

Biodiesel: A Future fuel (A Review)

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Abstract:

Due to the concern on the availability of recoverable fossil fuel reserves and the environmental problems caused by the use those fossil fuels, considerable attention has been given to biodiesel production as an alternative to petro diesel. The general way to produce biodiesel fuel is by Transesterification of vegetable oil with methanol in the presence of either alkaline or strong acid catalysts. Transesterification reaction is quite sensitive to various parameters. An ideal Transesterification reaction differs on the basis of variables such as fatty acid composition and the free fatty acid content of the oil. The benefits of biodiesel go far beyond the clean burning nature of the product. Bio diesel is a renewable resource helping to reduce the dependence of the economy on limited resources and imports, create a market for farmers and reduce the amount of waste oil, fat and grease being dumped into landfills and sewers. This paper reviews the production and use of Biodiesel fuel as an alternative fuel for existing IC engines.

Keywords: *biodiesel, Transesterification, economy, IC engines, fuel.*

1. Introduction

Biodiesel has become more attractive recently because of its environmental benefits and the fact that it is made from renewable resources. Biofuels can be produced across the world using resources that are available locally, thereby reducing foreign exchange for buying crude oil and increases the country's economy. Fuel is any material that stores energy that can later be extracted to perform mechanical work in a controlled manner. This may include combustion, exothermic chemical reactions and nuclear reactions [1]. Vegetable oil from plant sources is the best starting material to produce biodiesel because the conversion of pure triglyceride to fatty acid methyl ester is high and the reaction time is relatively short. The most common way to produce biodiesel is by transesterification, which refers to a catalyzed chemical reaction involving

vegetable oil and an alcohol to yield fatty acid alkyl esters and glycerol [2].

Biodiesel is a mixture of mono-alkyl esters obtained from vegetable oils like soy bean oil, jatropha oil, rapeseed oil, palm oil, sunflower oil, corn oil, peanut oil, canola oil and cottonseed oil (3 Ma, F., dan Hanna[3]. In the conventional transesterification process, Sunflower seed oils, methanol and NaOH in various concentrations) were refluxed together in a 500 ml glass reactor equipped with a glass anchor shaped mechanical stirrer, a water condenser and funnel. After the complete conversion of the vegetable oil, the reaction was stopped and the mixture was allowed to stand for phase separation: the ester mixture formed the upper layer and glycerine formed the lower layer[4]. The main advantages of using biodiesel is that it is biodegradable, can be used without modifying existing engines, and produces less harmful gas emissions such as sulfur oxide. Biodiesel reduces net carbon-dioxide emissions by 78% on a lifecycle basis when compared to conventional diesel fuel[5].

2. Synthesis of Biodiesel

2.1 Direct Use and Blending

The direct use of vegetable oils in diesel engine is not favorable and problematic because it has many inherent failings. Even though the vegetable oils have familiar properties as biodiesel fuel, it required some chemical modification before can be used into the engine. It has only been researched extensively for the past couple of decades, but has been experimented with for almost hundred years. Although some diesel engine can run pure vegetable oils, turbocharged direct injection engine such as trucks are prone to many problems. Energy consumption with the use of pure vegetable oils was found to be similar to that of diesel fuel. For short term use, ratio of 1:10 to 2:10 oil to diesel has been found to be successful [6].

2.2 Micro Emulsion Process

The problem of the high viscosity of vegetable oils was solved by micro-emulsions with solvents such as methanol, ethanol, and 1-butanol. Micro-emulsion is defined as a colloidal equilibrium dispersion of

optically isotropic fluid microstructures with dimensions generally in the 1-150 nm range formed spontaneously from two normally immiscible liquids and one or more ionic or non-ionic amphiphiles [7].

2.3 Thermal Cracking (Pyrolysis)

Pyrolysis can be defined as the conversion of one substance into another by means of heat in the absence of air (or oxygen) or by heat in the presence of a catalyst which result in cleavage of bonds and formation of a variety of small molecules. The pyrolysis of vegetable oil to produce biofuels has been studied and found to produce alkanes, alkenes, alkadienes, aromatics and carboxylic acids in various proportions. Another disadvantage of pyrolysis is the need for separate distillation equipment for separation of the various fractions. Also the product obtained is similar to gasoline containing sulphur which makes it less ecofriendly [8] Pyrolytic chemistry is difficult to characterize because of the variety of reaction path and the variety of reaction products that may be obtained from the reaction occur. The pyrolyzed material can be vegetable oils, animal fats, natural fatty acids and methyl esters of fatty acids. The first pyrolysis of vegetable oil was conducted in an attempt to synthesize petroleum from vegetable [9].

2.4 Mixing of Alcohol and Catalyst

This typical process is mainly done by mixing alkali hydroxide (commonly potassium hydroxide and sodium hydroxide) with common alcohols (methanol and ethanol) in the mixer with standard agitator to facilitate the mixing. Alkali hydroxide is dissolved in the alcohol to produce alkoxide solution [10].

3. Production of Biodiesel from vegetable oil

There are four basic routes to biodiesel production from oils and fats.

1. Base catalyzed transesterification
 2. Direct acid catalyzed transesterification
 3. Conversion of oil into its fatty acids and then into biodiesel
 4. Non - catalytic transesterification of oils and fats [11]
- Transesterification consists of a number of consecutive, reversible reactions. Triglycerides are converted step wise to diglycerides, monoglycerides and finally glycerol liberating a mole of ester in each step. The reactions are reversible, the equilibrium lies towards the production of fatty acid ester and glycerol .

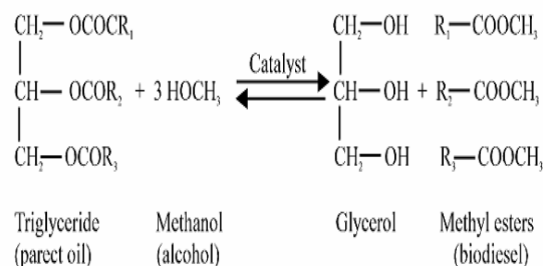


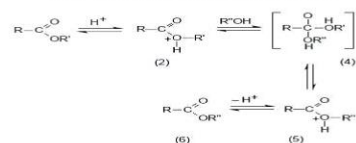
Figure 1. : The mechanism decomposition of triglycerides [11]

3.1 Acid catalyzed transesterification

Carboxylic acids can be esterified by alcohols in the presence of suitable acidic catalyst in water free Conditions. The most frequently cited reagent for the preparation of methyl esters is 5% anhydrous hydrogen chloride in methanol. In a typical esterification procedure, using methanolic hydrogen chloride, the lipid sample is dissolved in at least a 100-fold excess of the reagent and the solution is refluxed for about two hours or is held at 50° C overnight.



Scheme 1. Acid-catalysed esterification of fatty acids.



Scheme 2. Acid-catalysed transesterification of lipids.



Scheme 3. Preparation of methanolic hydrogen chloride via acetyl chloride.

Figure 2. : Mechanism of Acid catalyzed transesterification [12]

Free fatty acids in the oil were reduced by using acid catalyst. Free fatty acids reduce the yield of biodiesel by saponification, hence acid catalyzed transesterification has an advantage of more yield. However, the reaction is slow that one run can take more than a day [17].

3.2 Base catalyzed Transesterification

Esters in the presence of base such as an alcoholate anion form an anionic intermediate which can dissolve back to the original ester or form the new ester. The most useful basic transesterifying agents are sodium or potassium methoxide in anhydrous methanol [18]. The main advantage of base catalyzed transesterification over acid one is that it is fast and can be conducted at low temperatures (303-308K) and pressure (0.1 MPa) [13].

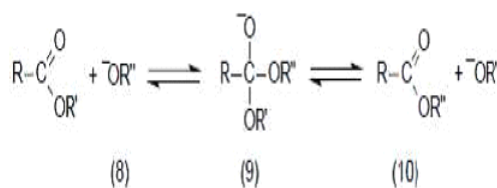


Figure 3.: Base catalyzed Transesterification of lipids [13]

4. Types of Transesterification:

4.1 Homogeneous base catalyzed transesterification

Homogeneous alkaline catalysts are more preferable and commonly used since Transesterification reaction using its acid counterpart has slower rate [17]. The most common basic catalysts are potassium hydroxide (KOH), potassium methoxide (KOCH₃), sodium hydroxide (NaOH), sodium methoxide (NaOCH₃), and sodium ethoxide (NaOCH₂CH₃). These catalysts are commonly used because of several advantages such as able to catalyze reaction at low reaction temperature and atmospheric pressure, high conversion in shorter time, and economically available [18]. Sodium methoxide (NaOCH₃) and potassium methoxide (KOCH₃) are better catalyst than sodium hydroxide (NaOH) and potassium hydroxide (KOH) due to the ability to dissociate into CH₃O⁻ and Na⁺ and CH₃O⁻ and K⁺ respectively when comparing on biodiesel yield [19].

4.2 Homogeneous acid-catalyzed Transesterification

Waste oils contain free fatty acids which cannot be converted to biodiesel using an alkaline catalyst. These FFAs will produce soap that inhibit the separation of the ester, glycerin, and wash water when react with an alkaline catalyst [21]. Hence, liquid acid-catalyzed Transesterification is proposed in order to overcome lots of conundrum caused by liquid base catalysts. Sulfuric acid, sulfonic acid, hydrochloric acid, organic sulfonic acid, and ferric sulphate are most commonly acids used as catalysts in transesterification. In the production of biodiesel, hydrochloric acid and sulfuric acid are favoured as catalyst. Despite of its insensitivity to FFA in the feedstock and can catalyzes esterification and transesterification simultaneously, acid catalyst has been less popular in transesterification reaction because it has relatively slower reaction rate. Thus, alcohol to oil molar ratio is the main factor influencing the reaction. Addition of excess alcohol can speeds up the reaction and favours the formation biodiesel product. The steps involve during acid-catalyzed transesterification are initial protonation of

the acid to give an oxonium ion followed by the oxonium ion and an alcohol undergo exchange reaction to give the intermediate which later loses a proton to become an ester [22]. In a study of acid-catalyzed transesterification of sunflower oil using HCl reported 95.2% of biodiesel yield with 100°C reaction temperature and 1.85 wt% catalyst loading. used H₂SO₄ in the acid-catalyzed transesterification with 0.5 wt% catalyst loading reported 92.5% biodiesel yield from *Chlorella pyrenoidosa*.

4.3 Acid - base transesterification

In the homogenous acid and base catalyzed transesterification processes, the most commonly used base catalysts are NaOH and KOH while acid catalysts are sulphuric acid and sulphonic acid. The major drawback of base transesterification is saponification which may be due to the presence of free fatty acids in the oil that is subjected for transesterification. Esterification of free fatty acids is possible by acid catalysis which reduces the soap formation, ultimately leads to more yield of alkyl ester (biodiesel). In transesterification process, biodiesel is produced from a reaction between vegetable oil, alcohol in the presence of base or acid catalyst. Methanol is most preferred alcohol because of its low cost and its physical and chemical properties. The highest biodiesel yield was at 3% weight of catalyst, 60° C and reaction time of 120 minutes. The results of acid -base catalyzed synthesis showed that biodiesel yield was improved when compared to base catalyzed synthesis alone. The kinematic viscosity of acid -base catalyzed biodiesel satisfied the standard test method for kinematic viscosity [22]

4.4 Heterogeneous case catalyzed transesterification

Numbers of research have been conducted on heterogeneous catalysts to overcome the problems caused by homogeneous catalyst in biodiesel production. Most of the heterogeneous catalysts developed for production of biodiesel are either alkaline oxide or alkaline earth metal oxide supported over large surface area. Heterogeneous basic catalysts are more active than heterogeneous acid catalyst, similar to their homogeneous counterparts [23]. In addition, solid alkaline catalysts, for instance, calcium oxide (CaO) provide many advantages such as higher activity, long catalyst life times, and could run in moderate reaction condition [24]. Nonetheless, CaO as catalyst can also slow down the reaction rate of biodiesel production [25]. Notwithstanding, reaction of CaO with glycerol can leach out calcium diglyceroxide during transesterification which result in necessity of an extra purification step such as ion exchange resin to remove the soluble content in the biodiesel both

reported transesterification by using eggshells as catalyst which is the closest comparison to this study. [22]. Furthermore, base-catalyzed transesterification was proved to have higher reaction rate as compared to acid-catalyzed transesterification which need more vigorous reaction condition including higher temperature during transesterification [26]. The effect of base catalyst, Potassium hydroxide (KOH) and volume of methanol on the rate of transesterification of croton oil was investigated by separately varying the mass of base or volume of methanol added to different portions the reaction mixture containing 100 g croton oil and determining their effects on the % yield of biodiesel relative to the mass of croton oil. The KOH was dissolved in methanol then added to the reaction mixture in a biodiesel reactor at constant temperature and stirred continuously at 350 rpm. The effects of temperature and volume of methanol on the rate of transesterification were also investigated by separately varying the temperature and volume of methanol and determining their effects on the % yield of biodiesel.

4.5 Heterogeneous acid-catalyzed transesterification:

Despite of the effectiveness of homogeneous acid catalyst, it can lead to absolute contamination problems which require good separation and product purification processes [11]. This will be resulted in higher production cost. Some of advantages of heterogeneous acid catalyst are insensitive to FFA content, can simultaneously conduct esterification and transesterification, eliminate the washing step of biodiesel, simpler separation process of catalyst from product, regenerating and reusing the catalyst is possible and also reduce the corrosion problems. In addition, solid acid catalysts are preferred over liquid acid catalysts due the fact that they contain a multiple sites with different strength of Bronsted or Lewis acidity [27]. Bronsted acid catalysts have the advantage of promoting simultaneous esterification and transesterification with the use of cheaper feedstock containing high concentration of free fatty acids [28]. A Lewis acid site is more active as compared to Bronsted but is at risk to poisoning from water and/or free fatty acids. The volume of methanol required to react completely with FFA in croton oil was calculated from the acid value. Since the esterification reaction is a reversible reaction, a FFA:methanol ratio of 1:3 was used to shift the equilibrium to the right and improve the yield of ester. Exactly 2mL of 99% pure methanol was added to different portions of 100 g of croton oil in a reactor and mixed for 5 minutes by stirring at 350 rpm.

4.6 Enzyme (Biocatalyst) catalyzed transesterification:

Enzymatic transesterification has drawn researcher's attention due to the downstream processing problem posed by chemical transesterification. Huge amount of wastewater generation and difficulty in glycerol recovery are some of the problems that eventually increase the overall production cost of biodiesel and being not environmental benign. In contrast, enzyme catalysis occurs without the generation of by-products, easy recovery product, mild reaction condition, insensitive to high FFA oil and catalyst can be reuse. However, enzyme catalyzed biodiesel production has some limitations especially when implemented in industrial scale because of high cost of enzyme, slow reaction rate and enzyme deactivation [29]. alcoholysis, esterification and transesterification they have a wide spectrum of biotechnological applications. Lipases are also highly specific as chemo-, regio- and enantioselective catalysts. Thanks to direct evolution and protein engineering it is possible to enhance catalytic potential of lipases and to 'tailor' them to specific application and process conditions, enabling further expansion of their industrial use. Among lipases of plant, animal and microbial origins, most commonly used are microbial lipases. In addition, the enzymatic yield is independent of potential seasonal variations and it is possible to achieve rapid growth of microorganism in low-cost culture media [30].

4.7 Microwave assisted transesterification

In conventional transesterification process heating of material takes place by conduction. Whereas microwaves heat by means of thermal radiation and it is very efficient mode of heat transfer. In microwave assisted continuous transesterification, vegetable oil and methanolic NaOH solution were fed separately via 2 pumps and mixed at the tee connector at the inlet of modified house hold microwave oven (800W). Microwave energy is delivered directly to the reacting molecules which undergo chemical reaction. Reaction time was reduced to 60 seconds with palm oil, 5 to 10 minutes in case of non-edible oils of Honge and Jatropa where their respective conversion in conventional method was 1 hour and 1 to 2 hours. Biodiesel produced by microwave assisted transesterification has reduced performance due to their comparatively high viscosity. Also smoke opacity, HC and CO emissions increased with such oils [31].

4.8 Ultra sonically assisted transesterification

Transesterification is a chemical reaction between triglycerides (oil /fats) and methanol, which form immiscible phases when they are in a reaction vessel, the reaction takes place at contact surface

between oil and methanol. Ultrasonic reactor drastically reduces the amount of catalyst, molar ratio of oil to methanol 1:6, reaction time, reaction temperature, mass transfer resistance elimination and energy input for production of biodiesel [32]. Transesterification reaction takes place at interfacial region of two immiscible phases (oil and methanol), which may be achieved by vigorous mixing using ultra sonic waves and produces pure fuel with highest possible conversion percentage. The reaction rate constants were evaluated and found that rate constants are 3-5 times higher than that of mechanical agitation. Acoustic and hydrodynamic cavitation found effective tool for intensification of esterification of fatty acids with acid catalyst (H_2SO_4) 99.4 % conversion soybean oil to FAME (fatty acid methyl ester) was achieved in about 15 minutes at 40 °C and molar ratio of 6:1 methanol to oil with 20kHz ultrasonic frequency. Lipase catalyzed transesterification activity enhanced with ultrasonic irradiation. also ultrasonic assisted transesterification reduced amounts of lipase and reaction time. Yields of biodiesel were always higher when ultrasound was used [33]

5. Results

5.1 Sulfuric Acid Process (Two-Step Transesterification)

Acid esterification reaction was studied for four different molar ratios. The sulfuric acid catalyst amount was varied in the range of 0.3% to 2%. These percentages are based on volume of the oil used for the acid esterification reaction. The catalyst amount also affects the yield of process is shown in Figure 1. The acid-catalyst process attained maximum yield for waste cooking oil at 0.5% catalyst concentration. It was observed that the yield started to decline when the catalyst concentration was increased to above 0.5%. The yield remains the moreover same with further increase in the methanol to oil molar ratio. The excess methanol in the ester layer can be removed by distillation. Canakci and Van Gerpan [34] advocate the use of large excess quantities of methanol (15:1-35:1) while using the sulphuric acid as catalyst. Figure 2 shows the methanol to oil molar ratio effect in alkali transesterification (Step 2). [37]. An alkali catalyst was studied in the range of 0.3% to 2.5% using KOH as an alkali catalyst. The influence of the alkali catalyst amount on the yield is shown in Figure 3. The maximum yield was achieved for waste cooking oil at 2% of catalyst loading. During the experiments, it was also observed that transesterification could not take place properly with an insufficient amount of an alkali catalyst loading. The production yield slightly decreased above 2% of catalyst loading. At higher concentrations, the intensification of mass transfer became more important than increasing the amount of catalyst.

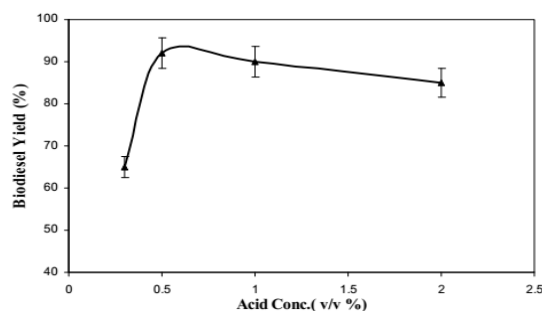


Figure 5: Effect of acid concentration on biodiesel yield (Step 1) in sulfuric acid process.

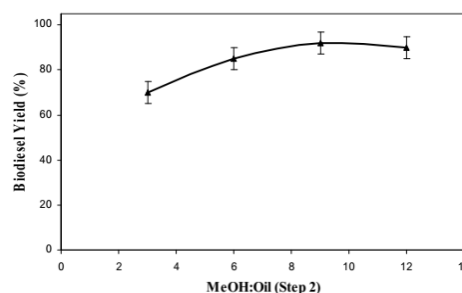


Figure 5: Effect of molar ratio on biodiesel yield (Step 2) in sulfuric acid process.

5.2 Microwave Catalytic Transesterification

The effect of methanol to oil molar ratio and catalyst concentration (BaO and KOH) on waste biodiesel yield using microwave method. Methanol-to-oil ratios of 6:1, 9:1, 12:1, and 15:1 were tested for both catalysts. In this study, for homogeneous catalysts (KOH), a molar ratio of 9:1 and 2% catalyst were found to be effective with maximum biodiesel yield of 92%. For BaO, excess methanol started to interfere in the separation of glycerin due to an increase in the solubility and resulted in lower biodiesel yield [27]. In combine effect of excess alcohol and KOH catalyst leads to saponification resulting in lower biodiesel yield and lower biodiesel quality. An advantage associated with heterogeneous catalysts is that they can be recovered and reused several times. The reactivity of BaO catalyst was found to be quite different than KOH catalyst as it posses different catalytic activity, basicity, leach- ing tendency, and specific surface area, which influence the transesterification of oil [38]

5.3 Effect of mass of base catalyst on transesterification

The % yield of biodiesel initially increased with increase in mass of base catalyst until a maximum yield was obtained at a base concentration of 1 g (1% w/w of croton oil). An increase in formation of emulsion was observed at lower catalyst concentration which necessitated the need for extra washing. At higher concentrations (>1.0%), both soap and emulsion formation occurred and a lower

yield of biodiesel was obtained. Figure 2 shows the effect of mass of KOH added on the % yield of biodiesel[36]

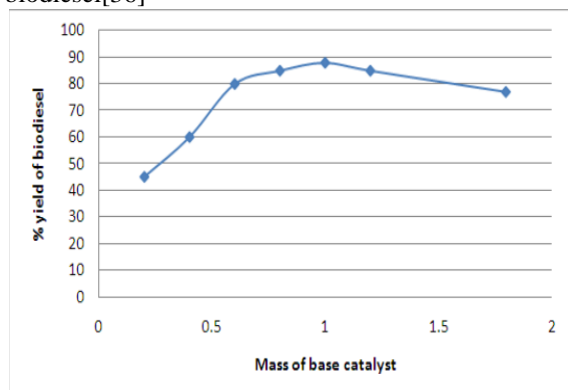


Figure 6: Effect of temperature and time on Esterification

The % yield of biodiesel increased with increase in reaction temperature. The maximum yield of biodiesel was obtained after one hour at a reaction temperature of 60°C. Although the maximum yield of biodiesel increased with increase reaction time at 30°C, 40°C and 50°C, no significant increase in maximum yield of biodiesel was observed at the reaction temperature of 60°C after one hour. Figure 4 shows the variation of yield of croton biodiesel with time at various reaction temperatures.[38]

6. DISCUSSIONS

6.1 Flash Point and Alcohol Control

The volatility of biodiesel is extremely low, and properly produced biodiesel presents minimal risk for handling and safety. To ensure safe handling of biodiesel, a minimum flash point of 130°C is set to limit residual methanol. The flash points of the samples are shown in Figure 1, with only four samples having a flash point below 130°C. Certain feed stocks and process conditions may result in slightly lower flash points, and the methanol content of these samples needed to be tested. The samples with flash points below 130°C were sent for methanol analysis. Three of the four samples with a flash point below 130°C met the methanol content requirement. The final sample had excess methanol content and failed both requirements. [39]

6.2 Acid Number

The acid number of biodiesel should be low to ensure no residual free fatty acids or processing acids are present in the fuel. The presence of excess acids can lead to corrosion and deposits in fuel systems. Previous surveys have shown few failures on acid value [38,39], and the current survey shows no failures for acid number. The average acid number of these samples was 0.24 mgKOH/g, approximately half of the specification limit. One sample was close

to the specification limit, at 0.48 mgKOH/g and was the highest value recorded in this survey.

6.3 Cold Soak Filtration

Previously, biodiesel users experienced many cold weather operability problems with biodiesel blends [40]. In an effort to combat the impact of minor components not measured in D6751, the CSFT was added as an annex in 2008. The annex was converted into a full ASTM test method, D7501, in 2010. Reports of problems in cold weather have been significantly reduced since the addition of the CSFT. Immediately after the adoption of the CSFT into D6751, over 50 biodiesel samples from different feedstocks and processes were tested, with the results being bimodal; the biodiesel samples either passed or failed the test [41]. Failure of the CSFT was not correlated to biodiesel feedstock.

6.4 Oxidation

Stability Because biodiesel contains polyunsaturated fatty acid chains, it will degrade by the well-known peroxidation mechanism [42]. A large body of work has been conducted on the oxidation and stabilization of biodiesel with synthetic and natural antioxidants [43,44]. An oxidation stability limit was adopted in D6751 in early 2007. The 2007 survey showed a wide variety of oxidation stability levels in B100, with 30% of samples collected failing the specification, although this represented only 8.5 million gallons of the nearly 500 million gallons in the market at that time. The current study shows a significant improvement in meeting the oxidation stability limit. Only two samples had oxidation stability results below the 3-hr limit. Two samples were very close to the 3-hr limit but met the limit based on the method variability, as the error bars are over 3 hours. On average, the median value for both the producer samples and terminal samples is around 9.5 hrs.

6.5 Cloud Point

CP is not limited in D6751 but is an important operability parameter that must be reported. The CP is the temperature where the fuel first begins to form crystals and is used to predict cold weather operability. For biodiesel, the CP is particularly important because biodiesel is almost always blended with petroleum diesel, and the CP of the biodiesel will have a significant impact on the final blend properties. CP is determined by the FAME profile and SMG content of the biodiesel, with higher levels of saturated FAME and SMG increasing CP [45] The average CP was around 2°C, although several samples had higher CPs [7].

7. CONCLUSION

Biofuels are renewable fuels which are produced by living organisms by carbon fixation. These fuels do not influence climatic adverses like global warming that are caused by conventional, non-renewable fossil fuels. With exception of hydropower and nuclear energy, the major part of all energy consumed worldwide comes from petroleum, charcoal and natural gas. However, these sources are limited, and will be exhausted in the near future. Thus, Biodiesel is an alternative and renewable fuel for diesel engines and has become more attractive in recent times. The catalysts used in the production of biodiesel are acids, bases and enzymes. Transesterification is a commonly employed method to reduce the viscosity during the production of biodiesel. The purpose of this method is to reduce the viscosity of oil or fat using acid or base catalyst in the presence of methanol or ethanol. However, the biodiesel production by transesterification is strongly affected by molar ratio of alcohol, reaction temperature, reaction time and catalyst concentration. The main advantage in biodiesel usage is attributed to lesser exhaust emissions in terms of carbon monoxide, hydrocarbons and particulate matter. Biodiesel is a good alternative fuel for diesel engines because it is environmentally friendly and renewable in nature. Biomass is the least expensive and most globally available resource. Therefore, priority should be shifted towards utilizing biomass, leaving aside food for human consumption.

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