ Higher freezing point than diesel fuel. This may be inconvenient in cold climates.
  - ✓ It is less stable than diesel fuel, and therefore long-term storage (more than six months) of biodiesel is not recommended.
  - ✓ May degrade plastic and natural rubber gaskets and hoses when used in pure form, in which case replacement with Teflon components is recommended.
  - ✓ It dissolves the deposits of sediments and other contaminants from diesel fuel in storage tanks and fuel lines, which then are flushed away by the biofuel into the engine, where they can cause problems in the valves and injection systems.
  - ✓ In consequence, the cleaning of tanks prior to filling with biodiesel is recommended.
  - ✓ It must be noted that these disadvantages are significantly reduced when biodiesel is used in blends with diesel fuel.

➢ RAW MATERIALS FOR BIODIESEL PRODUCTION

- The raw materials for biodiesel production are vegetable oils, animal fats and short chain alcohols.
- The oils most used for worldwide biodiesel production are rapeseed (mainly in the European Union countries), soybean (Argentina and the United States of America), palm (Asian and Central American countries), sunflower, peanut, linseed, vegetable oils, and also animal fats.
- Methanol is the most frequently used alcohol although ethanol can also be used.
- Since cost is the main concern in biodiesel production and trading (mainly due to oil prices), the use of non-edible vegetable oils is recommended.
- Besides its lower cost, another undeniable advantage of non-edible oils for biodiesel production lies in the fact that no foodstuffs are spent to produce fuel.
- These and other reasons have led to medium- and large-scale biodiesel production trials in several countries, using non-edible oils such as castor oil, tung, cotton, jojoba and jatropha.
- Animal fats are also an interesting option, especially in countries with plenty of livestock resources, although it is necessary to carry out preliminary treatment since they are solid; furthermore, highly acidic grease from cattle, pork, poultry, and fish can be used.
- Microalgae appear to be a very important alternative for future biodiesel production due to their very high oil yield; however, it must be taken into account that only some species are useful for biofuel production.
- Although the properties of oils and fats used as raw materials may differ, the properties of biodiesel must be the same, complying with the requirements set by international standards.

- **Typical Oil Crops Useful for Biodiesel Production**

  The main characteristics of typical oil crops that have been found useful for biodiesel production are summarized in the following paragraphs:

  ✓ **Rapeseed and Canola**

    - Rapeseed adapts well to low fertility soils, but with high sulfur content.
    - With a high oil yield (40–50%), it may be grown as a winter-cover crop, allows double cultivation and crop rotation.
    - It is the most important raw material for biodiesel production in the European Community.
    - However, there were technological limitations for sowing and harvesting in some Central and South American countries, mainly due to the lack of adequate information about fertilization, seed handling, and storage (the seeds are very small and require specialized agricultural machinery).
    - Moreover, low prices in comparison to wheat (its main competitor for crop rotation) and low production per unit area have limited its use.
    - Rapeseed flour has high nutritional value, in comparison to soybean; it is used as a protein supplement in cattle rations.

Rapeseed plants　Rapeseed Plants and Oil　Rapeseeds

Figuire-4: Representation of rapeseed plants and seed.

    - Sometimes canola and rapeseed are considered to be synonymous; canola (Canadian oil low acid) is the result of the genetic modification of rapeseed in the past 40 years, in Canada, to reduce the content of erucic acid and glucosinolates in rapeseed oil, which causes inconvenience when used in animal and human consumption.
    - Canola oil is highly appreciated due to its high quality, and with olive oil, it is considered as one of the best for cooking as it helps to reduce blood cholesterol levels.

Calonma Plant with Flowers　Calona Seed

Figuire-5: Representation of calona plants with flower and calona seed.

- **Soybean**
  - It is a legume originating in East Asia.
  - Depending on environmental conditions and genetic varieties, the plants show wide variations in height.
  - Leading soybean producing countries are the United States, Brazil, Argentina, China, and India.
  - Biodiesel production form soybean yields other valuable sub-products in addition to glycerin: soybean meal and pellets (used as food for livestock) and flour (which have a high content of lecithin, a protein).
  - Grain yield varies between 2,000 and 4,000 kg/hectare.
  - Since the seeds are very rich in protein, oil content is around 18%.

Figuire-6: Representation of soybean plants, fruit and seed.

- **Oil Palm**
  - Oil palm is a tropical plant that reaches a height of 20–25 m with a life cycle of about 25 years.
  - Full production is reached 8 years after planting.
  - Two kinds of oil are obtained from the fruit: palm oil proper, from the pulp, and palm kernel oil, from the nut of the fruit (after oil extraction, palm kernel cake is used as livestock food).
  - Several high oil-yield varieties have been developed.
  - Indonesia and Malaysia are the leading producers.
  - International demand for palm oil has increased steadily during the past years, the oil being used for cooking, and as a raw material for margarine production and as an additive for butter and bakery products.
  - It is important to remark that pure palm oil is semisolid at room temperature (20–22 ^{0}C), and in many applications is mixed with other vegetable oils, sometimes partially hydrogenated.

Figuire-7: Representation of tree, fruit, shelland flesh.

- ✓ **Sunflower**
    - Sunflower ‘‘seeds’’ are really a fruit, the inedible wall (husk) surrounding the seed that is in the kernel.
    - The great importance of sunflower lies in the excellent quality of the edible oil extracted from its seeds.
    - It is highly regarded from the point of view of nutritional quality, taste and flavor.
    - Moreover, after oil extraction, the remaining cake is used as a livestock feed.
    - It must be noted that sunflower oil has a very low content of linoleic acid, and therefore it may be stored for long periods.
    - Sunflower adapts well to adverse environmental conditions and does not require specialized agricultural equipment and can be used for crop rotation with soybean and corn.
    - Oil yield of current hybrids is in the range 48–52%.

Sunflower Plants Sunflower Field Sunfloer with Seeds Sunflower Seeds

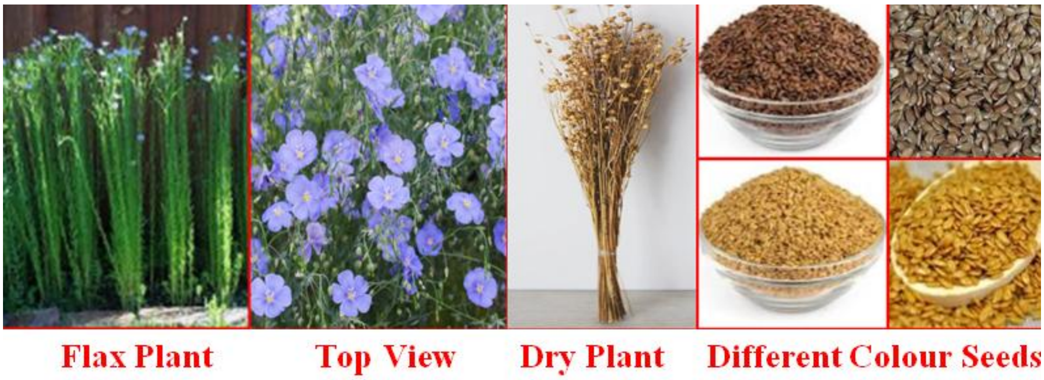
Figuire-8: Representation of sunflower plant, field and seed.

- ✓ **Peanut**
    - The quality of peanut is strongly affected by weather conditions during the harvest.
    - Peanuts are mainly used for human consumption, in the manufacture of peanut butter, and as an ingredient for confectionery and other processed foods.
    - Peanuts of lower quality (including the rejects from the confectionery industry) are used for oil production, which has a steady demand in the international market.
    - Peanut oil is used in blends for cooking and as a flavoring agent in the confectionery industry.
    - The flour left over, following oil extraction, is of high quality with high protein content; in pellet form, it is used as a livestock feed.

Peanut Field Individual Plant Peanut Plants with Root Raw Peanut

Figuire-9: Representation of peanut field, individual plant and raw peanut.

- ✓ **Flax**
    - Flax is a plant of temperate climates, with blue flowers.
    - Linen is made with the threads from the stem of the plant and the oil from the seeds is called linseed oil, used in paint manufacture.
    - Flax seeds have nutritional value for human consumption since they are a source of polyunsaturated fatty acids necessary for human health.
    - Moreover, the cake left over, following oil extraction, is used as a livestock feed.
    - The plant adapts well to a wide range of temperature and humidity; however, high temperatures and plentiful rain do not favor high yields of seed and fiber.
    - Flax seeds contain between 30 and 48% of oil, and protein content is between 20 and 30%.
    - It is important to remark that linseed oil is rich in polyunsaturated fatty acids, linolenic acid being from 40 to 68% of the total.



Flax Plant Top View Dry Plant Different Colour Seeds

Figuire-10: Representation of flax plant, top view, dry plants and seed.

- ✓ **Safflower**
    - Safflower adapts well to dry environments.
    - Although the grain yield per hectare is low, the oil content of the seed is high, from 30 to 40%.
    - Therefore, it has economic potential for arid regions.
    - Currently, safflower is used in oil and flour production and as bird feed.
    - There are two varieties, one rich in mono-unsaturated fatty acids (oleic acid) and the other with a high percentage of polyunsaturated fatty acids (linoleic acid).
    - Both varieties have a low content of saturated fatty acids.
    - The oil from safflower is of high quality and low in cholesterol content.
    - Other than being used for human consumption, it is used in the manufacture of paints and other coating compounds, lacquers and soaps.
    - It is important to note that safflower oil is extracted by means of hydraulic presses, without the use of solvents, and refined by conventional methods, without anti-oxidant additives.
    - The flour from safflower is rich in fiber and contains about 24% proteins.
    - It is used as a protein supplement for livestock feed.

Safflower Orange Safflower White Safflower Seed

Figuire-11: Representation of safflower ornge-white and sed.

- ✓ **Castor Seed**
  - ○ The castor oil plant grows in tropical climates, with temperatures in the range 20–30 ^{0}C; it cannot endure frost.
  - ○ It is important to note that once the seeds start germinating, the temperature must not fall below 12 ^{0}C.
  - ○ The plant needs a warm and humid period in its vegetative phase and a dry season for ripening and harvesting.
  - ○ It requires plenty of sunlight and adapts well to several varieties of soils.
  - ○ The total rainfall during the growth cycle must be in the range 700–1,400 mm; although it is resistant to drought, the castor oil plant needs at least 5 months of rain during the year.
  - ○ Castor oil is a triglyceride, ricinolenic acid being the main constituent (about 90%).
  - ○ The oil is non-edible and toxic owing to the presence of 1–5% of ricin, a toxic protein that can be removed by cold pressing and filtering.
  - ○ The presence of hydroxyl groups in its molecules makes it unusually polar as compared to other vegetable oils.

Castor Plants Castor Plant with Fruit Castor Seeds

Figuire-12: Representation of castor plants, fruit and seed.

- ✓ **Tung**
  - ○ Tung is a tree that adapts well to tropical and sub-tropical climates.
  - ○ The optimum temperature for tung is between 18 and 26 ^{0}C, with low yearly rainfall.
  - ○ During the harvest season, the dry nuts fall off from the tung tree and are collected from the ground.
  - ○ Nut production starts 3 years after the planting.
  - ○ The oil from tung nuts is non-edible and used in the manufacture of paints and varnishes, especially for marine use.

Tung Plants Blossom Tung Plant Plant with Fruit Tung Plant Seed

Figuire-13: Representation of yung plants, fruit and seed.

- ✓ **Cotton**
  - Among non-foodstuffs, cotton is the most widely traded commodity.
  - It is produced in more than 80 countries and distributed worldwide.
  - After the harvest, it may be traded as raw cotton, fiber or seeds.
  - In cotton mills, fiber and seeds are separated from raw cotton.
  - Cotton fiber is processed to produce fabric and thread, for use in the textile industry.
  - In addition, cotton oil and flour are obtained from the seed; the latter is rich in protein and is used in livestock feed and after further processing, for human consumption.

Cotton Plant Plant with Edible Seed Fieild-Ripe Cotton Cotton Seed

Figuire-14: Representation of castor plants, fruit and seed.

- ✓ **Jojoba**
  - Although jojoba can survive extreme drought, it requires irrigation to achieve an economically viable yield.
  - Jojoba needs a warm climate, but a cold spell is necessary for the flowers to mature.
  - Rainfall must be very low during the harvest season (summer).
  - The plant reaches its full productivity 10 years after planting.
  - The oil from jojoba is mainly used in the cosmetics industry; therefore, its market is quickly saturated.

Jojoba Plant Plant-Fruit Seed

Figuire-15: Representation of castor plants, fruit and seed.

- ✓ **Jatropha**
  - Jatropha is a shrub that adapts well to arid environments.

- Jatropha curcas is the most known variety; it requires little water or additional care; therefore, it is adequate for warm regions with little fertility.
- Productivity may be reduced by irregular rainfall or strong winds during the flowering season.
- Yield depends on climate, soil, rainfall and treatment during sowing and harvesting.
- Jatropha plants become productive after 3 or 4 years, and their lifespan is about 50 years.
- Oil yield depends on the method of extraction; it is 28–32% using presses and up to 52% by solvent extraction.
- Since the seeds are toxic, jatropha oil is nonedible.
- The toxicity is due to the presence of curcasin (a globulin) and jatrophic acid (as toxic as ricin).

Jtropha Tree **Jatropha Fruit** **Jatropha Seeds**

Figure-16: Representation of castor plants, fruit and seed.

✓ **Avocado**

- Avocado is a tree between 5 and 15 m in height.
- The weight of the fruit is between 120 and 2.5 kg and the harvesting period varies from 5 to 15 months.
- The avocado fruit matures after picking and not on the tree.
- Oil may be obtained from the fruit pulp and pit.
- It has a high nutritional value, since it contains essential fatty acids, minerals, protein and vitamins A, B6, C, D, and E.
- The content of saturated fatty acids in the pulp of the fruit and in the oil is low; on the contrary, it is very high in mono-unsaturated fatty acids (about 96% being oleic acid).
- The oil content of the fruit is in the range of 12–30%.

Avocado Tree with Fruits **Fruit** **Fruit Composition**

Figuire-17: Representation of avocado plants, fruit and seed.

Figuire-18: Representation of avocado oil extraction.

- ✓ **Microalgae**
  - ○ Microalgae have great potential for biodiesel production, since the oil yield (in liters per hectare) could be one to two orders of magnitude higher than that of other raw materials.
  - ○ Oil content is usually from 20 to 50%, although in some species it can be higher than 70%.
  - ○ However, it is important to note that not all microalgae are adequate for biodiesel production.
  - ○ High levels of CO_2, water, light, nutrients and mineral salts are necessary for the growth of microalgae.
  - ○ Production processes take place in raceway ponds and photobiological reactors.

Microalgae Production of Biodiesel from Fatty Oil of Microalgae

Figuire-19: Representation of algae and production of diesel.

- **Yield of Oil Crops**
  - ✓ The following Figure- presents approximate oil-yield values (ton oil per hectare) for some crops.
  - ✓ It is important to note that the data in Figure only show the oil yields of different crops.
  - ✓ However, for the comparison of economical suitability it must be borne in mind that in addition to oil, some crops are grown for fiber or protein production.

✓ For instance, soybean has an oil content of 18% (maximum), whereas the remainder is mostly protein (usually used as livestock feed).

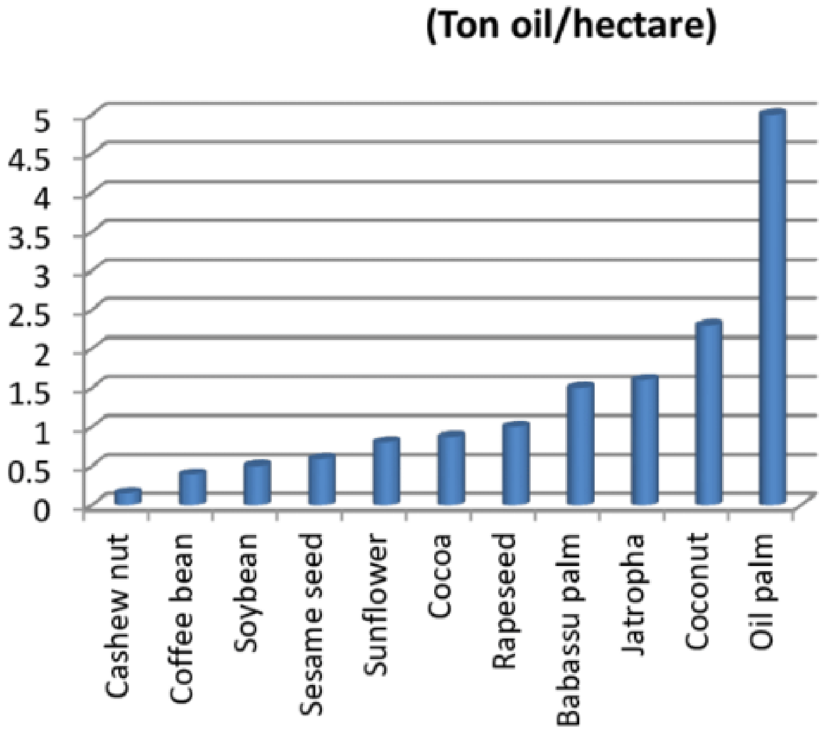


Figure-20: Approximate oil yields for different crops as reported by Oil World 2013.

- **Biodiesel yields of some of the crops are enlisted in the Table-1**

Source	Biodiesel yield (barrels per year per square mile)
Cotton	382
Soybean	542
Sesame	807
Safflower	905
Tung oil tree	1091
Sunflower	1113
Peanuts	1233
Rapseed	1385
Rapseed	1407
Jojoba	2116
Jatropha	2204
Coconut	3131
Oil palm (Oil palm)	6927

➢ CHARACTERISTICS OF OILS AND FATS USED IN BIODIESEL PRODUCTION

- Oils and fats, known as lipids, are hydrophobic substances insoluble in water and are of animal or vegetal origin.
- They differ in their physical states at room temperature.
- From a chemical viewpoint, lipids are fatty glycerol esters known as triglycerides.
- How triglyceride is formed by condensation from glycerol and fatty acids, and the general chemical formula are shown by the **Figure-20**.

- The three hydrocarbon chains may be of equal or different lengths, depending on the type of oil; they may also differ on the number of double-covalent bonds in each chain.

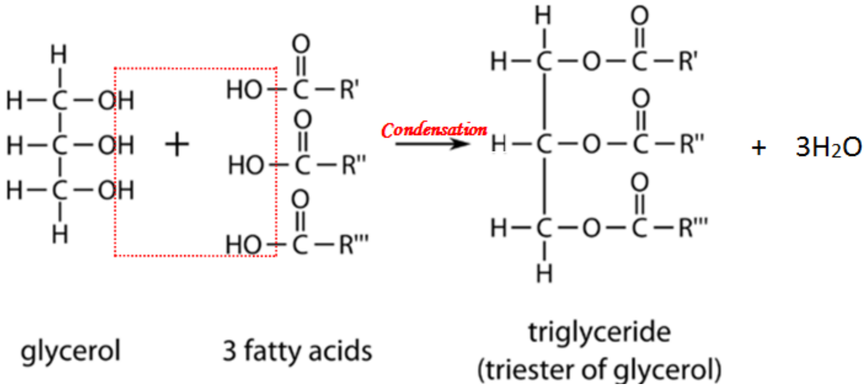


Figure-20: **Formation of triglyceride by condensation from glycerol and fatty acids.**

- R', R'' and R''' represent hydrocarbon chains of fatty acids, which in most cases vary in length from 12 to 18 carbon atoms.
- Fatty acids may be saturated fatty acids (SFA) or non-saturated fatty acids (NSFA).
- In the former, there are only single covalent bonds in the molecules.
- The names of the most important fatty acids in oils are listed in **Table-2** along with their chemical formulas.

Table-2: Important Fatty Acids in Oil

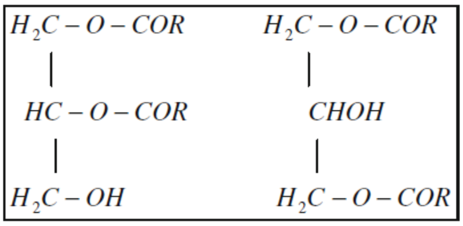
Fatty acid	Chemical formula
Lauric (12:0)	$CH_3\ (CH_2)_{10}\ COOH$
Palmitic (16:0)	$CH_3\ (CH_2)_{14}\ COOH$
Estearic (18:0)	$CH_3\ (CH_2)_{16}\ COOH$
Oleic (18:1)	$CH_3\ (CH_2)_7\ CH = CH\ (CH_2)_7\ COOH$
Linoleic (18:2)	$CH_3\ (CH_2)_4\ CH = CH\ CH_2\ CH = CH\ (CH_2)_7\ COOH$
Linolenic (18:3)	$CH_3\ CH_2\ (CH = CH\ CH2)_3\ (CH2)_6\ COOH$
Erucic (22:1)	$CH_3\ (CH_2)_7\ CH = CH\ (CH2)_{11}\ COOH$
Ricinoleic (18:1)	$CH_3\ (CH_2)_5\ CHOH\ CH_2\ CH = CH\ (CH_2)_7\ COOH$

- The notation x:y indicates the number of carbon atoms in the oil molecule (x) and the number of unsaturations, i.e. double-covalent bonds (y). For instance, y = 0 for all the SFAs.
- **Table-3** indicates the approximate content (in weight) of saturated and non-saturated fatty acids in some vegetable oils and animal fats.
- The most frequent fatty acids in oils are lauric, palmitic, estearic, linoleic and linolenic, although others may also be present.
- It is important to note that vegetable oils differ in their content of fatty acids.
- For instance, ricinoleic acid is the main component of castor oil, whereas in olive oil it is oleic acid, in soybean oil it is linoleic acid, and in linseed oil it is linolenic acid.
- The compositions indicated in **Table-3** do not discriminate between the different saturated or unsaturated fatty acids.

Table-3: Fatty Acids Composition (approximate) of some Vegitable Oils

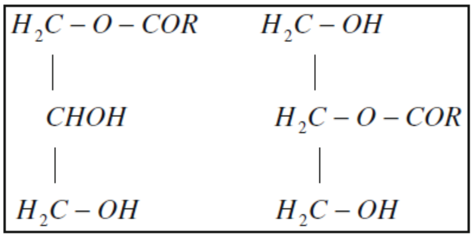
Oil/fat	SFA ($\approx$ % w/w)	NSFA ($\approx$ % w/w)
Coconut	90	10
Corn	13	87
Cottonseed	26	74
Olive	14	86
Palm	49	51
Peanut	17	83
Rapeseed	6	94
Soybean	14	86
Sunflower	11	89
Safflower	9	91
Castor	2	98
Yellow grease	33	67
Lard	41	59
Beef tallow	48	52

- For instance, coconut oil has about 90% of SFAs in its composition (more than half being lauric acid), and palm oil has about 49% SFAs (more than 80% palmitic acid).
- Similarly, 60% of NSFAs content in soybean oil is linoleic acid, while in peanut more than 50% is oleic.
- The US Department of Energy indicates that a perfect biodiesel should only comprise mono-unsaturated fatty acids.
- Vegetable oils may also contain small percentages of monoglycerides and diglycerides.
- Their chemical formulae are shown in Figs. 21 and 22.



$$\begin{array}{ll} H_2C-O-COR & H_2C-O-COR \\ | & | \\ HC-O-COR & CHOH \\ | & | \\ H_2C-OH & H_2C-O-COR \end{array}$$

Figure-21: Diglyceride.



$$\begin{array}{ll} H_2C-O-COR & H_2C-OH \\ | & | \\ CHOH & H_2C-O-COR \\ | & | \\ H_2C-OH & H_2C-OH \end{array}$$

Figure-22: Monoglyceride.

- In addition, there will also be small amounts of free fatty acids (in most vegetable oils, less than 1%, except for palm oil, where they can reach up to 15%).
- The composition of vegetable oils influences their properties.
- For instance, the pour point and cloud point temperatures, cetane number and the iodine index depend on the number of unsaturations and the length of the fatty acid chains.

- A higher content of double-covalent bonds gives a lower solidification point and a higher iodine index.

➢ CHARACTERISTICS OF ALCOHOLS USED IN BIODIESEL PRODUCTION

- Alcohols that can be used in biodiesel production are those with short chains, including methanol, ethanol, butanol, and amylic alcohol.
- The most widely used alcohols are methanol (CH_3OH) and ethanol (C_2H_5OH) because of their low cost and properties.
- Methanol is often preferred to ethanol in spite of its high toxicity because its use in biodiesel production requires simpler technology; excess alcohol may be recovered at a low cost and higher reaction speeds are reached.
- The comparison between the two alcohols is summarized in **Box-1**.

Box-1

Most Important Alcohols used in Biodiesel Production

Methanol: Most widely used, in spite of its toxicity. It is a substance of petrochemical origin.

Ethanol: Less used, requires more complex production technology and the reaction speeds are lower. It can be produced from biomass.

- It must be remembered that in order for biodiesel to be a fully renewable fuel, it should be obtained from vegetable oils and animal fats, together with an alcohol that is produced from biomass, such as bioethanol, instead of being a petrochemical product.
- Several countries are carrying out research towards this objective, such as Spain and Brazil.

➢ BIODIESEL PRODUCTION PROCESS

- Biodiesel is produced from vegetable oils or animal fats and an alcohol, through a transesterification reaction.
- This chemical reaction converts an ester (vegetable oil or animal fat) into a mixture of esters of the fatty acids that makes up the oil (or fat).
- Biodiesel is obtained from the purification of the mixture of fatty acid methyl esters (FAME).
- A catalyst is used to accelerate the reaction.
- In order to understand the chemical process, we must know the chemistry of esterification and transesterification.
- **Chemistry of Esterification**
  - ✓ It is the general name for a chemical reaction in which two reactants (typically an alcohol and an acid) form an ester as the reaction product.
  - ✓ Esters are common in organic chemistry and biological materials, and often have a pleasant characteristic, fruity odor. The reaction:

$$CH_3\ COOH + CH_3\ CH_2\ OH \text{ ====> } CH_3COOCH_2CH_3 + H_2O$$

  - ✓ The esterification reaction may also be written as reflected by **Figure-23**.

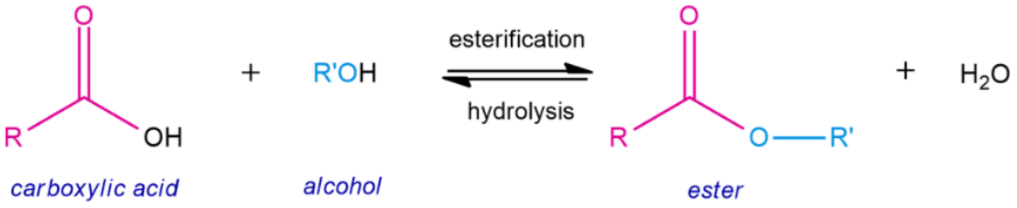


Figure-23: Esterification reaction (R, R' different alkyle groups).

- **Chemistry of Transesterification**
  - In organic chemistry, transesterification is the process of exchanging the organic group R″ of an ester with the organic group R′ of an alcohol.
  - These reactions are often catalyzed by the addition of an acid or base catalyst.
  - The reaction can also be accomplished with the help of enzymes (biocatalysts) particularly lipases.
  - A generic transesterification reaction has been reflected by the reaction (1) and **Figure-24**.

$$R'OH + R''COOR \xrightleftharpoons{\text{Catalyst}} R''OH + R'COOR \qquad (1)$$

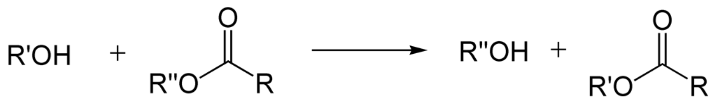


Figure-24: A generic Transesterification reaction.

- According to the catalyst used, transesterification can be basic, acidic or enzymatic, the former being the most frequently used, as indicated in **Box 2.**
- RCOOR' indicate an ester, R"OH an alcohol, R'OH another alcohol (glycerol), RCOOR" an ester mixture and a catalyst:

Box 2

Transesterification Reactions for Biodiesel Production

Basic: Most frequently used at all production scales.

Acid: Less frequent in industrial production, sometimes used a first stage with highly acidic raw materials.

Enzymatic: Less used; the enzymes are usually lipases.

- When methanol is the alcohol used in the transesterification process, the product of the reaction is a mixture of methyl esters (fatty esters); as shown in **Figure-25**.

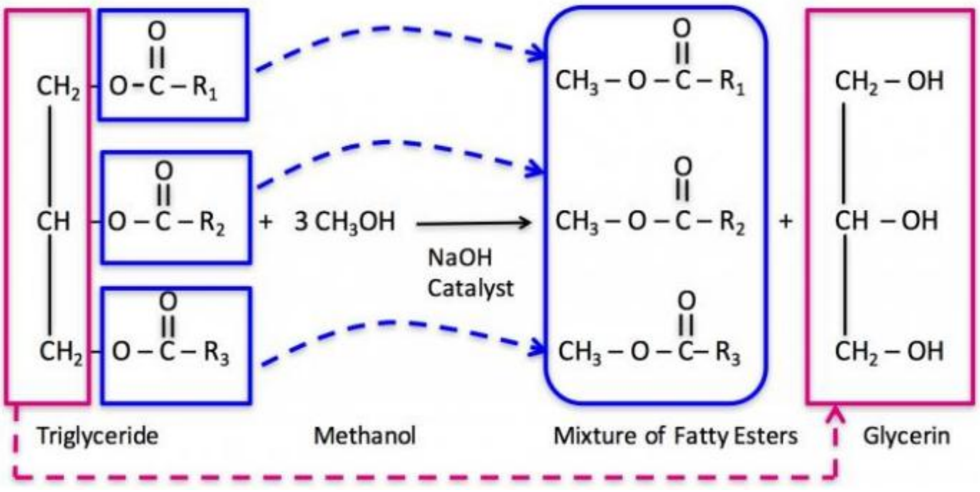


Figure-25: Transesterification with methyl alcohol.

✓

- ✓ Similarly, when ethanol (ethyl alcohol) is used in the process of transesterification, the product is a mixture of ethyl esters (fatty esters) as shown in the **Figure-26**.

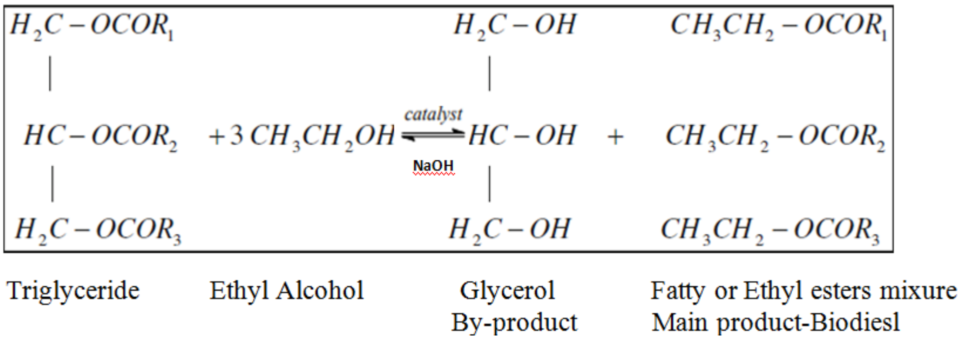


Figure-26: Transesterification with ethyl alcohol.

- ✓ In both cases, glycerin is the by-product (co-product) of the reaction.
- ✓ Although transesterification is the most important step in biodiesel production (since it originates the mixture of esters), additional steps are necessary to obtain a product that complies with international standards, as shown in **Box 3**.

Box 3

Stages of Biodiesel Production Process

Treatment of raw materials
Alcohol-catalyst mixing
Chemical reaction
Separation of the reaction products
Purification of the reaction products

- ✓ In consequence, once the chemical reaction is completed and the two phases (mix of esters and glycerin) are separated, the mix of methyl esters must be purified to reduce the concentration of contaminants to acceptable levels.
- ✓ These include remnants of catalyst, water and methanol; the latter is usually mixed in excess proportion with the raw materials in order to achieve higher conversion efficiency in the transesterification reaction.
- ✓ In the succeeding sections, the process flow chart along with the steps of the purification are discussed in detail.

➢ TECHNICAL PROCESS DESCRIPTION FOR PRODUCTION OF BIODIESEL

As mentioned earlier, the steps involved are:

a. Treatment of raw materials
b. Alcohol-catalyst mixing
c. Chemical reaction
d. Separation of the reaction products
e. Purification of the reaction products

- **Treatment of Raw Materials**
  - ✓ The content of free fatty acids, water and non-saponificable substances are key parameters to achieve high conversion efficiency in the transesterification reaction.

- ✓ The use of basic catalysts in triglycerides with high content of free fatty acids is not advisable, since part of the latter reacts with the catalyst to form soaps.
- ✓ In consequence, part of the catalyst is spent, and it is no longer available for transesterification.
- ✓ In summary the efficiency of the reaction diminishes with the increase of the acidity of the oil; basic transesterification is viable if the content of free fatty acids (FFAs) is less than 2%.
- ✓ In the case of highly acidic raw materials (animal fats from cattle, poultry, pork; vegetable oils from cotton, coconut, most used oils, etc.) an acid transesterification is necessary as a preliminary stage, to reduce the level of FFAs to the above-mentioned value.
- ✓ Besides having low humidity and acid content, it is important that the oil presents a low level of non-saponificable substances.
- ✓ If the latter were to be present in significant amounts and soluble in biodiesel, it would reduce the level of esters in the product, making it difficult to comply with the minimum ester content required by the standards.
- ✓ The American Oil Chemists Society (AOCS) standards list the required properties of oils.
- ✓ The properties required by the oils are finally determined by the biodiesel industry in each country.
- ✓ For instance, in Argentina the oils for biodiesel production usually have:
    - Acidity level\0.1 mg KOH/g
    - Humidity\500 ppm
    - Peroxide index\10 meq/kg
    - Non-saponificable substances\1%.

- **Alcohol-Catalyst Mixing**
    - ✓ The alcohol used for biodiesel production must be mixed with the catalyst before adding the oil.
    - ✓ The mixture is stirred until the catalyst is completely dissolved in the alcohol.
    - ✓ It must be noted that the alcohol must be water-free (anhydrous) for the reasons explained in the previous paragraph.
    - ✓ Sodium and potassium hydroxides are among the most widely used basic catalysts.
    - ✓ For production on an industrial scale, sodium or potassium methoxides or methylates are commercially available.
    - ✓ Of course, due caution must be exercised, and all applicable safety regulations must be followed, when working with methanol, hydroxides and methoxides, independently of the production scale.
    - ✓ The alcohol-to-oil volume ratio, R, is another key variable of the transesterification process.
    - ✓ The stoichiometric ratio (**Figs. 25, 26**) requires 1 mol of oil to react with 3 mol of alcohol, to obtain 3 mol of fatty acids methyl esters (FAME) and1 mol of glycerin.
    - ✓ However, since the reaction is reversible, excess alcohol as a reactant will shift the equilibrium to the right side of the equation, increasing the amount of products (as it may be inferred from Le Chatelier’s principle).

- ✓ Although a high alcohol-to-oil ratio does not alter the properties of FAME, it will make the separation of biodiesel from glycerin more difficult, since it will increase the solubility of the former in the latter.
- ✓ Usually, a 100% alcohol excess is used in practice, that is, 6 mol of alcohol per mole of oil.
- ✓ This corresponds to a 1:4 alcohol-to-oil volume ratio (R = 0.25).
- ✓ Finally, it must be noted that the necessary amount of catalyst is determined taking into account the acidity of the oil, by titration.

- **Chemical Reaction**
  - ✓ The chemical reaction takes place when the oil is mixed with the alkoxide (alcohol–catalyst mix) described in the previous paragraph.
  - ✓ This requires certain conditions of time, temperature and stirring.
  - ✓ Since alcohols and oils do not mix at room temperature, the chemical reaction is usually carried out at a higher temperature and under continuous stirring, to increase the mass transfer between the phases.
  - ✓ Usually, emulsions form during the course of the reaction; these are much easier and quicker to destabilize when methanol is used, in comparison to ethanol.
  - ✓ Due to the greater stability of emulsions formed, difficulties arise in the phase separation and purification of biodiesel when ethanol is used in the reaction.
  - ✓ The transesterification process may be carried out at different temperatures.
  - ✓ For the same reaction time, the conversion is greater at higher temperatures.
  - ✓ Since the boiling point of methanol is approximately 68 ^{0}C (341 K), the temperature for transesterification at atmospheric pressure is usually in the range between 50 and 60 ^{0}C.
  - ✓ It is very useful to know the chemical composition of the mixture during the reaction; then, if the reaction mechanism and kinetics are known, the process can be optimized.
  - ✓ However, the determination of the mixture composition is not easy, since more than a hundred substances are known to be present.
  - ✓ For instance, for biodiesel production from rapeseed oil (whose main SFAs are palmitic, oleic, linoleic and linolenic) and methanol, with potassium hydroxide as a catalyst, it could be theoretically possible to find 64 isomers of triglycerides, 32 diglycerides, 8 monoglycerides, their methyl esters, potassium salts of the fatty acids, potassium methoxide, water, etc.
  - ✓ The studies on this subject indicate the following general guidelines:
    - ○ For longer reaction times, the concentration of triglycerides diminishes the concentration of esters, increases and the concentration of mono- and diglycerides increases to a maximum and then decreases.
    - ○ Most of the chemical reaction takes place during the first minutes.
    - ○ The absence of mono- and diglycerides at the beginning of the chemical reaction and the increase and reduction of their concentration during the reaction confirm that the production of esters from the triglycerides takes place in three steps, as represented in the equations below:

$$\mathrm{TG} + \mathrm{MOH} \xrightarrow{\mathrm{KOH}} \mathrm{DG} + \mathrm{ME} \tag{2}$$

$$\mathrm{DG} + \mathrm{MOH} \xrightarrow{\mathrm{KOH}} \mathrm{MG} + \mathrm{ME} \tag{3}$$

$$\mathrm{MG} + \mathrm{MOH} \xrightarrow{\mathrm{KOH}} \mathrm{G} + \mathrm{ME} \tag{4}$$

where MOH indicates methanol, ME are the methyl esters, TG, DG and MG are tri-, di- and monoglycerides, respectively, and G is the glycerin.

- **Catalysts**
  - ✓ The catalysts used for the transesterification of triglycerides may be classified as basic, acid or enzymatic, as indicated in **Box 2**.
  - ✓ Basic catalysts include sodium hydroxide (NaOH), potassium hydroxide (KOH), carbonates and their corresponding alcoxides (for instance, sodium methoxide or ethoxide).
  - ✓ There are many references on basic catalysts in the scientific literature.
  - ✓ Acid catalysts include sulfuric acid, sulfonic acids and hydrochloric acid; their use has been less studied by many workers.
  - ✓ Heterogeneous catalysts that have been considered for biodiesel production include enzymes, titanium silicates, and compounds from alkaline earth metals, anion exchange resins and guanidines in organic polymers.
  - ✓ Lipases are the most frequently used enzymes for biodiesel production.

- **Separation of the Reaction Products**
  - ✓ The separation of reaction products takes place by decantation: the mixture of fatty acids methyl esters (FAME) separates from glycerin forming two phases, since they have different densities; the two phases begin to form immediately after the stirring of the mixture is stopped.
  - ✓ Due to their different chemical affinities, most of the catalyst and excess alcohol will concentrate in the lower phase (glycerin), while most of the mono-, di-, and triglycerides will concentrate in the upper phase (FAME).
  - ✓ Once the interphase is clearly and completely defined, the two phases may be physically separated.
  - ✓ It must be noted that if decantation takes place due to the action of gravity alone, it will take several hours to complete.
  - ✓ This constitutes a ‘‘bottleneck’’ in the production process, and in consequence the exit stream from the transesterification reactor is split into several containers.
  - ✓ Centrifugation is a faster, albeit more expensive alternative.
  - ✓ After the separation of glycerin, the FAME mixture contains impurities such asremnants of alcohol, catalyst and mono-, di-, and triglycerides.
  - ✓ These impurities confer undesirable characteristics to FAME, for instance, increased cloud point and pour point, lower flash point, etc.
  - ✓ In consequence a purification process is necessary for the final product to comply with standards.
  - ✓ This is being discussed in the next section.

- **Purification of the Reaction Products**
  - ✓ The mixture of fatty acids methyl esters (FAME) obtained from the transesterification reaction must be purified in order to comply with established quality standards for biodiesel.
  - ✓ Therefore, FAME must be washed, neutralized and dried.
  - ✓ Successive washing steps with water remove the remains of methanol, catalyst and glycerin, since these contaminants are water-soluble.
  - ✓ Care must be taken to avoid the formation of emulsions during the washing steps, since they would reduce the efficiency of the process.
  - ✓ The first washing step is carried out with acidified water, to neutralize the mixture of esters.

- Then, two additional washing steps are made with water only.
- Finally the traces of water must be eliminated by a drying step.
- After drying, the purified product is ready for characterization as biodiesel according to international standards.
- An alternative to the purification process described above is the use of ion exchange resins or silicates.
- Glycerin as obtained from the chemical reaction is not of high quality and has no commercial value.
- Therefore, it must be purified after the phase separation.
- This is not economically viable in small scale production, due to the small glycerin yield.
- However, purification is a very interesting alternative for large-scale production plants, since, in addition to the high quality glycerin, part of the methanol is recovered for reutilization in the transesterification reaction (both from FAME and glycerin), and thus lowering biodiesel production costs.
- The steady increase of biodiesel production is fostering research for novel uses of glycerin in the production of high-value-added products.
- It must be noted that the stages of the biodiesel production process (summarized in **Box 3**) are the same for all the production scales (laboratory, pilot plant, small, medium-, and large-scale industries).
- However, the necessary equipment will be significantly different.

- **Common Flow Chart for Production of Biodiesel**

  Common flow diagrams have been given in figures 27-29.

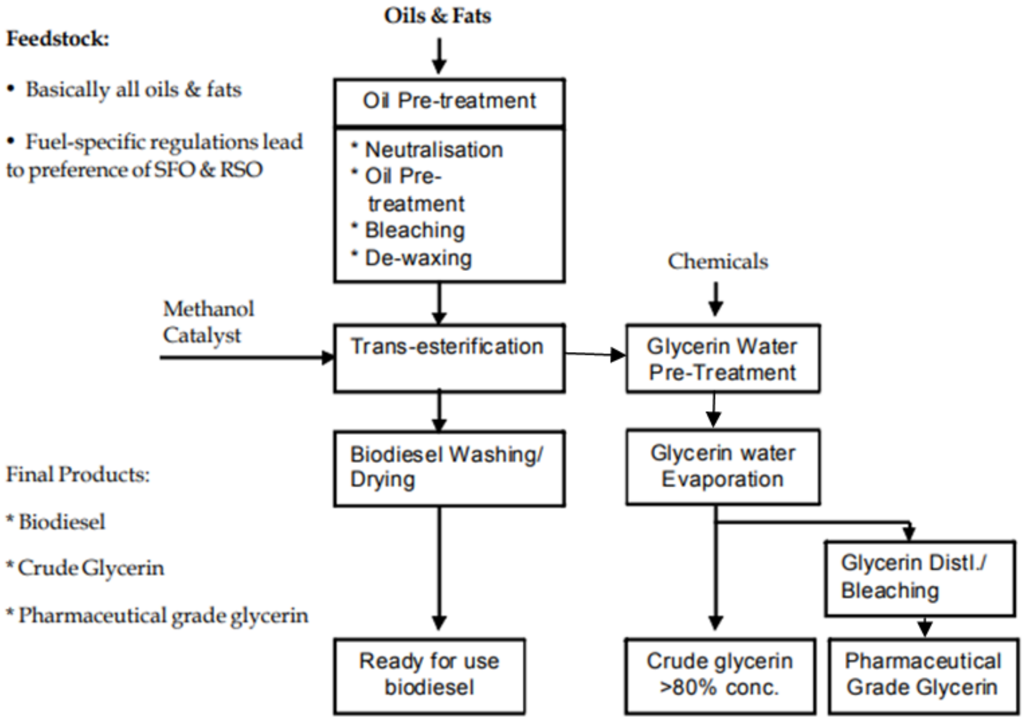


Figure-27: General flow chart for production biodiesel using methanol.

Although, the diagrams are self explained may be further elaborated by the students while answering the questions.

- Another diagram indicating the unit operations along with the equipment has been shown by **Figure-28** may be explained in the light of the process stages already discussed.

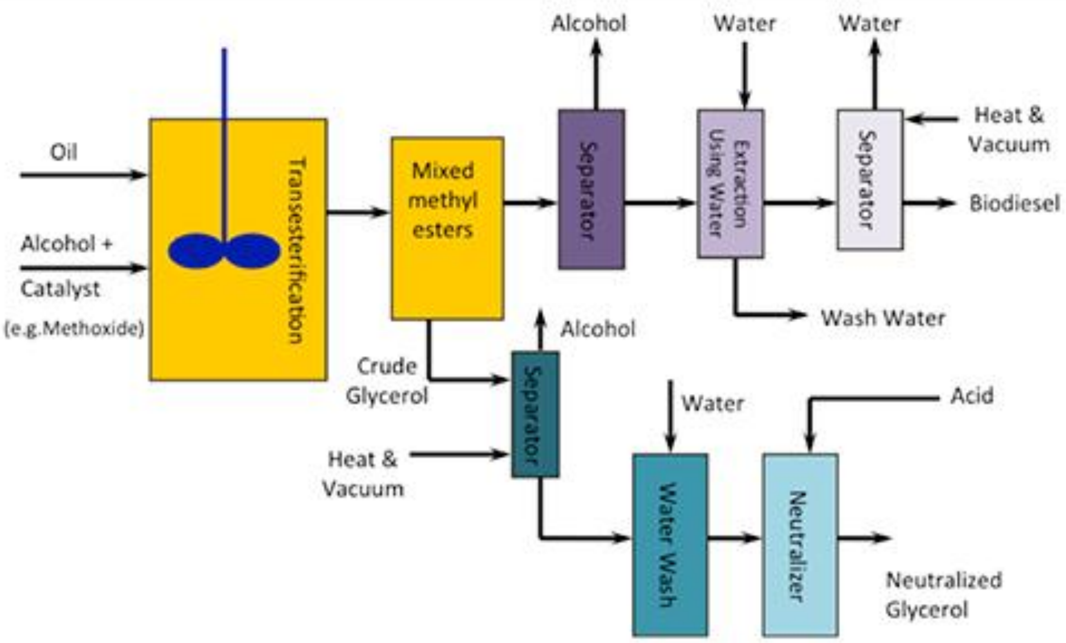


Figure-28: A general schematic of biodiesel process using transesterification.

- **Figure-29** presents a process flow diagram built up with the conceptual elements examined so far.
  - ✓ The flow sheet aims to illustrate different reaction and separation techniques.
  - ✓ Note that the heat-integration elements are not presented in order to keep the flowsheet simple.
  - ✓ The feed of oil and methanol, including recycle, ensures a molar ratio methanol : oil of 6 : 1.
  - ✓ Good mixing of reactants is necessary to ensure a homogeneous reaction phase, otherwise an induction period will affect the conversion.
  - ✓ The mixing of reactants can be done preferably in a static device.
  - ✓ After heating at about 65 ° C the esterification starts in the reactor R-1, which can be a CSTR, but preferably a PFR or a combination PFR/CSTR.
  - ✓ The first reactor should ensure a conversion slightly above 90%.
  - ✓ Intermediate removal of glycerol takes place to shift the equilibrium and get lower content of monoglyceride.
  - ✓ A simple phase split by decanting can be applied at temperatures of 40 to 60 °C.
  - ✓ The decanting time could be very variable, between a few minutes and 1 h.
  - ✓ The presence of soaps and monoglycerides hinders the phase separation, while more neutral pH and lower methanol content helps.
  - ✓ Modern coalescence separators can ensure a relatively smooth separation if the amount of soap is not excessive.
  - ✓ After makeup in methanol and catalyst the oil phase is submitted to a second transesterification step in the reactor R-2.
  - ✓ The conversion should rise to a minimum of 98.5%.
  - ✓ The phase separation is done this time by means of the centrifuge K-1.

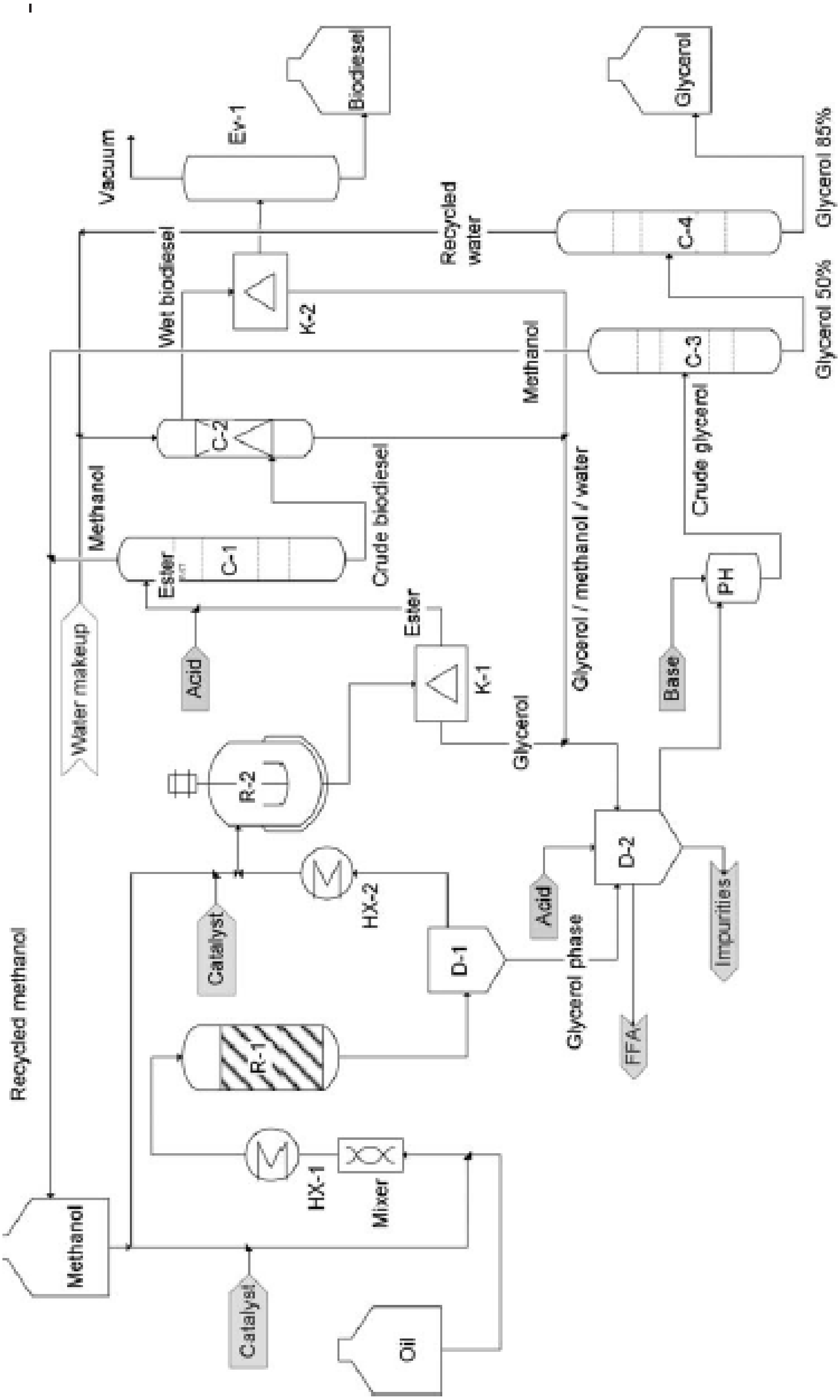
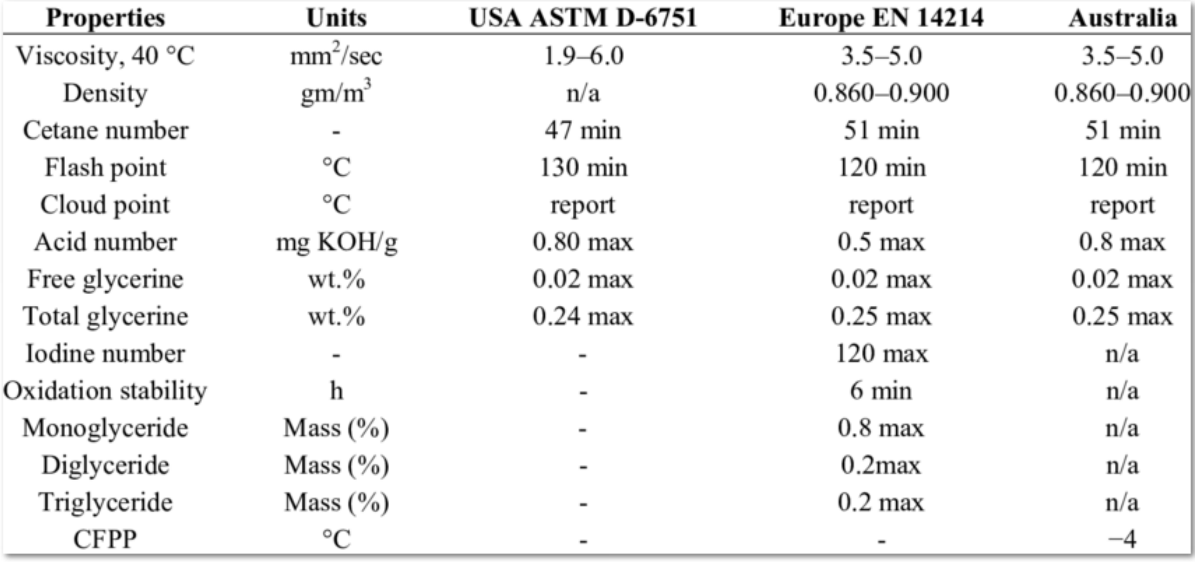


Figure-29: Process flow diagram for biodiesel manufacturing.

- ✓ It is worth noting that centrifugal phase separation is becoming a state - of - the - art method in biodiesel technology.
- ✓ Centrifugation can be applied in all separation steps, including neutralization, washing and soap removal.
- ✓ Centrifuges of various capacities are available, such as from 60 to 1600 tonne/day.
- ✓ After neutralization with acid, H_3PO_4 , the resulting mixture is submitted to methanol stripping.
- ✓ In this way, about 90% from the excess methanol can be recycled directly to reaction section.
- ✓ The next stage is a wash with hot water at 50 °C for deep purification from methanol, glycerol, soaps and salt.
- ✓ This operation can be done in a counter-current column, or directly in a centrifugal separator.
- ✓ Because of the presence of double bounds and ester groups the oil phase will contain a significant amount of water, well above the threshold of 300 ppm set by the specification norm.
- ✓ The removal of water and residual solids from the biodiesel is done by centrifugation in the unit K-2, and the final polishing before shipping by a vacuum flash evaporator.
- ✓ The glycerol recovered in the previous stages is treated with acid for catalyst neutralization.
- ✓ The soaps are converted to fatty acids that precipitate at the top of the glycerol phase, and are sent back to the esterification stage.
- ✓ After pH adjustment the crude glycerol is submitted to methanol recovery by vacuum distillation.
- ✓ The bottom product is usually a glycerol – water solution of about 50%.
- ✓ Its concentration can be increased up to 85% by vacuum evaporation.
- ✓ The water is recycled to the washing step. Normally, the glycerol is shipped to a specialized refiner, where the purity can be increased further to 99.5 and 99.7% by ion-exchange techniques and vacuum distillation.

➢ STANDARD PROPERTIES OF BIODIESEL

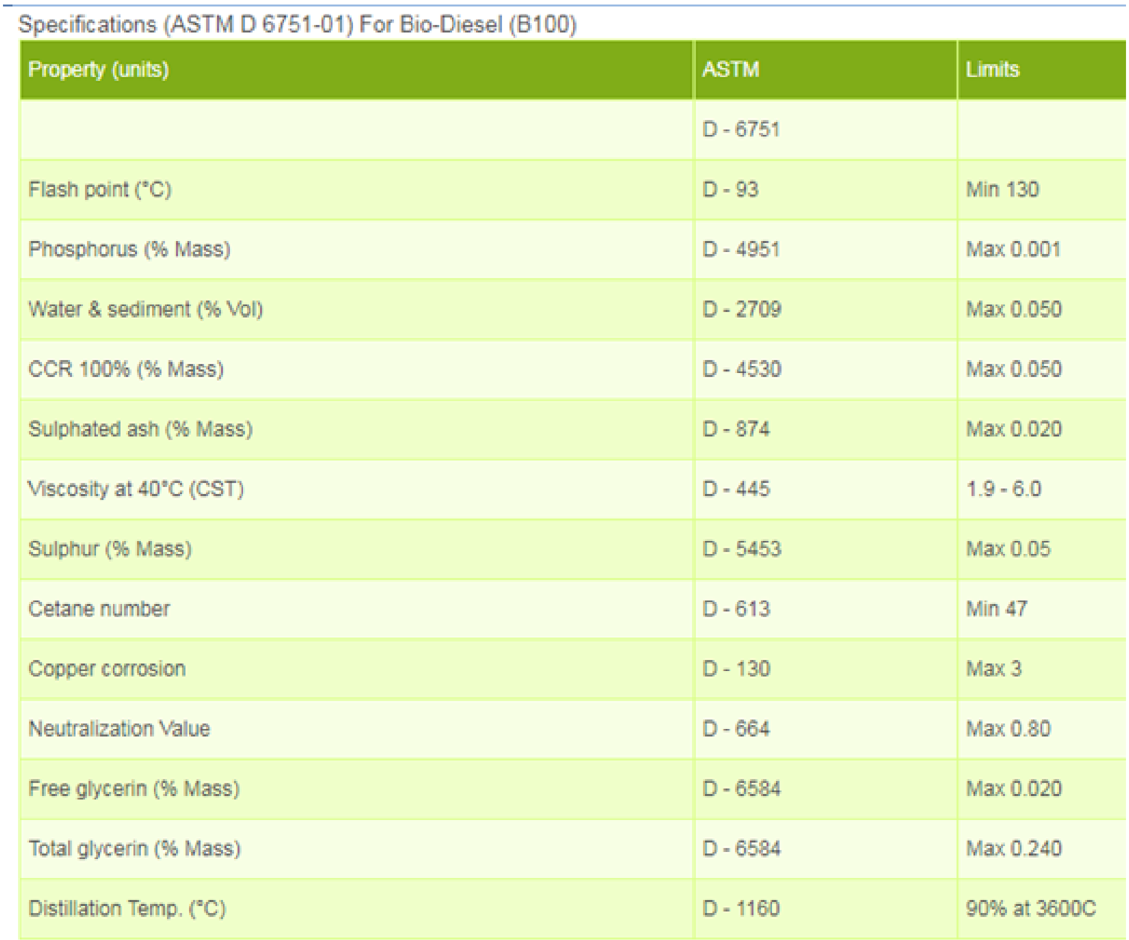
- **International Biodiesel Standard Table-4**



Properties	Units	USA ASTM D-6751	Europe EN 14214	Australia
Viscosity, 40 °C	mm^2/sec	1.9–6.0	3.5–5.0	3.5–5.0
Density	gm/m^3	n/a	0.860–0.900	0.860–0.900
Cetane number	-	47 min	51 min	51 min
Flash point	°C	130 min	120 min	120 min
Cloud point	°C	report	report	report
Acid number	mg KOH/g	0.80 max	0.5 max	0.8 max
Free glycerine	wt.%	0.02 max	0.02 max	0.02 max
Total glycerine	wt.%	0.24 max	0.25 max	0.25 max
Iodine number	-	-	120 max	n/a
Oxidation stability	h	-	6 min	n/a
Monoglyceride	Mass (%)	-	0.8 max	n/a
Diglyceride	Mass (%)	-	0.2max	n/a
Triglyceride	Mass (%)	-	0.2 max	n/a
CFPP	°C	-	-	−4

- **PCRA, GOI Biodiesel should meet the following International Standards**

Table-5: International Standards for Biodiesel

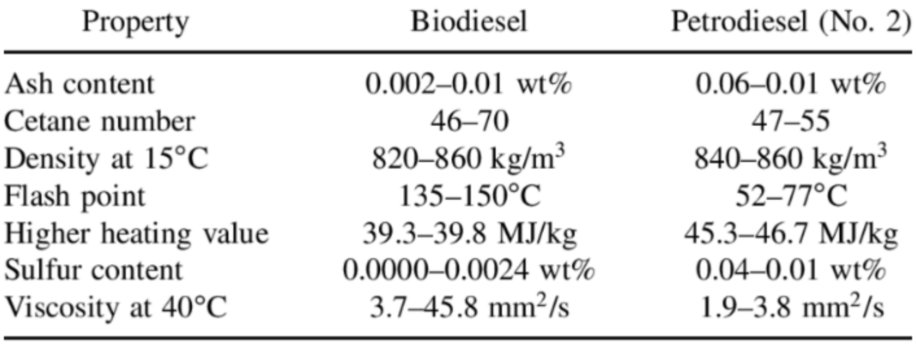


Specifications (ASTM D 6751-01) For Bio-Diesel (B100)

Property (units)	ASTM	Limits
	D - 6751	
Flash point (°C)	D - 93	Min 130
Phosphorus (% Mass)	D - 4951	Max 0.001
Water & sediment (% Vol)	D - 2709	Max 0.050
CCR 100% (% Mass)	D - 4530	Max 0.050
Sulphated ash (% Mass)	D - 874	Max 0.020
Viscosity at 40°C (CST)	D - 445	1.9 - 6.0
Sulphur (% Mass)	D - 5453	Max 0.05
Cetane number	D - 613	Min 47
Copper corrosion	D - 130	Max 3
Neutralization Value	D - 664	Max 0.80
Free glycerin (% Mass)	D - 6584	Max 0.020
Total glycerin (% Mass)	D - 6584	Max 0.240
Distillation Temp. (°C)	D - 1160	90% at 3600C

Source: Website: PCRA, Ministry of Petroleum and Natural Gas, GOI

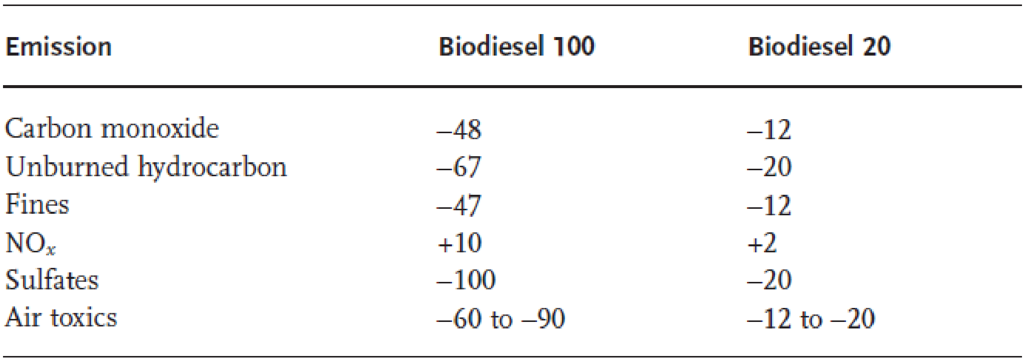
- **Table-6: Physical Properties of Biodiesel and Petro Diesel**



Property	Biodiesel	Petrodiesel (No. 2)
Ash content	0.002–0.01 wt%	0.06–0.01 wt%
Cetane number	46–70	47–55
Density at 15°C	820–860 kg/m^3	840–860 kg/m^3
Flash point	135–150°C	52–77°C
Higher heating value	39.3–39.8 MJ/kg	45.3–46.7 MJ/kg
Sulfur content	0.0000–0.0024 wt%	0.04–0.01 wt%
Viscosity at 40°C	3.7–45.8 mm^2/s	1.9–3.8 mm^2/s

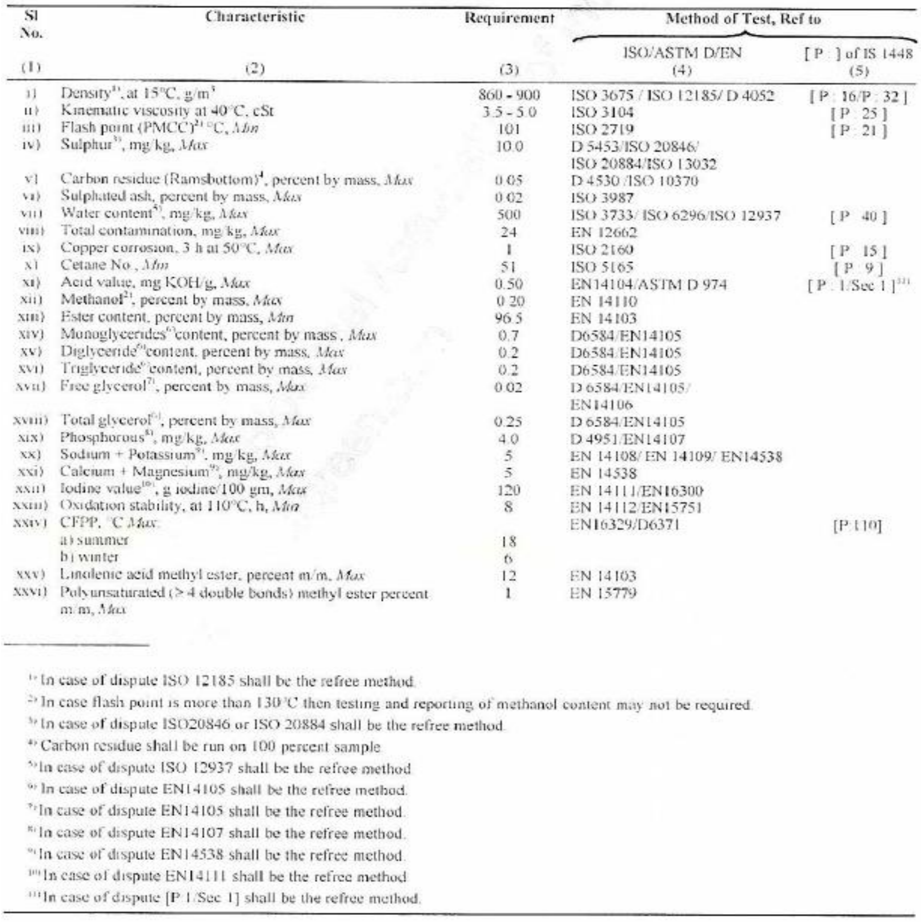
Source: Demirbas (2002).

- **Table-7: Emissions Generated by Biodiesel Compared with Normal Diesel (in %)**.



Emission	Biodiesel 100	Biodiesel 20
Carbon monoxide	–48	–12
Unburned hydrocarbon	–67	–20
Fines	–47	–12
NO_x	+10	+2
Sulfates	–100	–20
Air toxics	–60 to –90	–12 to –20

- **Table-8: Indian Biodiesel Standard (BIS-15607: 2016)**



Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			ISO/ASTM D/EN	[P :] of IS 1448
(1)	(2)	(3)	(4)	(5)
i)	Density[1], at 15°C, g/m³	860 - 900	ISO 3675 / ISO 12185/ D 4052	[P : 16/P : 32]
ii)	Kinematic viscosity at 40°C, cSt	3.5 - 5.0	ISO 3104	[P : 25]
iii)	Flash point (PMCC)[2] °C, *Min*	101	ISO 2719	[P : 21]
iv)	Sulphur[3], mg/kg, *Max*	10.0	D 5453/ISO 20846/ ISO 20884/ISO 13032	
v)	Carbon residue (Ramsbottom)[4], percent by mass, *Max*	0.05	D 4530 /ISO 10370	
vi)	Sulphated ash, percent by mass, *Max*	0.02	ISO 3987	
vii)	Water content[5], mg/kg, *Max*	500	ISO 3733/ ISO 6296/ISO 12937	[P : 40]
viii)	Total contamination, mg/kg, *Max*	24	EN 12662	
ix)	Copper corrosion, 3 h at 50°C, *Max*	1	ISO 2160	[P : 15]
x)	Cetane No., *Min*	51	ISO 5165	[P : 9]
xi)	Acid value, mg KOH/g, *Max*	0.50	EN14104/ASTM D 974	[P : 1/Sec 1][11]
xii)	Methanol[2], percent by mass, *Max*	0.20	EN 14110	
xiii)	Ester content, percent by mass, *Min*	96.5	EN 14103	
xiv)	Monoglycerides[6] content, percent by mass , *Max*	0.7	D6584/EN14105	
xv)	Diglyceride[6] content, percent by mass, *Max*	0.2	D6584/EN14105	
xvi)	Triglyceride[6] content, percent by mass, *Max*	0.2	D6584/EN14105	
xvii)	Free glycerol[7], percent by mass, *Max*	0.02	D 6584/EN14105/ EN14106	
xviii)	Total glycerol[6], percent by mass, *Max*	0.25	D 6584/EN14105	
xix)	Phosphorous[8], mg/kg, *Max*	4.0	D 4951/EN14107	
xx)	Sodium + Potassium[9], mg/kg, *Max*	5	EN 14108/ EN 14109/ EN14538	
xxi)	Calcium + Magnesium[9], mg/kg, *Max*	5	EN 14538	
xxii)	Iodine value[10], g iodine/100 gm, *Max*	120	EN 14111/EN16300	
xxiii)	Oxidation stability, at 110°C, h, *Min*	8	EN 14112/EN15751	
xxiv)	CFPP, °C *Max*:		EN16329/D6371	[P:110]
	a) summer	18		
	b) winter	6		
xxv)	Linolenic acid methyl ester, percent m/m, *Max*	12	EN 14103	
xxvi)	Polyunsaturated (≥ 4 double bonds) methyl ester percent m/m, *Max*	1	EN 15779	

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[1] In case of dispute ISO 12185 shall be the refree method.

[2] In case flash point is more than 130°C then testing and reporting of methanol content may not be required.

[3] In case of dispute ISO20846 or ISO 20884 shall be the refree method.

[4] Carbon residue shall be run on 100 percent sample.

[5] In case of dispute ISO 12937 shall be the refree method.

[6] In case of dispute EN14105 shall be the refree method.

[7] In case of dispute EN14105 shall be the refree method.

[8] In case of dispute EN14107 shall be the refree method.

[9] In case of dispute EN14538 shall be the refree method.

[10] In case of dispute EN14111 shall be the refree method.

[11] In case of dispute [P 1/Sec 1] shall be the refree method.

PCRA: Some of the queries and their answers w.r.t. Bio Diesel

Q: What is Bio-Diesel? How can it be manufactured?

A: Bio-Diesel is an eco-friendly, alternative diesel fuel prepared from domestic renewable resources i.e. vegetable oils and animal fats. These natural oils and fats are made up mainly of triglycerides. These triglycerides when reacted chemically with lower alcohols in presence of a catalyst result in fatty acid esters. These esters show striking similarity to petroleum derived diesel and are called "Bio-Diesel".

Q: What are the plant species that are suitable for preparation of Bio-Diesel? What size of Bio-Diesel plant works out to be economical?

A: In the US bio-diesels are mainly derived from Soybean oil and in Europe from the Sunflower oil. However, as India is deficient in edible oils, non-edible oils may be material of choice for producing Bio-Diesel like Jatropha, Karanjia and Rice bran oils, etc. Since, in India bio-diesel is in research phase, so, economics would only be generated later.

Q: What are the advantages of Bio-Diesel for a country like India?
A: Bio-Diesel has been more attractive recently because of its environmental benefits and the fact that it is made from renewable sources and can be prepared locally. Since India is deficient in edible oils, therefore, the non-edible oil like Karanjia, Jatropha, etc. could be the desirable source for India for production of bio-diesel. These plants could be grown on wasteland, about 80 million hectare of which is available in India . These crops grow in arid and semi-arid region and require almost no post plantation management and care. Since, all most all the wasteland is available in rural and economically underdeveloped region, the large-scale bio-diesel production has an enormous potential for employment and development of these areas.

Q: How compatible is the Bio-Diesel with the vehicle and pump engines? What are the changes that are required to be made in the engine?
A: The major application of Bio-Diesel is in transportation sector as an alternate to mineral diesel. Many automobiles builders like Ford, John Deere, Massey-Ferguson, Mercedes, BMW, Volkswagen, Volvo, etc have accepted Bio-diesel as the fuel suitable for their vehicles in the existing diesel engines. However, mostly Bio-diesel is used in 10% or 20% blends rather than as neat Bio-diesel. This blending approach also avoids the need to build a separate & costly infrastructure for storing Bio-diesel. Though Bio-diesel is recommended for use in almost all diesels run vehicles, the fuel must meet the ASTM/DIN specifications.

Q: What made IOC take up research in Bio-Diesel? Is the technology and the product a proven success?
A: The depletion of the world petroleum reserves coupled with the global environmental problems stimulated the search for the alternative source for petroleum fuel. Bio-Diesel is one of such candidates for the exploitation as an alternative to diesel fuel. In laboratory process, the Bio-Diesel prepared, has been evaluated for various physico-chemical properties. Subsequently, we have successfully scaled up the production of Bio-Diesel at pilot scale (60 lit) at IOC (R&D) Centre. The product obtained has been evaluated for various physico-chemical properties and it was found that the synthesized Bio-Diesel meets the ASTM bio-diesel specification. Field trials and emission testing of Bio-diesel is being pursued at IOC (R&D) Centre.

Q: What are the areas in the country wherein cultivation of species suitable for manufacturing Bio-Diesel can be taken up?
A: As discussed above, that Bio-Diesel will be manufactured in the rural areas because of the wasteland and cheap labor availability. More precisely, it will depend upon the wasteland locations in the country.

Q: What is the present status of Bio-Diesel research under IOC? By when would the technology become commercially available?
A: IOC (R&D) has examined and optimized the synthetic processes for the preparation of Bio-Diesel from various vegetable oils, which include oils from Rice bran, Jatropha curcas,

Palm, Karanjia, Sunflower etc. using methanol and ethanol as alcohol. In laboratory process, the Bio-Diesel prepared, has been evaluated for various physico-chemical properties. Subsequently, we have successfully scaled up the production of Bio-Diesel at pilot scale (60 lit) at IOC (R&D) Centre. The product obtained has been evaluated for various physico-chemical properties and it was found that the synthesized bio-diesel meets the ASTM bio-diesel specification. Field trials and emission testing of Biodiesel is being pursued at IOC (R&D) Centre.

Q: Why Bio-diesel is important for Indian Railways?

A: Indian Railways is the largest user of diesel, about two million MTPA. Indian Railway is also owner of very large area of land, which presently is without organized plantation. Railways have shown interest to plant the Bio-Crops along the rail tracks and other land available and it is estimated that the railways can produce enough Bio-diesel to replace about 5 to 10% of diesel required for their use. Introduction of bio-diesel in Railways may serve two objectives. Firstly, lowering of emissions and secondly providing enough lubricity to the diesel. The lubricity of low sulphur diesel is likely to cause concerns in near future when the sulphur level of diesel will fall below 500 ppm. Bio-diesel at 1-2% level in petrodiesel will retain the lubricity of the low sulphur fuel.

(Reaffirmed 0)

भारतीय मानक
Indian Standard

IS 15607 : 2016

बायोडीजल (बी100) — फैटी एसिड मिथाइल एस्टर (प्रसिद्धि) — विशिष्टि

(पहला पुनरीक्षण)

Biodiesel (B100) — Fatty Acid Methyl Esters (Fame) — Specification

(First Revision)

ICS 75.160.20

भारतीय मानक ब्यूरो
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May 2016 **Price Group 2**

Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee, PCD 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

India is a growing economy and the energy is a critical input for socio-economic development. Petro-based fuels meet about 95 percent of the requirement of transportation fuels and the demand has been steadily rising. The domestic crude oil is able to meet only about 20 percent of the demand, while the rest is met through imports. Moreover, the fossil fuels resources are limited, non renewable, polluting and therefore, need to be used prudently. On the other hand, renewable energy sources are indigenous, non polluting and virtually inexhaustible and India is endowed with abundant renewable energy sources. Therefore, their use is required to be encouraged in all possible ways. Biodiesel is one such renewable and alternative fuel which can be used as standalone or its blend in diesel fuel as clean fuel. Biodiesel is non-toxic and biodegradable. This would also help to improve our energy security as well as to reduce exhaust emissions. Moreover, in order to meet the National Policy targets on Biofuels, use in transport sector, use of high concentration of Biodiesel in diesel fuel would play a major role.

Biofuels — Ethanol and Biodiesel — are being increasingly used in many countries as renewable fuels and is better option from environmental aspects also.

Biodiesel is made from virgin or used vegetable oils (both edible and non-edible), waste vegetable oils, by-products of edible oil manufacture like, fatty acids, stearin, etc. and animal fats through trans-esterification or esterification. It is finding use as an automotive fuel for diesel engines at 100 percent concentration, or as an extender. Diesel engines require very little or no modification to use the biodiesel up to 20 percent blend in normal diesel fuel and minor modification for higher percentage blends. The use of biodiesel results in substantial reduction of un-burnt hydrocarbons, carbon monoxide and particulate matter compared to conventional diesel. It has almost no sulphur, no aromatics and about 10 percent built-in oxygen which help in ensuring complete combustion. Its higher cetane number also improves combustion. Biodiesel besides helping in reducing the air pollution, improves lubricity of low sulphur diesel fuels required for meeting Euro III, Euro IV and Euro V emission norms.

While sunflower and rapeseed are the raw materials used in Europe for manufacturing biodiesel, soya bean is mostly used in USA. Thailand uses palm oil. Ireland uses frying oil and animal fats. In India, edible oil is not expected to be used for production of biodiesel as its demand being higher than its domestic production. Keeping in view the climatic conditions and availability of considerable under stocked forest land and non-forest land, it is envisaged that Jatropha Curcas and Pongamia Pinnata ('Honge' or 'Karanja') and such plants may be cultivated profitably in India. The seeds of these plants bear rich in oil which is expected to be used for production for biodiesel through trans-esterification in combination with methanol.

The stability and usability of biodiesel after long term storage is being studied. Annex A provides guidance to consumers of biodiesel (B 100) who may wish to store quantities of fuels for extended periods.

The standard was first published in 2005, wherein requirements for biodiesel were stipulated for its as blend stock up to 20 percent in automotive diesel fuel. The feedstock defined was very limited and both fatty acid alkyl esters of both methanol and ethanol were allowed.

In this revision title and scope have been modified to include use of standalone biodiesel as pure fuel. Further it will cover only fatty acid methyl esters (FAME). Requirements of residuum oil and dye/marker has been deleted. Requirement of glycerides, phosphorus have been included for a check on feedstock. More variety of feedstock that can be used to manufacture biodiesel have been included in line with international practices. This revision is formulated keeping in view of the end use application, production and feed stock availability.

Considerable assistance has been drawn from ASTM D 6751-15 and EN 14214-14, while formulating this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant placed retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BIODIESEL (B100) — FATTY ACID METHYL ESTERS (FAME) — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and tests for biodiesel (B100) — fatty acid methyl esters (FAME) for use in compression ignition engines designed for using as stand-alone fuel and as a blend stock for diesel fuel. B 100 stand-alone can also be used for heating applications and industrial engines.

2 REFERENCES

The standards listed in Annex B contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex B.

3 TERMINOLOGY

For the purpose of this standard the following terms shall apply.

3.1 Biodiesel — Fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100. The term 'Biodiesel' refers to mono alkyl methyl esters of vegetable oils like rapeseed, soya bean, sunflower, jatropha curcas, karanja, palm stearin, etc. used cooking oils, waste vegetable oils, by- products of edible oil manufacture like free fatty acids, stearin, etc, and other fats.

3.2 B6 to B20 — Fuel blend consisting of 6 volume percent to 20 volume percent of biodiesel (B100) conforming to the requirements of this standard with the remainder being a light middle or middle distillate grade diesel fuel (*see* IS 1460) and meeting the requirements of this specification.

4 REQUIREMENTS

4.1 General

The biodiesel (B100) shall be mono-alkyl methyl esters of long chain fatty acids from vegetable oil and animal fats.

4.2 Additives

It may contain small amounts of hydrocarbon or non-hydrocarbon additives (*see* Annex A) to improve ignition or other characteristics.

In order improve the performance, use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount to avoid deterioration of drivability and help emission control durability. Other technical means with equivalent effect may also be used.

In order to improve the oxidation stability of FAME, it is recommended to add suitable oxidation stability enhancing additives to FAME at the production stage and before storage to obtain maximum benefits.

Biocides or biostats may be added which destroy or inhibit the growth of fungi and bacteria, which can grow at fuel water interfaces to give high particulate concentrations in the fuel.

4.3 The materials shall be free from grit, suspended matter and other visible impurities.

4.4 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods as given in col 4 and col 5 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable airtight sealed containers as agreed to between the purchaser and the supplier. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel. Use of copper and copper-containing alloy materials should be avoided for packing and transportation of biodiesel as such material may cause increased sediment and deposit formation detrimental to the performance. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

5.2 Marking

5.2.1 Each container shall be marked with the following information:

a) Name and grade of the material;
b) Indication of the source of manufacture, initials or trade-mark, if any;
c) Volume of the contents, in litre;
d) Batch No./Month and year of manufacture or packing; and
e) Any other information, if required.

5.3 BIS Certification Marking

The container may also be marked with the standard Mark.

5.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of the Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).

Table 1 Requirement for Biodiesel

(*Clause* 4.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			ISO/ASTM D/EN	[P :] of IS 1448
(1)	(2)	(3)	(4)	(5)
i)	Density[1], at 15°C, g/m³	860 - 900	ISO 3675 / ISO 12185/ D 4052	[P : 16/P : 32]
ii)	Kinematic viscosity at 40°C, cSt	3.5 - 5.0	ISO 3104	[P : 25]
iii)	Flash point (PMCC)[2] °C, *Min*	101	ISO 2719	[P : 21]
iv)	Sulphur[3], mg/kg, *Max*	10.0	D 5453/ISO 20846/ ISO 20884/ISO 13032	
v)	Carbon residue (Ramsbottom)[4], percent by mass, *Max*	0.05	D 4530 /ISO 10370	
vi)	Sulphated ash, percent by mass, *Max*	0.02	ISO 3987	
vii)	Water content[5], mg/kg, *Max*	500	ISO 3733/ ISO 6296/ISO 12937	[P : 40]
viii)	Total contamination, mg/kg, *Max*	24	EN 12662	
ix)	Copper corrosion, 3 h at 50°C, *Max*	1	ISO 2160	[P : 15]
x)	Cetane No., *Min*	51	ISO 5165	[P : 9]
xi)	Acid value, mg KOH/g, *Max*	0.50	EN14104/ASTM D 974	[P : 1/Sec 1][11]
xii)	Methanol[2], percent by mass, *Max*	0.20	EN 14110	
xiii)	Ester content, percent by mass, *Min*	96.5	EN 14103	
xiv)	Monoglycerides[6] content, percent by mass, *Max*	0.7	D6584/EN14105	
xv)	Diglyceride[6] content, percent by mass, *Max*	0.2	D6584/EN14105	
xvi)	Triglyceride[6] content, percent by mass, *Max*	0.2	D6584/EN14105	
xvii)	Free glycerol[7], percent by mass, *Max*	0.02	D 6584/EN14105/ EN14106	
xviii)	Total glycerol[6], percent by mass, *Max*	0.25	D 6584/EN14105	
xix)	Phosphorous[8], mg/kg, *Max*	4.0	D 4951/EN14107	
xx)	Sodium + Potassium[9], mg/kg, *Max*	5	EN 14108/ EN 14109/ EN14538	
xxi)	Calcium + Magnesium[9], mg/kg, *Max*	5	EN 14538	
xxii)	Iodine value[10], g iodine/100 gm, *Max*	120	EN 14111/EN16300	
xxiii)	Oxidation stability, at 110°C, h, *Min*	8	EN 14112/EN15751	
xxiv)	CFPP, °C *Max*:		EN16329/D6371	[P:110]
	a) summer	18		
	b) winter	6		
xxv)	Linolenic acid methyl ester, percent m/m, *Max*	12	EN 14103	
xxvi)	Polyunsaturated (≥ 4 double bonds) methyl ester percent m/m, *Max*	1	EN 15779	

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[1] In case of dispute ISO 12185 shall be the refree method.

[2] In case flash point is more than 130°C then testing and reporting of methanol content may not be required.

[3] In case of dispute ISO20846 or ISO 20884 shall be the refree method.

[4] Carbon residue shall be run on 100 percent sample

[5] In case of dispute ISO 12937 shall be the refree method

[6] In case of dispute EN14105 shall be the refree method.

[7] In case of dispute EN14105 shall be the refree method.

[8] In case of dispute EN14107 shall be the refree method.

[9] In case of dispute EN14538 shall be the refree method.

[10] In case of dispute EN14111 shall be the refree method

[11] In case of dispute [P:1/Sec 1] shall be the refree method.

ANNEX A

(*Foreword*)

LONG-TERM STORAGE OF BIODIESEL

A-1 PRINCIPLE

This annex provides guidance to consumers of biodiesel (B 100) who may wish to store quantities of fuels for extended periods. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage. This Annex is directed towards biodiesel (B 100) and may be more or less applicable to blends of biodiesel with petro-diesel.

Normally produced biodiesel has adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products, although data suggests some biodiesel may degrade faster than petro-diesel. The selected biodiesel that is to be stored for prolonged periods should be incorporated with suitable concentration of antioxidants to avoid formation of sediment, high acid number, and high viscosities that can clog filters, affect fuel pump operation or plug combustors nozzles or injectors. The selection of biodiesel should result from supplier user discussions.

These suggested practices are general in nature and should not be considered substitutes for any requirement imposed by the warranty of the distillate fuel equipment manufacturers or by federal, state, or local government regulations. Although they cannot replace knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the biodiesel fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when and how fuel quality should be monitored.

A-2 TERMINOLOGY

A-2.1 Bulk Fuel — Fuel in the storage facility in quantities over 50 gallon.

A-2.2 Combustor Fuel — Fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

A-2.3 Fuel Contaminants — Foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

A-2.4 Fuel-Degradation Products — Those materials formed in fuel after it is produced. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (acids and gums) may be more or less volatile than the fuel and may cause an increase in injector and nozzle deposits. The formation of degradation products may be catalyzed by contact with metals, especially those containing copper and, to a less extent, iron.

A-2.5 Long-Term Storage — Storage of fuel for longer than 6 months after it is received by the user.

A-3 FUEL SELECTION

The stability properties of biodiesel are not fully understood and appear to depend on the vegetable oil and animal fat sources, severity of processing, and whether additional production plant treatment has been carried out or stability additives are present.

The composition and stability properties of biodiesel produced at specific production plants may be different. Any special requirement of the user, such as long-time storage, should be discussed with the supplier.

A-4 FUEL ADDITIVES

In order to improve the performance, use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount to help avoid deterioration of drivability and emission control durability. Other technical means with equivalent effect may also be used.

In order to improve the oxidation stability of FAME, it is recommended to add suitable oxidation stability enhancing additives to FAME at the production stage and before storage to obtain maximum benefits.

Biocides or biostats destroy or inhibit the growth of fungi and bacteria, which can grow at fuel water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in the fuel phase or the water phase, or both (*see* ASTM D 6469 for additional information).

A-5 TESTS FOR FUEL QUALITY

Test methods for estimating the long term storage stability of biodiesel (B100) are being developed. Modifications of ASTM D 2274 to use glass fiber filters, varying times and temperatures, and the measurement of pre-test and post-test acid number and viscosity appear promising. However, correlation of this test with actual storage stability is unknown and, may depend upon field conditions and fuel composition.

Performance criteria for accelerated stability tests that ensure satisfactory long-term storage of biodiesel (B100) have not been established.

A-6 FUEL MONITORING

A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful monitoring program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

Stored fuel should be periodically sampled and its quality assessed. ASTM D 4057 provides guidance for sampling. Fuel contaminants and degradation products may settle to the bottom of quiescent tank although detrimental changes to biodiesel can now occur (rising acid value) without causing sediment formation. A bottom or clearance sample, as defined in ASTM D 4057, should be included in the evaluation along with an all level sample.

The quantity of insoluble fuel contaminants present in biodiesel can be determined using ASTM D 6217 with glass fibre filters and abundant washing although no precision or bias testing has been performed with biodiesel using ASTM D 6217.

The acid value of biodiesel appears to exceed its specified maximum before other deleterious fuel property changes occur. A conscientious program of measuring the acid value of biodiesel may be sufficient for monitoring biodiesel stability.

A-7 FUEL STORAGE CONDITIONS

Contamination levels in fuel can be reduced by storing in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water affects the storage life of biodiesel. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface (*see* ASTM D 6469 for additional information). Underground or isothermal storage is preferred to avoid temperature extremes; above ground storage tanks should be sheltered or painted with reflective paints. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel.

Copper and copper-containing alloys should be avoided with biodiesel due to increased sediment and deposit formation. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

ANNEX B

(*Clause* 2)

LIST OF REFERRED INDIAN/INTERNATIONAL STANDARDS

IS No./International Standards	*Title*
1460 : 2005	Automotive diesel fuel — Specification (*fifth revision*)
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling (*first revision*)
1448	Methods of test for petroleum and its products
[P : 1/Sec 1] : 2002	Determination of acid number of petroleum products by potentiometric titration (*second revision*)
[P 9] : 2013/ISO 5165	Cetane number (*first revision*)
[P : 15] : 2004/ ISO 2160	Corrosiveness to copper — Copper strip test (*third revision*)
[P : 16] :1990	Density, relative density or API gravity to crude petroleum and liquid petroleum products by hydrometer method (*third revision*)
[P : 21] : 2012/ ISO 2719	Flash point (closed) by Pensky Martens apparatus (*third revision*)
[P : 25] :1976	Determination of kinematic and dynamic viscosity (*first revision*)
[P : 32] : 1992	Density and relative density (*second revision*)
[P : 40] : 1987	Water by distillation (*third revision*)
[P :110] : 1981	Determination of cold filter plugging point of distillate fuels
ISO 3104	Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity
ISO 3675	Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method

IS No./International Standards	*Title*
ISO 3987 : 2010	Petroleum products — Determination of sulfated ash in lubricating oils and additives
ISO 10370	Petroleum products — Determination of carbon residue — Micro method
ISO 12185	Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method
ISO 13032	Methods of test for petroleum and its products: determination of low concentration of sulphur in automotive fuels — energy-dispersive x-ray fluorescence spectrometric method
ISO 20846	Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method
ISO 20884	Petroleum products — Determination of sulfur content of automotive fuels — Wavelength-dispersive X-ray fluorescence spectrometry
ASTM D 2274	'Standard test method for oxidation stability of distillate fuel oil (accelerated method)
ASTM D 4057	Standard practice for manual sampling of petroleum and petroleum products
ASTM D 4530	Standard test method for determination of carbon residue (Micro method)
ASTM D 4951	Standard test method for determination of additive elements in lubricating oils by inductively coupled plasma atomic emission spectrometry
ASTM D 5453	Standard test method for determination of total sulfur in light hydrocarbons, spark ignition engine fuel, diesel engine fuel, and engine oil by ultraviolet fluorescence
ASTM D 6217	'Standard test method for particulate contamination in middle distillate fuels by laboratory filtration'
ASTM D 6371	Standard test method for cold filter plugging point of diesel and heating fuels
ASTM D 6469	Standard guide for microbial contamination in fuels and fuel systems
ASTM D6584	Standard test method for determination of total monoglycerides, total diglycerides, total triglycerides, and free and total glycerin in B-100 biodiesel methyl esters by gas chromatography
EN 14103	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of ester and linolenic acid methyl ester contents
EN14105	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of free and total glycerol and mono-, di-, triglyceride contents
EN14106	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of free glycerol content
EN14107	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of phosphorous content by inductively coupled plasma (ICP) emission spectrometry
EN 14108	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of sodium content by atomic absorption spectrometry
EN 14109	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of potassium content by atomic absorption spectrometry
EN 14111	Fat and oil derivatives. Fatty acid methyl esters (FAME)- Determination of iodine value
EN 14112	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of oxidation stability (accelerated oxidation test)
EN14538	Fat and oil derivatives. Fatty acid methyl ester (FAME). Determination of Ca, K, Mg and Na content by optical emission spectral analysis with inductively coupled plasma (ICP AES)
EN15751	Automotive fuels. Fatty acid methyl ester (FAME) fuel and blends with diesel fuel. Determination of oxidation stability by accelerated oxidation method
EN 15779	Petroleum products and fat and oil derivates. Fatty acid methyl esters (FAME) for diesel engines. Determination of polyunsaturated (≥ 4 double bonds) fatty acid methyl esters (PUFA) by gas chromatography
EN16300	Automotive fuels. Determination of iodine value in fatty acid methyl esters (FAME). Calculation method from gas chromatographic data
EN16329	Diesel and domestic heating fuels. Determination of cold filter plugging point. Linear cooling bath method

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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Amendments Issued Since Publication

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