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# The Design of Vegetable Oil Biodiesel Reactor

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***Abstract:*** ***It has since been realized that sources of fossil fuel are finite and that the use of fossil fuel is degrading the environment. These have prompted the search for alternative energy sources especially those that are either carbon neutral or have significantly reduced carbon footprint. It has to be accepted that it is the aggregate of all these alternatives that will replace parts of the fossil fuels. This can then explain why studies are ongoing on various alternative energy areas. Most transesterification work found in the literature appear to deal with laboratory scale studies. For a difference therefore, the purpose of this study is to design a scalable biodiesel reactor for the transesterification of vegetable oil. Most data available in this area are proprietary and not available in the open literature. Stainless steel has been selected for the design so as to cater for both base and acid catalised processes and the reactor has been designed. Methods for determining the extra catalyst required to neutralize the free fatty acid in restaurant waste oil and testing for completion of reaction have been outlined. Precaution in handling the chemicals used in the transesterification process is recommended.***

**Keywords: transesterification, methoxide, renewable energy, biofuel, glycerol, triglyceride.**

## 1. Introduction

The search for alternative energy sources is prompted not only by the realization that fossil fuel sources are finite but also by the fact that fossil fuels are degrading the environment. The burning of fossil fuel is associated with the release and growth of greenhouse gases and particulates. The alternative energy sources of interest embrace those that are either carbon neutral or the net release to the environment is significantly lower per kilowatt compared with equivalent kilowatt generated from fossil fuel sources. These alternative energy sources include solar, wind turbine, tidal waves, hydro and biofuel among others. Unfortunately so far, none of the alternatives is in a strong enough position to replace, on its own, a significant fraction of the fossil fuels. It appears that it has to be accepted that no one alternative energy source can fill the gap rather it is the aggregate of all the alternatives that will make a significant contribution to replacing the fossil fuel energy sources. This can explain, therefore why studies are taking place on many fronts to find alternative energy sources Various attempts to use vegetable oil neat in compression ignition engines have not been very successful. Most of the vegetable oils available possess properties that render them unsuitable for use neat in these engines. Their viscosity and volatility are some of the primary properties to be considered. Most vegetable oils are very viscous and these can clog fuel flow systems, burn poorly, release hydrocarbon particulates to the environment and leave carbon deposits in the cylinder. The volatile component in the fuel has to be significant for the injected fuel spray to be fully atomized within the restricted space of the engine cylinder. The chemical composition of most of the vegetable oils gives an insight why it is impossible to use them neat in diesel engines. Lots of these oils consist of three long chain fatty acids on a base of glycerin. These long chain fatty acids need to be broken down to simpler components so as to make them suitable for use in diesel engines and this is what transesterification is all about. It has been found that biofuel is cleaner and less polluting than fossil fuels and as a result a lot of studies are on-going to better understand the kinetics, and improve the quality and quantity of yield in transesterification processes. In a laboratory scale study, methyl ester was produced by the transesterification of palm oil in the presence of the base, potassium hydroxide (KOH) as the catalyst [1]. In this work the rate of transesterification was found to increase with temperature up to 60$^{o}$C beyond this temperature the rate remained constant. Another study was based on the kinetics of alkaline catalysed transesterification of palm oil. In this work efforts were focused on parameters such as oil/alcohol ratio, catalyst concentration and temperature to find that both sodium hydroxide and sodium methoxide have high kinetic constant an indication of fast formation of palmoil methyl ester and conversion was found to be above 99% [2]. Sodium hydroxide/Aluminium oxide has been used as the catalyst to study the dependence of palm oil conversion to methyl ester on certain variables. These variables include methanol/oil ratio, catalyst used, operation temperature and conversion time In this study 99% of conversion is claimed to have been achieved under optimum condition [3]. Higher production cost when compared with petroleum diesel, has been recognized as the downside in the transesterification process of vegetable oils. The study however acknowledged that biodiesel is “green fuel” with several advantages including biodegradability and being non toxic [4]. There are possible techniques that can be used to reduce the high production cost. Optimisation of the various parameters involved in the transesterification process may go a long way to reducing costs. The study has recommended one possible way of improving production process which is the use of heterogeneous catalyst. This is interesting because it goes to confirm the findings of Taufiq-Yap et al. A study has been carried out to look at how some parameters influence the transesterification of palm oil to find that the type of catalyst, oil/methanol ratio, temperature and stirring speed do affect residence time and yield.. The study found that sodium, sodium hydroxide and potassium hydroxide are effective catalysts for the transesterification process of palm oil but doubts whether potassium hydroxide (KOH) would be many users choice on ground of cost (see table 1) [5]. Up to 4% water in methanol makes no much difference on transesterification rate and this can help to cut down production cost since by implication excess drying of recovered methanol is not necessary (see table2). The study found that minimum amount of methanol required for total conversion to be 233% at a methanol/oil ratio of 10/1. The study noted that the use of too much catalyst would lead to solidification of the reaction mixture as a result of soap formation. A methanol/oil ratio of 10/1 as indicated in this study appears suspect, a methanol/oil ratio of 1/10 would appear much more realistic. In a review work on transesterification of vegetable oil, it has been noted that industry favours the use of base catalyst rather than acid catalyst [6]. The study went on to explain that acid catalyst is very corrosive and as a result could attack parts of the reactor material. Work on direct transesterification of all classes of lipids in one step reaction has been undertaken to find that the process of transesterification of various lipids could be accelerated by the use of methanol benzene 4:1 with acetyl chloride [7]. Of all the attempts to use neat vegetable oil in diesel engines, this study has recorded a measure of success. In the work, a mixture of cottonseed oil and orange peel oil was used. The study found that a mixture of cottonseed oil with 15% blend of orange peel oil performed satisfactorily [8]. In a study of the oxidation stability of Jatropha curcas biodiesel it was found that a blend with 20% petro-diesel results in improved oxidation stability but that less than 20% petro-diesel would require the use of antioxidant for stable oxidation [9].

In this study ethanol instead of methanol was employed in the transesterification of palm kernel oil to find that the optimal conditions for the production of ethyl esters from crude palm kernel oil are 1:5 mass ratio of ethanol to oil, 1 % catalyst concentration by weight of oil, 90 minutes reaction time at a temperature of 30ºC [10]. Most of the work on transesterification of vegetable oil have been carried out on laboratory scale where this has been scaled up, the work is not often found in the open literature as the findings are proprietary. As a result, the purpose of this work is to design a scalable vegetable oil transesterication reactor capable of accepting biodiesel production processes using either base or acid catalysts.

Table 1: Effect of Catalyst on Transesterification of Palm Oil (Source [5])

| Catalyst | Amount (wt % based on oil) | Reaction Time min | Remarks |
|---|---|---|---|
| Na | 0.1 | 16 – 32 | 99% yield |
| NaOH | 0.2 | 16 – 32 | 98% yield |
| KOH | 1 | 16 – 32 | 98% yield |
| H2SO4 (con) | 1 | >300 | 50% yield |
| HCl (con) | 1 | >300 | 30% yield |
| Ion exchange resin (H+) | 2 | >300 | Too low |
| Dowex (Na+) | 1 | >300 | Too low |
| Acid treated Florisil | 2 | >300 | Too low |
| Activated Silica gel | 1 | >300 | Not suitable |

Table 2: Effect of Water in the Methanol on Transesterification (Source [5])

| Amount of Water % of Oil | Triglyceride % unreated after 7 min | Completion Time (min) |
|---|---|---|
| 0 | Trace | 7 |
| 1 | Trace | 7 |
| 2 | 5 | 10 |
| 3 | 5 | 10 |
| 4 | 5 | 10 |
| 5 | 10 | 20 |
| 10 | 30 | 150 |

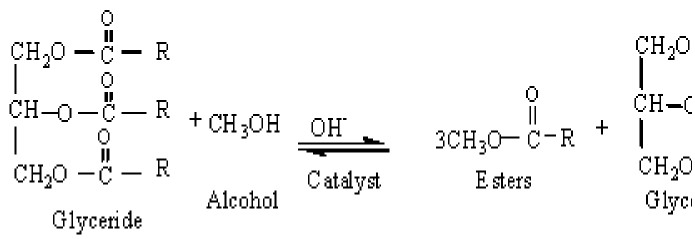
## 2. Chemistry - Theory

A basic understanding of the chemistry of reaction of vegetable oil /animal fat with alcohol is essential in the production of biodiesel..

Mono alkyl methyl esters (biodiesel ) is produced by reacting vegetable oil or animal fat with an alcohol in the presence of an alkaline catalyst. In this process, glycerin is produced as a by-product i.e.

Triglyceride + methanol = mono-alkyl methyl ester + glycerin.

An alkaline catalyst in the form of sodium hydroxide or potassium hydroxide is most often used. If ethanol is used as the alcohol then potassium hydroxide has to be the catalyst for reasonable biodiesel yield. This process can also be stated in chemical symbol as follows:




Source (ESRU [11])

Typical transesterification reaction of vegetable oil can be represented as shown

Fatty acid, a type of organic acid contains the carboxyl group (COOH) and, although not a strong acid, reacts with strong bases to produce salt called soap. The length of the carbon chain and the type of bond determine the physical characteristics of the fatty acid. Alcohols contain carbon plus one or more of the hydroxyl group (OH). The carboxyl group (COOH) of the carboxylic acid plus the hydroxyl group (OH) of the alcohols combine to produce ester.

Simple alcohol includes methanol ($CH_3OH$) and ethanol ($C_2H_5OH$). Glycerol ($C_3H_8O_3$) is an alcohol but more complex as the chemical formula shows. The alcohols relevant for biodiesel production are strongly polar and mix well with water and are good solvent of salts.

Esters are the result of bonding an organic acid with alcohol. Both vegetable oil and biodiesel contain esters and as a result the process of making biodiesel is often referred to as transesterification. Vegetable oils are esters that contain 3 fatty acids bonded to a backbone of glycerol.

Transesterification: vegetable oil + 3methanol = 3methyl ester + glycerol. Basically the glycerol is exchanged with methanol which can only bond singly with one fatty acid instead of three. This breaks up the large vegetable oil molecule into 3 smaller methyl esters which are of similar energy content as the triglyceride but less viscous and has a lower melting point. Because the reaction is reversible, it may be difficult to encourage reaction to completion. For reasonable reaction rate therefore, 100% excess methanol may be needed and the process heated to about 65°C. In the presence of a catalyst, methanol reacts to form a methoxide which then reacts with the triglyceride to form methyl ester and diglyceride anions. The catalyst may be regenerated.

The reaction of a base catalyst with methanol is shown below:

$CH_3OH + NaOH = CH_3O^- + Na^+ + H_2O$

Methanol + Sodium Hydroxide = methoxide anion + sodium cation + Water

Glycerol is very dense and quite polar and tends to carry catalyst, excess methanol, soap and water with it during separation. The biodiesel also has a little bit of all these in it especially if there was a lot of soap. Washing with water can be used to remove these but can be problematic due to emulsion formation. Soap makes it difficult to wash without emulsification but softened water may help as well as acid but acid leaves free fatty acid (FFA) in the fuel. There is however no substitute for high quality biodiesel of low water and low FFA and this can be achieved by washing.

## 3. Material and Equipment

Materials needed for the transesterification process of vegetable oil include alchohol, alkaline, vegetable oil (neat or waste) gloves, overall, water, pipette, syringe etc. The reactor is schematically shown in fig. 1. It consists of two main stainless steel vessels. The main vessel by the right contains the vegetable oil. The operator has the choice to raise the temperature of the vegetable oil in this vessel using steam jacket or raise the temperature in a different vessel and transfer the oil to the reactor vessel. The reactor carries a glass tube for indicating the separation level between the glycerol and biodiesel during the settling stage. The smaller vessel by the left is also made of stainless steel and its function is for mixing the alcohol and catalyst. Protective clothes, hand gloves, goggles etc are also required to protect personnel during the operation. There should also be available water and containers for mixing.

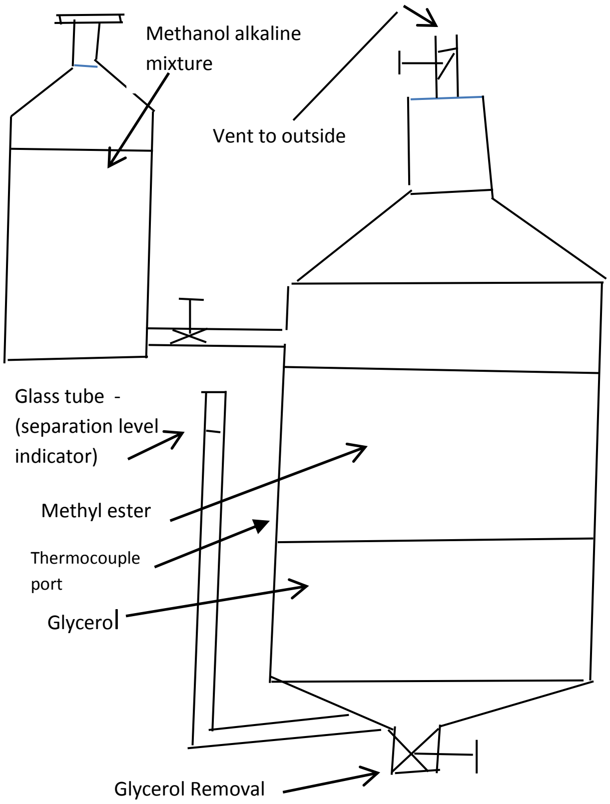



Fig. 1 Vegetable Oil Transesterification Reactor(schematic)

## 4. Method and Result

Fig. 2 shows a typical processing sequence in transesterification to produce biodiesel. This figure covers additionally material recovery after the biodiesel production.

Basically, measured quantity of the alcohol and sodium hydroxide, the catalyst are mixed in the smaller reactor vessel of fig. 1. The vegetable oil which has been preheated to about 60°C is in the bigger reactor vessel.

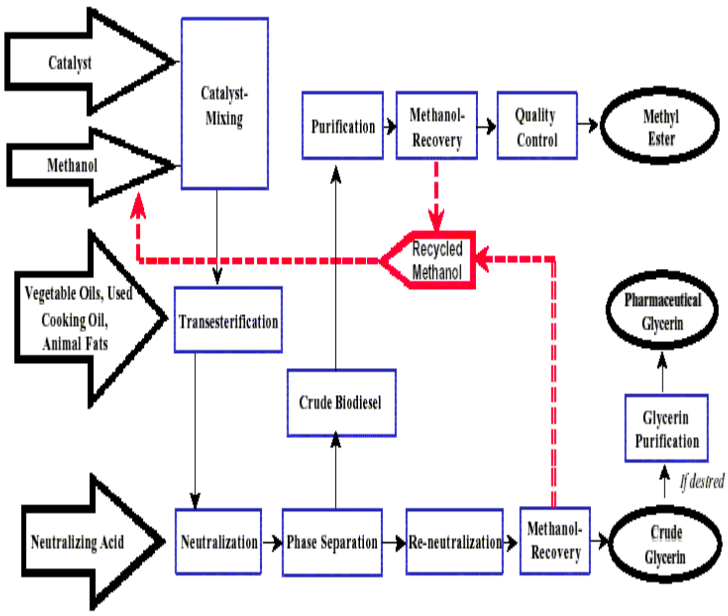



Fig. 2 Typical TransesterificationProcessing Sequence (source ESRU [11])

Opening the valve on the vessel containing the mixture of alcohol and catalyst, feeds the mixture slowly to the vegetable oil vessel. It may be necessary to open the valve on top of the big vessel to vent fumes to the outside especially if the operation is carried out in a confined space. There may be need to stir or agitate the vessel to aid mixing. The reaction takes place and the mixture is allowed to separate with the biodiesel on top and the denser glycerol at the bottom. When the separation is complete, the glycerol is run out by opening the valve at the bottom of the big reactor vessel.

### 4.1 Titration for Free Fatty Acid (PPA)

Some vegetable oil feedstock especially the waste oil from restaurants contain free fatty acids (FFA) and to produce biodiesel by transesterification from this class of feedstock, it will be necessary to neutralise the free fatty acid first and this is achieved by titration. In the titration, the extra catalyst required to neutralise the FFA is determined and this extra is added to the normal quantity required to catalise the transesterification process. Basically the alkaline first reacts with the FFA to produce soap before reacting with the alcohol to produce biodiesel.

For the titration, it is necessary to make the reference alkaline solution first. The titration is carried out by reacting a small measured quantity of FFA in the waste oil with a known quantity of the alkaline. The Ph number, usually 8.5, is used to tell when the FFA has all been neutralised..

One possible way to carry out the test is to mix 1g of the alkaline catalyst in 1 litre of distilled water. In a beaker, add about 10 ml of isopropyl alcohol using a syringe or a pipette. In the same beaker add 1ml of the waste oil whose PPA is to be neutralised. Finally, start adding in small bits, the catalyst/distilled water mixture and stirring (swirling).Keep a record of the amount of mixture added. Continue until when swirled, the oil reactants mixture turns pink and stop at this stage. The millilitres of the catalyst/distilled water mixture used per ml of oil translates to grams of catalyst per litre of waste oil.

### 4.2 Testing for Reaction Completion

Testing for completion of reaction can be made using the "27 – 3" method. If reaction is not complete rerun using additional catalyst say about 10% to drive the reaction to completion.. If the biodiesel fails the 27-3 test, it will not pass the ASTM test. In this test 3ml of biodiesel is used with 27ml of methanol. The test fails if traces of triglyceride are precipitated. Triglyceride is not as soluble in methanol as the diglyceride and the monoglyceride.

## 5. Discussion

In order to avoid the wrath of the moralist group, it is pertinent to inform readers outright that feedstock for this work is sourced from kitchen vegetable oil waste and from other non edible vegetable oil sources such as jatropha and rapeseed. The moralist argue that it is not proper to divert food crop to biofuel feedstock just to satisfy the lust of the few vocal and wealthy minority to the detriment of the poor and hungary silent majority. Kitchen oil waste may be delivered with debris therefore screening and sieving may be necessary. Kitchen oil waste may also contain a lot of water which could reduce biodiesel yield so there is need to heat to drive the excess water away. Oil waste containing water, when heated makes exploding popping noisy sound and the noise goes down as the water content reduces.

Operation with biodiesel blends could lead to slightly less power output from an engine due to lower energy content per unit mass. The cloud point (temperature at which components begin to precipitate) for the biodiesel depends on the feedstock, it ranges from 0ºC for Soy biodiesel to 25ºC for coconut biodiesel. These compare with an average of -24ºC for petro-diesel. Biodiesel will "gel" before petroleum diesel and in the presence of water, is vulnerable to bacteria attach. The biodiesel produced should meet the ASTM standards not only to guarantee engine warranty but also to prevent engine damage. There may be need to test for completion of reaction and this could be achieved by the use of the 27-3 test method. The resulting by product, glycerol may be used in soap making.

Biodiesel is a biodegradable renewable energy and many consider it to be carbon neutral arguing that the live plant absorbs the same quantity of carbon dioxide ($CO_2$) released when the fuel is burnt. This statement is not really strictly true because the carbon required to produce the biodiesel and transport it to the destination of use has to be accounted for. It has to be accepted however that biodiesel is cleaner fuel than petroleum diesel. Biodiesel is virtually free of sulphur and aromatics and these translate to reduced SOx, COx, unburnt hydrocarbons and particulates. Biodiesel is regarded as a "green fuel" and it is non-toxic and safe to use and is produced from vegetable oils, animal fat and waste oil from the food industry using methanol. Biodiesel from waste oil should be encouraged and supported because of the recycling potential.

Biodiesel possesses self-lubricating properties that help to keep the fuel system clean. It can be handled and stored like the petroleum diesel. Biodiesel has a higher flash point and therefore safer than petrol-diesel. Minimum flash point of biodiesel is about 130ºC compared with a minimum of 52ºC for petrol diesel. Biodiesel blend however, will "gel" sooner than petrol diesel blends at low temperatures. Outdoor, above ground storage tanks are potentially vulnerable to low temperature problems The fuel stability may be reduced if water is present. Splash blending of biodiesel with petroleum diesel will not work well at temperatures below the biodiesel gel point (typically -3 to 0°C) but this can be handled by blending in kerosene as done with ultra low sulphur diesel (ULSD). Bacon grease and other solid greases are not suitable for biodiesel production since they raise the cloud point and could cause cold weather problems. Oils from restaurants tend to be overused compared with domestic used oils from house wives, therefore extra treatment may be needed if reasonable biodiesel yield is to be expected if restaurant sourced.

### 5.1 Personnel Safety

In making Biodiesel a number of dangerous chemicals are involved and one of these is methanol a highly inflammable poison that can cause blindness and death and can be absorbed through the skin and inhaled as fumes. The other chemicals include caustic soda, caustic potash, and methoxide. In order to prevent inhaling caustic dust particles, it is advisable to wear dust mask when mixing the alkaline and methanol in the formation of methoxide. The wearing of chemical rated splash proof goggles are recommended to protect the eyes when making biodiesel. Most respirators will not stop methanol fumes and most gloves would late methanol seep through. If methanol splashes on the apron or gloves remove and replace them immediately. If caustic soda or methoxide accidentally splashes on the skin, douse with vinegar and wash with water. Ensure the vent on the main reactor tank is fully open and vented to the outside when mixing the methoxide and oil. An additional safety measure includes the insertion of a non-return valve on the methoxide pipeline feeding the main tank because under certain conditions, the oil-methoxide mixture may boil over and start sending the mixture back to the smaller reactor tank. Reflux pipe may be adequate in some cases but in violent cases both the check valve and vent may be needed as well.

## 6. Conclusion

A biodiesel reactor has been designed

In order to cater for both base and acid catalised reactor stainless steel construction has been adopted

As a means of reducing cost and materials, the design has chosen the gravity feed method for the methoxide feed to the main tank

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