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COMPARATIVE STUDY ON BIODEISEL PRODUCTION USING NON-EDIBLE OIL SEEDS.

AN M,SC DISSERTATION REPORT [ANALYTICAL CHEMISTRY]

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Introduction,

Generally, the seeds of the fruits and fruits vegetables and also oil seeds which are non-edible remain unattended and thrown away without any application. There is a practise in our country everywhere. When i had observation over these matter.

Then question arose with respect to what parameters i should take up the analysis of the seeds and how to focus literature survey.

In this regard, i had to make serious and sincere effort to analyse the respective parameters.

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 International (originally known as the 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.

In India oil provides energy for 95% of transportation for which domestic supply of crude will satisfy only about 22% of the demand and the rest will have to be met from imported crude. As a result, the import bill also increases. This adversely affects the economy of country. The cost of the diesel fuel increases due to increase in crude oil price which necessitate taking appropriate policy decisions in the country to fulfil future demand. In view of energy and environmental problems associated with the use of fossil fuels in power generation and for transportation,

Biodiesel may be produced from various sources such as vegetable oil/plant oil both edible as well as non-edible oils. Production of Bio-diesel from edible oil crops is not desirable as there are many concerns regarding the use of food crops as feedstock for fuel production and has created famous debates about food v/s fuel. The high price of biodiesel derived from food grade vegetable oils makes it non-viable to compete economically with fossil - based diesel. Less expensive, non-edible vegetable oil / plant oil as potential feedstock for biodiesel production.

(Aransiola et al., 2012; Humphrey et al., 2017; “Introduction to Biodiesel Production,” 2011; N. Patel et al., 2013)

Literature review,

Non edible seeds,

A fuel produced from natural, renewable sources such as vegetable oil, seeds and fats is the best alternative to present source of energy produced from fossil resource. Initially, the most commonly used oils for the production of Bio-diesel were **soya bean, sunflower, palm, rapeseed, canola, cottonseed and jatropha**. Use of such edible oil for the production of Bio-diesel is unfeasible in India because of a vast gap between demand and supply of such oils. Thus in India only those oils can be utilized for the production of Bio-diesel which comes under the class of the non-edible seeds, which might not compete with edible.

There is a long list of trees, shrubs and herbs available in India, which can be used for extracting oil and producing bio-diesel as fuel from them. There are many plants in India; however there are 77 non-edible Indian plants, which contain 30% or more oil in their seed, fruit or nut. Among all other alternative towards diesel substitution in Indian automobile sector as compared to Compressed Natural Gas (CNG), Liquefied Natural Gas (LNG), Compressed Natural Gas in combination with Hydrogen etc.

Biodiesel based on non-edible oil is best, due to following reasons

• Non-edible oil species, which can grow on wasteland.

• Can be cultivated as agro-forestry crops.

• Hardy plants have superior survivability under drought conditions.

• Yielding of seeds can be obtained over long period

("Introduction to Biodiesel Production," 2011)

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(seeds of the rape plant, used chiefly for the oil,) (mainly in the European Union countries), soybean (Argentina and the United States of America), palm (Asian and Central American countries) and sunflower, although other oils are also used, including peanut, linseed, safflower, used 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 has been studied for several years with good results.

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.

("Introduction to Biodiesel Production," 2011)

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 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. According to the catalyst used, transesterification can be basic, acidic or enzymatic.

Production of Oil and its Methyl Esters from Non-edible Seeds Oil extracted from non-edible seeds cannot be directly used as fuel in automobile engine due to higher viscosities of oils. Moreover problems like injector coking, more engine deposits, ring sticking and thickening of engine lubricant are observed using straight vegetable oil as fuel. There are many ways and procedures to convert seed oil in diesel like fuel but trans-esterification is the one of the best process for production of biodiesel. The new National Biofuel policy of 2008 seeks to explore the full potential for biofuels in India not only from wastelands but also from natural forest and other private forest areas. Fuel properties of oil extracted from different seeds and its methyl ester are compared with standard established by USA – **American Society for Testing and Materials** (ASTM D6751) and German Institute for Standardization **– Deutsches Institut fur Normung** (DIN EN 14214). Different standard fuel properties as per ASTM and DIN of biodiesel are given below along with the standard value of conventional fuel diesel.

(Eduardo Zavala-Gómez et al., 2020; "Introduction to Biodiesel Production," 2011; Roschat et al., 2017; Takase et al., 2021)

Properties of biodiesel as per ASTM and DIN.

PROPERTY	**UNIT**	**DEISEL**	**ASTM(D 6751)**	**DIN (EN 14214)**
Density at 15°C	kg/m3	850	875-900	860-900
Viscosity at 40°C	mm2 /s	2.60	1.9-6.0	3.5-5.0
Flash point	°C	70	>130	>120
Pour point	°C	-20	-	-
Water content	%	0.02	<0.03	<0.05
Ash content	%	0.01	<0.02	<0.02
Carbon residue	%	0.17	-	<0.3
Sulphur content	%	-	0.05	-
Acid value	mg KOH/g	0.35	<0.8	<0.5
Iodine value	-	-	-	-
Saponification value	-	-	-	-
Calorific value	MJ/kg	42	-	-
Cetane number	-	46	-	-

Cloud Point	°C	-	-	-
Induction Period-IP / hours / - / 3.0 min / 6.0 min	µm	-	-	-

(“Introduction to Biodiesel Production,” 2011; N. K. Patel et al., 2013)

What is transesterification reaction?

Transesterification reaction is an organic reaction in which the R group of an alcohol is exchanged with an R’ group of an ester. This is generally done via the introduction of an acid or a base catalyst of a reaction mixture. However it can also be done using an enzymatic catalyst such as lipase. An illustration detailing of an R group belonging to the alcohol R’’ group of an ester is known as transestrtification reaction.

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.

- A **generic transesterification** reaction is shown below. RCOOR indicates an ester, ROH an alcohol, ROH another alcohol (glycerol), RCOOR an ester mixture and a catalyst.

$$RCOOR + ROH \xrightarrow{\text{catalyst}} ROH + RCOOR$$

(Fadhil et al., 2019; “Introduction to Biodiesel Production,” 2011)

Stages of Biodiesel Production Process

1. Treatment of raw materials
2. Alcohol-catalyst mixing
3. Chemical reaction
4. catalyst
5. Separation of the reaction products
6. Purification of the reaction products.

1. Treatment of raw materials

The 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 it reacts with the catalyst to form soaps. In significance, 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 feasible if the content of free fatty acids is less than 2%. In the case of highly acidic raw materials (animal fats from cattle, poultry, vegetable oils from cotton, coconut, most used oils, etc.)

An acid transesterification is necessary as a preliminary stage, to reduce the level of free fatty acids. Besides having low humidity and acid content, it is important that the oil presents in a low level of non-saponificable substances. If it 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. Anyway, 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%.

(Aransiola et al., 2012; “Introduction to Biodiesel Production,” 2011)

2. 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 treatment of raw material. 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 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, is another key variable of the transesterification process. The stoichiometric ratio requires 1 mol of oil to react with 3 mol of alcohol, to obtain 3 mol of fatty acids methyl esters and 1 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 . 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 .

(Aransiola et al., 2012; "Introduction to Biodiesel Production," 2011)

3. Chemical reaction

The chemical reaction takes place when the oil is mixed with the alkoxide (alcohol–catalyst mix) described in alcohol catalyst mixing. 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 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 68C (341 K), the temperature for transesterification at atmospheric pressure is usually in the range between 50 and 60C. 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 (which are palmitic, oleic, 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, etc7.

While doing this process there are some 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:

TG + MOH $\rightarrow$ DG + ME7
DG + MOH $\rightarrow$ MG + ME7
MG + MOH $\rightarrow$ G + ME7
all three reactions in presence of catalyst.
where MOH indicates methanol, ME are the methyl esters, TG, DG and MG are tri-, di- and monoglycerides, respectively, and G is the glycerin.

4. **catalyst**

The catalysts used for the transesterification of triglycerides may be classified as basic, acid or enzymatic.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. 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.

(Aransiola et al., 2012; “Introduction to Biodiesel Production,” 2011)

5. **Separation of the reaction products**

The separation of reaction products is done by decantation:

The mixture of fatty acids methyl esters (FAME) separates from glycerin forming two phases, since they have different densities;

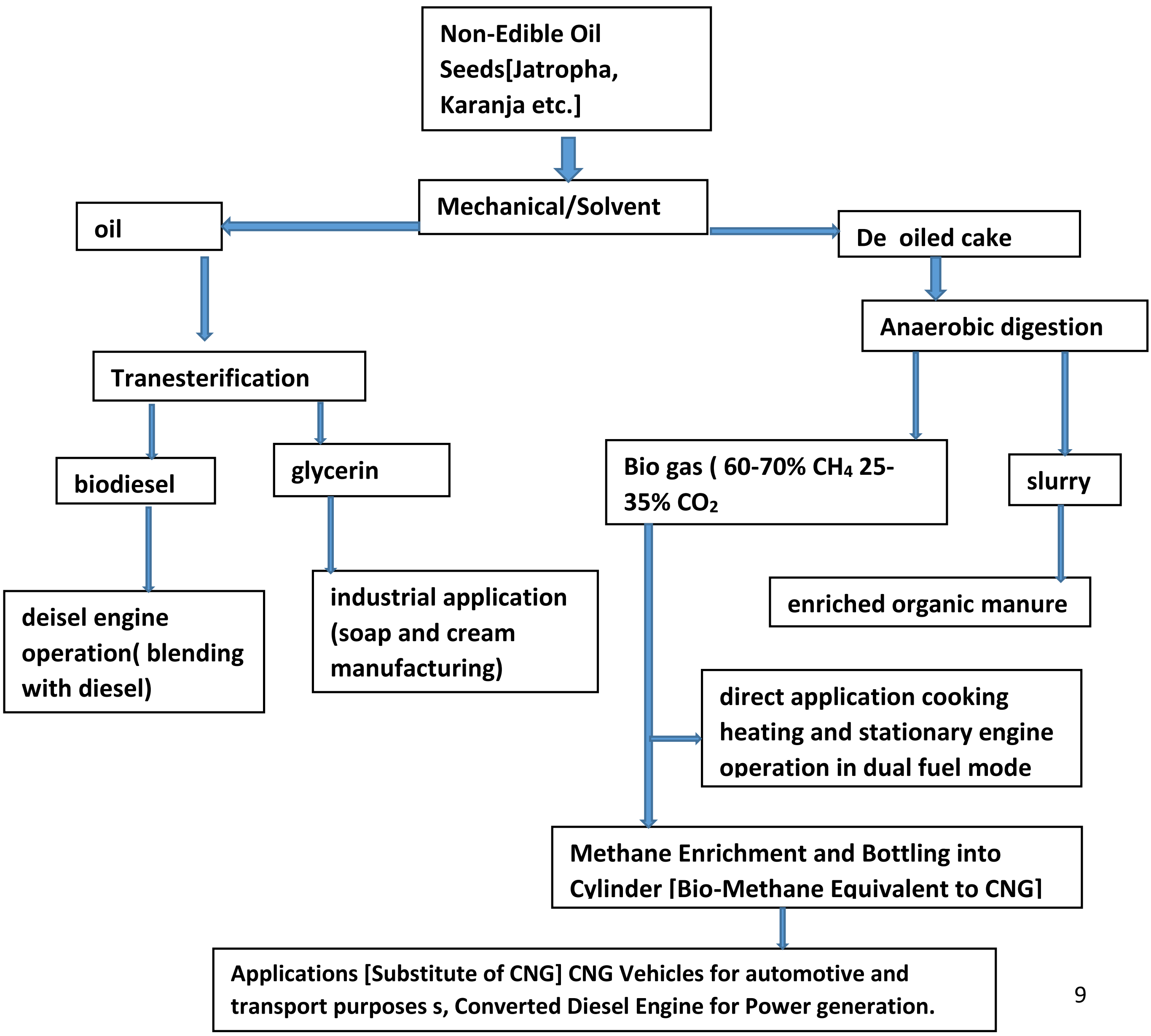
The two phases begin to form immediately after the stirring of the mixture is stopped. Due to their different chemical affinities, most of the catalyst and excess alcohol will concentrate in the lower phase (glycerin), while most of the mono-, di-, and triglycerides will concentrate in the upper phase (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, it is more expensive alternative. After the separation of glycerin, the FAME mixture contains impurities such as residue of alcohol, catalyst and mono-, di-, and triglycerides. These impurities consult 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.

6. **Purification of the Reaction Products**

The mixture of fatty acids methyl esters (FAME) obtained from the transesterification reaction must be purified just 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 are the same for all the production scales (laboratory, pilot plant, Introduction to Biodiesel Production small-, medium-, and large-scale industrial).

(Aransiola et al., 2012; Chauhan et al., 2012; Churchill & Srinivasan, n.d.; "Introduction to Biodiesel Production," 2011)

A possible way of utilization of non-edible oil seed.



(Aransiola et al., 2012; “Introduction to Biodiesel Production,” 2011)

Advantages of the Use of Biodiesel

Some of the advantages of using biodiesel as a replacement for diesel fuel are [1–4]:

- 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 (SO2) emissions.
- Higher flash point (100C minimum)10
- 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.

(Ahmad et al., 2011; “Introduction to Biodiesel Production,” 2011; *The 2 Nd Joint International Conference on "Sustainable Energy and Environment*, n.d.; Islam et al., 2018)

some of the oil seeds and their composition and properties

properties	**Neem Oil**	**Jatropha Oil**
Acid Value (mgKOH/g)	32.538	35.8
Iodine Value	81.28	
Viscosity	30 ^{o} C 43.75	room temp 41.4
Saponification	199.86	193
Physical state at room temperature	Liquid(Golden yellow)	Liquid(Golden yellow)
Cloud point (^{o} C)	13	10
Pour point (^{o} C)	7.0	2
Density at room temperature (Kg/m3)	918.2	895

(Humphrey et al., 2017; Sedeeq et al., 2021)

1. Jathropa oil seed

Most of the researchers used two stage (acid catalyzed and alkaline catalyzed) esterification for biodiesel production from J. curcas oil due to its high free fatty acid content. Ortho-phosphoric acid is used as catalyst and degumming agent in acid esterification stage . Pre-esterification is the first stage that uses sulfuric acid prepared by calcination of acid as a catalyst. The conversion of FFAs was higher than 97% under the reaction conditions of 90C, 2 h, 4% solid acid, and molar ratio of 20:1 of methanol to FFA. Then alkaline catalysis was carried out for 20 min, 64C using 1.3% KOH as catalyst and molar ratio of 6:1 of methanol to oil achieved 90% yield in 2 h alkaline transesterification produced biodiesel and achieved yield up to 98% from jatropha oil with FFA over 20%. Complete conversion and the highest yield was conducted in supercritical methanol within 4 min with temperature at 320C, pressure 8.4 MPa, molar ratio absolute methanol to oil was 43:1 as optimum ratio . Supercritical temperature was above 327C and pressure above 8 MPa since ester yield increase rapidly at that state. From technical and environmental point of view, supercritical temperature is appropriate for biodiesel production due to less glycerol waste but from economic analysis point of view; this method is not appropriate due to its high operating skill and cost. Two stages process can be a good choice, since it can reduce FFA to proper amount below to 1%.11

2. Soybean oil seed

It is being arising 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 glycerine. 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 4,000kg . Since the seeds are very rich in protein, oil content is around 18%.

3. Avocado oil seed

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 12–30%.

4. Jojoba oil seed

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.

5. Oil Palm seed

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 the production and as an additive for butter and bakery products. It is important to remark that pure palm oil is semisolid at room temperature (20–22C), and in many applications is mixed with other vegetable oils, sometimes partially it is being hydrogenated.12

6. Castor oil Seed

The castor oil plant grows in tropical climates, with temperatures in the range 20–30C; it cannot go through the frost. It is important to note that once the seeds starts raw Materials for Biodiesel Production12 germinates, the temperature must not fall below 12C. 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.12

7. 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.

(“Introduction to Biodiesel Production,” 2011)

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:

• 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.

• May degrade plastic and natural rubber gaskets and hoses when used in pure form.

• 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.

(Aransiola et al., 2012; Humphrey et al., 2017; “Introduction to Biodiesel Production,” 2011; N. Patel et al., 2013)

CONCLUSION

In India there are many plant species that cannot be utilised for biodiesel production , bio gas. Non edible oil seeds are the potential feedstock for bio diesel production in India. These oil seeds have been fulfilling various biodiesel standards. Proper process of non-edible oil seeds using transesterification reaction can give the better quality of biodiesel and can be fulfilling the commercial use .The future scope of these non-edible oil seeds is a long living source of feedstock for fuels industry Neem, Karanja and Jatropha yet has different medicinal and many other properties which is useful for us.

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