

Biodiesel Production and Processing Methods: A Review

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Abstract— Bio-diesel is the most valuable form of renewable energy that can be used directly in any existing, unmodified diesel engine. In these paper comparative study of different techniques of Bio-diesel production is done. In These paper the review of transestrification via acid, alkali and enzyme catalyst is carried out as well as modern techniques of transesterification such as methylation with Diazomethane, sodiummethoxide, Borontrifluride, Solid catalytic, Non catalytic conversion and Supercritical Methanolysis is presented. Nine practical processes of biodiesel production are introduced along with the brief review on acid catalysis bio-diesel production. **Keywords:** Closed loop Pulsating Heat Pipes (CLPHPs); working fluids; Nano fluids.

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I. 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 blend formation with diesel.

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, “B10” indicates a blend with 10% biodiesel and 90% diesel fuel; in consequence, B0 indicates pure petro-diesel.

The question of trying to achieve greater energy independence one day through the development of bio-fuels may become possible.

The Advantages of using Bio-Diesel as Follows:-

A. Economic Growth:

Production of Bio-diesel leads to stable economy by creations of new industries, new Technicians, new opportunities and new global markets.

B. Cleaner Air:

Bio-fuels are burn more cleanly than gasoline and diesel. Less emissions of carbon monoxide, particulates, and toxic chemicals and reduce cancerous emissions by 85%.

C. Less Global Warming:

The Fossil fuels are adding huge amounts of stored carbon dioxide(CO₂). Atmosphere, CO₂ traps the heat at outer blanket of earth and leads to global warming. Literature proved that bio-diesel minimizes carbon di-oxide emissions to a considerable extent and in some cases all most nearly to zero[34].

D. When Added To Regular Diesel Fuel In An Amount Equal To 1- 2%, It Can Convert Fuel With Poor

Lubricating Properties, E.G. Ultra-Low-Sulfur Diesel Fuel, Converted To Acceptable Fuel.

E. Bio-Diesel Can Decreces The Dependence On Petro-Fuels To Significant Amount.

The name “biodiesel” has been given to transesterified vegetable oil to describe its use as a Diesel fuel [35]. Biodiesel has significant potential for use as an alternative fuel in compression-ignition engines [36,37]. Biofuels are non-toxic, biodegradable and free of sulfur and carcinogenic compounds [38], as they are obtained from renewable sources. Biodiesel is obtained from plants and it has oxygen in its molecule which making it a cleaner burning fuel than petrol and Diesel [39].

II. TRANSESTERIFICATION PROCESSES

Biodiesel is produced through a process known as transesterification, as shown in the fig.(1). Triglyceride from oil when mixed with alcohol under certain catalyst and stirred vigoursly will produce the Bio-diesel.

Bases catalyze the reaction by removing a proton from the alcohol, thus making it more reactive, while acids can catalyze the reaction by donating a proton to the carbonyl group, thus making it more reactive [1].

The Transesterification reaction uses catalyst such as alkalis [2,3], acids [4], or enzymes [5-8].

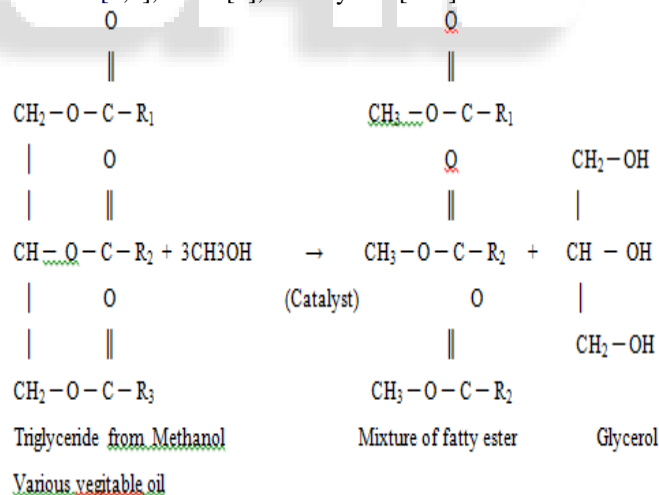


Fig. 1: Transesterification Process

III. VARIOUS PROCESSES OF BIO-DIESEL PRODUCTION ARE AS FOLLOWS

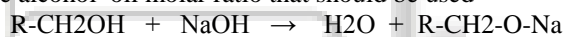
A. Alkali Catalytic Transesterification Method/Base Catalysis Transesterification:

In the alkali catalytic methanol transesterification method, the catalyst (KOH or NaOH) is dissolved in methanol by vigorous stirring in a small reactor. The oil is transferred into the biodiesel reactor, and then, the catalyst/alcohol mixture is pumped into the oil. The final mixture is stirred vigorously for 2 h at 340 K in ambient pressure. A

successful transesterification reaction produces two liquid phases: ester and crude glycerin. Crude glycerin, the heavier liquid, will collect at the bottom after several hours of settling. Phase separation can be observed within 10 min and can be complete within 2 h of settling. Complete settling can take as long as 20 h. After settling is complete, water is added at the rate of 5.5% by volume of the methyl ester of oil and then stirred for 5 min, and the glycerin is allowed to settle again. Washing the ester is a two-step process, which is performed with extreme care. A water wash solution at the rate of 28% by volume of oil and 1 g of tannic acid per liter of water is added to the ester and gently agitated. Air is carefully introduced into the aqueous layer while simultaneously stirring very gently. This process is continued until the ester layer becomes clear. After settling, the aqueous solution is drained, and water alone is added at 28% by volume of oil for the final washing [9, 10, 11].

Sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium methoxide (CH₃ONa) are the most common catalysts for transesterification. Sodium methylate (sodium methoxide) is more effective than NaOH and KOH as a catalyst, but it is more expensive.

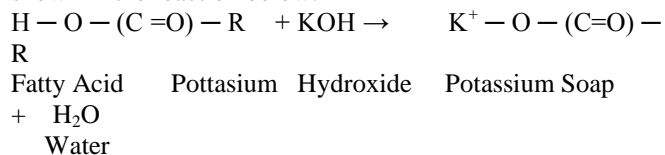
Methanol is the most common alcohol used for conversion of fats and oils to biodiesel. Methanol is flammable, so proper handling is required for safety. In this process it is better to produce the Alcoxy before the reaction to obtain a better global efficiency. The alcoxy reaction is



The alcohol-oil molar ratio that should be used varies from N =1:1–6:1. However N ¼=6:1 is the most used ratio giving an important conversion for the alkali catalyst without using a great amount of alcohol. The amount of catalyst that should be added to the reactor varies from 0.5% to 1% w/w [12,13], but some authors prefer advice any values between 0.005% and 0.35% w/ [14] should be used temperatures will give different degrees of conversion, and for that reason the temperature range should be from 25 to 120 degree [15–17,18–23].

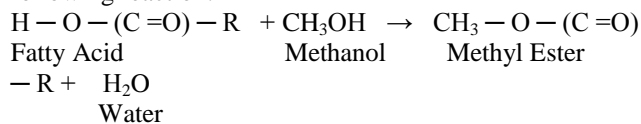
B. Acid Catalytic Transesterification:

Special processes are required if the oil or fat contains significant amounts of free fatty acids (FFAs). Used cooking oils typically contain 2–7% FFAs, and animal fats contain from 5% to 30% FFAs. Some very low quality feedstock's, such as trap grease, can approach 100% FFAs. When an alkali catalyst is added to these feedstocks, the free fatty acids react with the catalyst to form soap and water, as shown in the reaction below:



Up to about 5% FFAs, the reaction can still be catalyzed with an alkali catalyst, but additional catalyst must be added to compensate for the catalyst lost to soap. The soap that is created during the reaction is either removed with the glycerol or is washed out during the water wash. When the FFA level is above 5%, the soap inhibits separation of the methyl esters and glycerol and contributes to emulsion formation during the water wash. For these cases, an acid catalyst, such as sulfuric acid, can be used to

esterify the free fatty acids to methyl esters, as shown in the following reaction:



The use of acid catalysts has been found to be useful for pretreating high free fatty acid feedstock's to convert the FFAs to esters but reaction rates for converting triglycerides to methyl esters are too slow. Acid catalysis also is used for direct esterification of oils with high free fatty acid content or for making esters from soap stock, which is a byproduct of edible oil refining. The esterification of free fatty acids to alcohol esters is relatively fast; it would take about one hour at 60 degrees Celsius to complete the reaction. Water is formed during this reaction. To improve reaction rates, water needs to be removed from the reaction medium by phase separation.

Acid catalysis requires a high alcohol to free fatty acid ratio (20:1 or 40:1 mole ratio) and large amount of catalyst (5-25 percent). Sulfuric acid[27,28,29] and phosphoric acid are the most common acid catalysts. The feedstock is sometimes dried to 0.4 percent water and filtered before the reaction. Then, an acid and methanol mixture is added to the feedstock. Once the conversion of the fatty acids to methyl esters has reached equilibrium, the methanol, water and acid mixture is removed by settling or centrifugation. Fresh methanol and base catalyst are added into the remaining oil for transesterification.

Keim [26] describes using this approach to convert palm oil containing 50.8% free fatty acids to methyl esters. Methanol (77% of the weight of oil) and sulfuric acid (0.75% of the weight of oil) were added to the oil while stirring at 69 C for 1 h. After neutralization, 1.25% sodium methoxide was added, and the mixture was stirred for an additional hour at 50 C. Analysis showed a yield of 97% but a residual acid value equivalent to about 5% palmitic acid. The incomplete reaction was probably due to water in the reactant mixture. As shown in the reaction, water is formed, and if it accumulates, it can stop the reaction well before completion.

Freedman and Pryde [40] get the desirable product with 1 mol% of sulfuric acid with a molar ratio of 30:1 at 65 deegreeC and they get 99% of conversion in 50 h, while the butanolysis will need 117 deegreeC and the ethanolysis 78 deegreeC but the times should be 3 and 18 h, respectively.

Both transesterification and esterification reactions can be operated either as a batch or continuous process. A batch process is better suited to smaller plants that produce less than 1 million gallons per year and provide operation flexibility. Continuous processing allows use of high-volume separation systems, and therefore increases throughput.

C. Enzymatic Conversion:

Enzymes have shown good tolerance for the free fatty acid level of the feedstock, but the enzymes are expensive and unable to provide to meet the ASTM fuel specification [23]. Immobilization of the enzyme and use of multiple enzymes in sequence may provide future opportunities in this area [24-25].

There is interest in using lipases for enzymatic catalysis of oils for biodiesel production. The enzymes can be used in solution or immobilized onto a support material, which allows the use of fixed-bed reactors. The reaction can be performed at 35 to 45 degrees Celsius. However, the reaction is very slow, requiring from four to 40 hours. Because of the high cost of the enzymes, this process is not economically feasible for biodiesel production at this time.

D. Methylation Of Free Fatty Acids With Diazomethane (CH₂N₂):

CH₂N₂ reacts rapidly with free fatty acids to give methyl esters. The CH₂N₂ is generally prepared in ethereal solution by the action of alkali (a 30% solution of KOH) on a nitrosamide, e.g. N-methyl-N-nitroso-p-toluene-sulfonamide or nitroso-methyl-urea [30, 31].

E. Sodium Methoxide Catalyzed Transesterification:

There have been given a number of detailed recipes for sodium methoxide catalyzed transesterification [32]. The methodology can be used on quite a large scale if need be. The reaction between sodium methoxide in methanol and a vegetable oil is very rapid. It has been shown that triglycerides can be completely transesterified in 2–5 min at room temperature. The methoxide anion are prepared by dissolving the clean metals in anhydrous methanol. Sodium methoxide (0.5–2 M) in methanol effects transesterification of triglycerides much more rapidly than other transesterification agents. At equivalent molar concentrations with the same triglyceride samples, potassium methoxide effects complete esterification more quickly than does sodium methoxide. Because of the dangers inherent in handling metallic potassium, which has a very high heat of reaction with methanol, it is preferred to use sodium methoxide in methanol. The reaction is generally slower with alcohols of higher molecular weight. As with acidic catalysis, inert solvents must be added to dissolve the simple lipids before methanolysis will proceed [32]. The reaction between sodium methoxide in methanol and a vegetable oil is very rapid; for example, completely transesterified in 4–6 min at room temperature.

F. Borontrifluoride Catalyzed Transesterification:

The Lewis acid, boron trifluoride, in the form of its coordination complex with methanol is a powerful acidic catalyst for esterification of fatty acids. One of the most popular of all transesterification catalysts is boron trifluoride in methanol (12–14% w/v), and in particular, it is often utilized as a rapid means of esterifying free fatty acids. When it is compared critically with some of the other acidic catalysts under similar conditions, it does not even appear to be any more rapid in its reaction.

G. Solid Catalyst:

The processes discussed above, which also are referred to as homogeneous catalysis, involve utilization of a catalyst that is soluble in alcohol. In these systems the catalyst ends up in the byproducts, and it is not recovered for re-use. There are also solid catalysts that can be used for biodiesel production. This process, which is referred to as heterogeneous catalysis, utilizes fixed-bed reactors, and the catalyst stays in the reactor and is used for an extended time. Sulfonic resins such as Nafion NR50, sulphated zirconia (SZ) and

tungstated zirconia (WZ) have sufficient acid site strength to catalyze biodiesel-forming transesterification reactions as efficiently as sulfuric acid. Alkaline earth metal oxides, various alkaline metal compounds supported on alumina or zeolite can catalyze transesterification reactions. Information about a commercial system that uses a solid catalyst can be found at www.axens.net. In general, heterogeneous catalysis systems are designed for continuous operation and produce high-purity glycerine (greater than 98 percent). The product, fatty acid esters, does not require water washing, and yields are generally high. It also has been reported that catalyst requirements per ton of biodiesel for heterogeneous catalysis are much lower than for other processes. However, these systems operate under high temperature and pressure.

H. Non-Catalytic Conversion Techniques:

Due to poor methanol and oil miscibility, conversion of oil to biodiesel is a very slow reaction. Use of a co-solvent that is soluble in both methanol and oil may improve reaction rates. The BIOX Process (www.bioxcorp.com) uses either tetrahydrofuran (THF) or MTBE-methyl tert-butyl ether as a co-solvent to generate a one-phase system. In the presence of a co-solvent, the reaction is 95 percent complete in 10 minutes at ambient temperatures and does not require a catalyst. THF has a boiling point very close to that of methanol. The excess methanol and co-solvent are recovered in a single step after the reaction is complete. Co-solvents that are subject to the hazardous and/or toxic air Environmental Protection Agency (EPA) list for air pollutants must be completely removed from the biodiesel and its byproducts (glycerine and methanol). Emissions must be tightly controlled, and processing equipment must be "leak proof."

The second non-catalytic approach utilizes methanol at very high temperature and pressure (350 to 400 degrees Celsius and greater than 80 atm or 1200 psi) to convert oil to biodiesel. This process requires a high alcohol to oil ratio (42:1 mole ratio). The reaction is complete in about three to five minutes. The process requires high pressure vessels which can be quite expensive. The energy consumption also is higher than the conventional processes. The reaction must be quenched very rapidly so the products do not decompose.

Transesterification The non-catalytic supercritical methanol transesterification is performed in a stainless steel cylindrical reactor (autoclave) at 520 K [33]. In a typical run, the autoclave is charged with a given amount of vegetable oil and liquid methanol with changed molar ratios. After each run, the gas is vented, and the autoclave content is poured into a collecting vessel. All the rest of the contents are removed from the autoclave by washing with methanol. The most important variables affecting the methyl ester yield during the transesterification reaction are the molar ratio of alcohol to vegetable oil and the reaction temperature. Viscosities of the methyl esters from vegetable oils were slightly higher than that of No. 2 Diesel fuel.

In the transesterification process, the vegetable oil should have an acid value less than 1, and all materials should be substantially anhydrous. If the acid value were greater than 1, more NaOH or KOH would be spent to

neutralize the free fatty acids. Water also causes soap formation and frothing.

The stoichiometric ratio for the transesterification reaction requires three moles of alcohol and one mole of triglyceride to yield three moles of fatty acid ester and one mole of glycerol. Higher molar ratios result in greater ester production in a shorter time. The vegetable oils were transesterified 1:6–1:40 vegetable oil-alcohol molar ratios in catalytic and supercritical alcohol conditions [33].

To be economically viable and to compete commercially with petro-diesel, a continuous process for biodiesel production must be few in reaction steps and simple in separation processes, and obviously robust heterogeneous catalysts have to be employed.

Heterogeneous catalysts are categorized as solid acid and solid base. Solid base catalysts include a wide group of compounds in the category of alkaline earth metal oxides, hydrotalcites/layered double hydroxides, alumina loaded with various compounds, zeolites, and various other compounds showing high basicity coupled with active basic sites, pore size, and other parameters. Solid base catalysts have been quite successful with high conversion and yield of biodiesel obtained (Sharma and Singh, 2011) and can be recycled several times without regeneration (Science-Article.com 2009). However, the solid base catalysts are sensitive to the presence of free fatty acids (Sharma and Singh, 2011). Solid acids have a preference over solid base catalysts for they can tolerate free fatty acids. The heterogeneous catalysts have the advantages of easy separation from the product without requirement of washing and reusability of the catalysts (Sharma and Singh, 2011). Soybean Oil The following alkaline earth metal oxides have been used as catalysts for transesterification of soybean oil; CaO, SrO, Ca(OCH₃)₂ and Ca(OCH₂CH₃)₂. The results indicated that at a reaction temperature of 65°C, reaction time of 3 hours and mole ratio of 12:1 of methanol to oil, 95% biodiesel yield was obtained as reported by Science-Article.com (2009). The yield has little decrease after reused for 10-20 times. Among the four, Science-Article.com (2009) reported that, Ca(OCH₃)₂ has higher stability and better catalytic ability. A 95% yield of biodiesel at 65 degreeC over. Solid acids have a preference over solid base catalysts for they can tolerate free fatty acids. The heterogeneous catalysts have the advantages of easy separation from the product without requirement of washing and reusability of the catalysts (Sharma and Singh, 2011).

I. Catalytic Supercritical Methanol Transesterification:

Catalytic supercritical transesterification is performed in the autoclave in the presence of 1–5% NaOH as catalyst at 520 K. In the catalytic supercritical-methanol transesterification method, the yield of conversion rises to 60–90% for the first 1 min.

IV. CONCLUSIONS

Conventional processing involves an alkali catalyzed process, but this method found to be unsatisfactory for lower cost high free fatty acid feedstocks due to soap formation. the cost associated with using enzyme catalyst is high. non catalytic and super critical methanolysis require large set-up and extreme operation conditions which required extreme

engineering. to be economically viable and to compete commercially with petro-diesel, a continuous process for biodiesel production must be few in reaction steps and simple in separation processes, and obviously robust heterogeneous catalysts have to be employed. butanolysis and ethanolysis with acid catalyst show less reaction time. in future, pretreatment processes using strong acid catalysts may be proven to provide good conversion yields and high-quality final products. the acid catalysis is inviting method for producing bio-diesel. The Effectivity of Nitric Acid, Hydrochloric Acid And Sulfuric Acid is Future Scope For Production of Biodiesel.

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