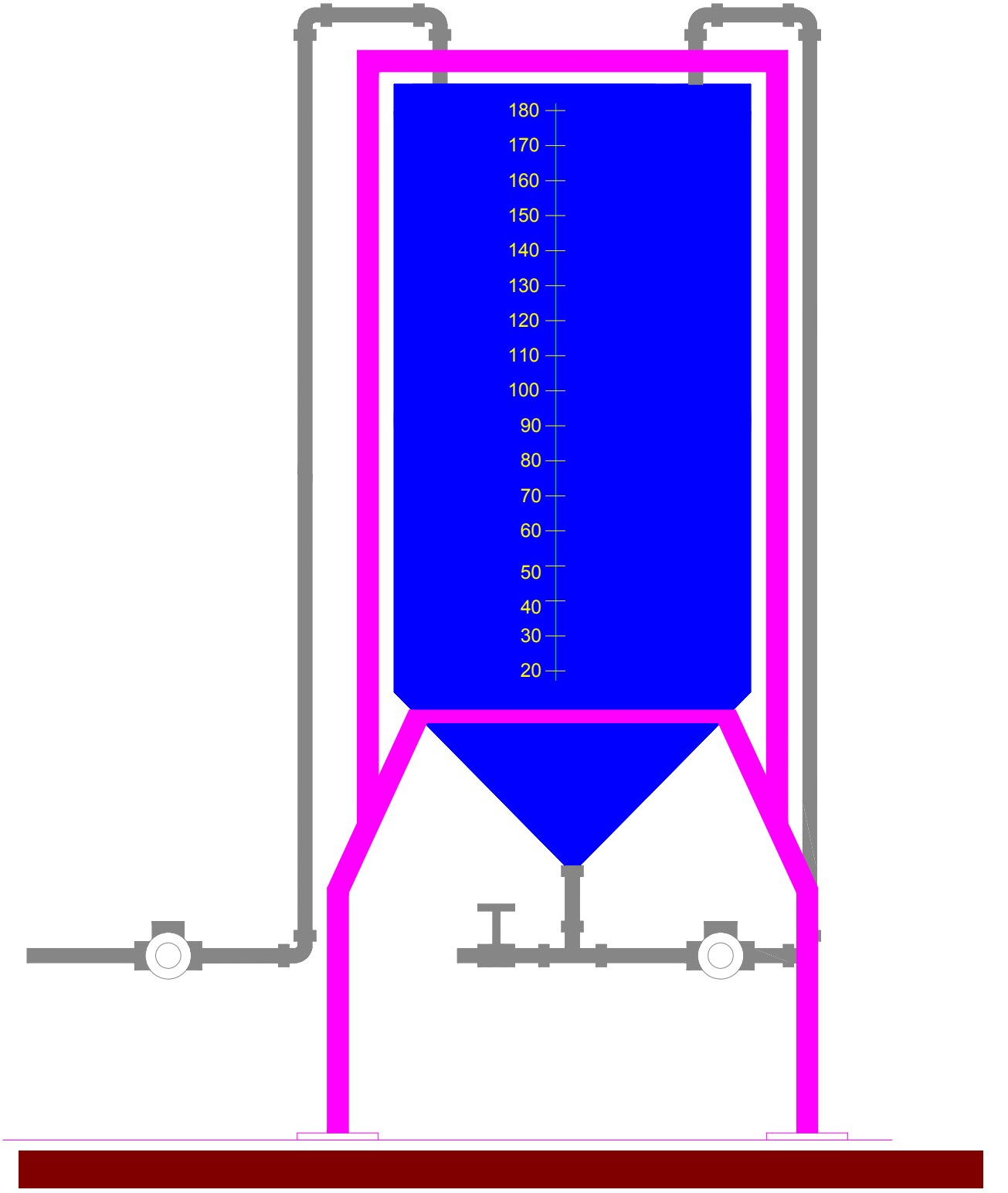
Making Biodiesel at Home



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MAKING BIODIESEL AT HOME

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INTRODUCTION

The utilization of biodiesel, as an alternative fuel is actively being promoted around the world. Biodiesel is a clean-burning fuel made from domestic, renewable sources, such as oils from vegetables, peanuts, soy beans, canola/rape seeds, hemp seeds and some grains. It can also be made from animal fats and waste vegetable oil from restaurants. It has undergone the process of transesterification, a simple chemical modification of ordinary vegetable oil that makes the fuel usable in diesel engines.

Using a food crop for fuel sets up competition between food and fuel. With biodiesel sharing feed stock with food, food prices could rise further. To address this problem non food grade vegetable feed stocks are being considered to make biodiesel. However, farmers can switch from producing food crops to producing biofuel crops to make more money, even if the new crops are not edible. From the law of supply and demand, if less farmers are producing food the price of food will rise.

Biodiesel and feedstock oils produced in Asia, South America and Africa are currently less expensive than those produced in Europe and North America suggesting that imports to these wealthier nations are likely to increase in the future. If deforestation, and large-scale farming techniques were used to grow biofuel crops, biodiesel is predicted to become a serious threat to the environment.

Waste vegetable oil which is the main focus of this book offers several advantages. As a waste product, its main route is to the wastewaster treatment plants where it requires additional resources to convert it to an environment–friendly waste matter. Utilizing waste vegetable oil as an alternative fuel will not only clean our environment but also push us to be more fossil fuel independent. Producing biodiesel from waste vegetable oil does not pose a treat to the food industry.

Diesel engines can run on three basic types of fuel: petroleum diesel, biodiesel, and straight vegetable oil (SVO). Diesel fuel produces carbon dioxide, pollution, particulates and sulfur emissions.

The purpose of this book is to provide a step by step learning process so that biodiesel processing can become simple enough for any ordinary person to do it. Sure enough, there are a lot of materials in the internet where we can learn from but we don’t know which one gives the best result. This book has done most of the hard work and is giving you a proven and ready to use method.

Biodiesel is non-toxic and safe to use. But the ingredients used in making it are hazardous. For this reason, safety concerns are inserted in many parts of this book.

This is not meant to scare you. Nobody has yet burned or killed themselves or anyone else making homebrewed biodiesel. Large numbers of ordinary people all over the world are making their own biodiesel, and so far there have been no serious accidents. It's safe if you're careful and sensible. Food could even be more dangerous. A lot of people died every year due to high blood pressure which can be linked directly to eating habit. If you always use your common sense in curbing your eating habit you could avoid it. The same thing is true with biodiesel home brewing. So don’t be scared. Making biodiesel is safe if you're careful and sensible. Nothing about life is safe if you're not careful and sensible.

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GLOSSARY

Acid
a classification of substances that liberate hydrogen ions in water, and are normally sour and corrosive, with a pH lower than 7. A compound or atom that donates protons.

Alcohol
a large classification of organic compounds containing one or more hydroxyl groups attached to carbon atoms.

Alkali
a classification of substances that liberate hydroxide ions in water, to form caustic and corrosive solutions which turn litmus paper blue, with a pH higher than 7. A compound that reacts with or neutralises hydrogen ions.

Anhyrdous
without water, dry. Transesterification of biodiesel must be an anhydrous process. Water in the vegetable oil causes either no reaction or cloudy biodiesel, and water in lye or methanol renders it less useful or even useless, depending on how much water is present.

Aromatic
any organic compound containing de-localised electrons in a ring structure - e.g. benzene, benzoic acid.

ASTM
American Society for Testing and Materials - an international voluntary standards organization that develops and produces technical standards for materials, products, systems and services.

Azeotrope

a liquid mixture that maintains a constant boiling point and that produces a vapour of the same composition as the mixture.

Base
a classification of substances which when combined with an acid will form a salt plus water, usually producing hydroxide ions when dissolved.

Biodiesel
"Biofuel" - An environmentally safe, low polluting fuel for most diesel internal combustion and turbine engines. Can be mixed with petroleum fuel and stored anywhere petroleum is. Made from fresh or waste vegetable oils (triglycerides). Both commercially and privately made around the world.

Bubble Wash
a method of final washing of biodiesel through air agitation. Water is added into biodiesel. Bubbles are injected into the water causing the bubbles to rise. Bubbles carry water up through the biodiesel by surface tension. Simple diffusion causes water soluble impurities in the biodiesel to be extracted into the water. As the bubble reaches the surface and breaks, the water is freed and percolates back down through the biodiesel again extracting more impurities.

Canola
see “rape seed oil”.

Catalyst
a substance which without itself undergoing any permanent chemical change, facilitates or enables a reaction between other substances.

Cetane Number
measure of fuel ignition characteristics. Like the octane number used for gasoline, the higher the value, the better the fuel performance. A higher cetane number correlates with improved combustion, improved cold starting, reduced noise, white smoke, HC, CO and particulate emissions particularly during early warm-up phase. Typical Cetane number is 40 to 57.

Cloud point
the temperature at which the first wax crystals appear and a standardised ASTM test protocol is used to determine this temperature.

Emulsion
a usually unstable dispersion of two liquids which do not normally mix. Emulsions can be formed either by mechanical agitation, or by chemical processes. Unstable emulsions will separate over time or temperature, stable emulsions will not separate.

Ester
a classification of organic compounds occurring naturally as oils and fats, produced by replacing the hydrogen of an acid by an alkyl, aryl, radical. Any of a large group of organic compounds formed when an acid and alcohol is mixed. Biodiesel is often described as a Fatty Acid Methyl Ester (FAME).

Ethanol (C2H6O or CH3-CH2-OH)
an organic alcohol also called ethyl alcohol, formed when fermenting sugars or glycerine.
Ethanol is also a great solvent. Can be used in place of methanol in making biodiesel.

Exothermic Reaction
in chemistry, a chemical reactions which produces heat.

FAME
Fatty Acid Methyl Ester, a commonly used chemical term for biodiesel.

Fat
a classification of natural esters of glycerol, and fatty acids existing as solids at room temperature.

Fatty Acid
a carboxylic acid (or organic acid), often with a long aliphatic tail (long chains), either saturated or unsaturated.

Flash Point
the lowest temperature at which it can form an ignitable mix with air.

Free Fatty Acids (FFA)
fatty acid hydrocarbon chains detached from other molecules, like glycerol.

Glycerine
a byproduct of biodiesel production.

Hydrocarbon
a compound of hydrogen and carbon, often occurring as long atomic chains in which each carbon atom is attached to two hydrogen atoms forming a long chain. They store a great deal of energy.

Hygroscopic
the tendency of something to absorb water (usually from humidity in the air). Biodiesel absorbs water to about 1200 parts per million (PPM). Methanol and NaOH are also hygroscopic.

Indicator
a substance which changes colour at a given stage in or as a result of a chemical reaction. Phenolphthalein is commonly used in titrations measuring the acidity of vegetable.

Life-cycle analysis
a total valuation of a process, in which all the inputs and outcomes of a reaction are fully considered.

Lipid
a classification of organic compounds, including fatty acids, oils, waxes and steroids, that are insoluble in water but soluble in organic solvents. A triglyceride is a type of lipid consisting glycerine and ester chains.

Lye (KOH or NaOH)
basic catalyst used in making biodiesel.

Methanol (CH3OH)
a volatile colourless alcohol, derived originally as wood alcohol, used as a racing fuel and as a solvent. Also called methyl alcohol, used to make methoxide in biodiesel production.

Methoxide
an organic salt, in pure form a white powder. In biodiesel production, "methoxide" is a product of mixing methanol and lye, yielding a methoxide solution and a significant amount of heat. Methoxide is a liquid that kills nerve cells before you can feel the pain.

Organic
compounds that contain carbon, which are often created as a result of a life process.

Oxidation
burning in oxygen, normally highly exothermic (heat releasing), but also any increase in oxidization state, (i.e. loss of electrons). Results in the formation of an oxide, rusting or corroding. Various materials, such as copper and brass, may oxidise in the presence of vegetable oil or biodiesel.

pH
a measure of acidity and alkalinity of a solution on a scale with 7 representing neutrality. Lower numbers indicate increasing acidity, and higher numbers increasing alkalinity. Each unit of change represents a tenfold change in acidity or alkalinity. The units are gram equivalents per litre of the solution.

Potassium Hydroxide (KOH)
a strong metallic base used as a catalyst in making biodiesel in place of Sodium Hydroxide.

Pour point
the temperature at which the fuel is no longer pumpable.

Rape Seed Oil
food grade oil produced from rape seed is called Canola oil. Canola is a name taken from "Canada oil" due to the fact that much of the development of the oil was performed in Canada. Makes good biodiesel.

Saponification
the reaction of an ester with a metallic base and water. The making of soap. This happens sometimes when you use too much lye in a biodiesel reaction.

Saturated fat
a fat or fatty acid in which there are no double bonds between the carbon atoms of the fatty acid chain. Saturated fats tend to be solid at room temperature. Diets high in saturated fat correlate in some studies with an increased incidence of atherosclerosis and coronary heart disease.

Sodium Hydroxide (NaOH)
a metallic base, strongly alkaline and extremely corrosive. Mixing with fluids usually causes heat, and can create enough heat to ignite flammables (such as methanol), so add slowly. For biodiesel, this is one of the main reactants.

Suspension
a dispersion of a solid in a gas or liquid

SVO
Straight Vegetable Oil or ordinary vegetable oil. Burns well in many diesel engines, but has many undesirable effects

Tallow
one of the harder organic fats derived from animal carcasses. This material is an important material in making soap and candle wax. Also makes biodiesel.

Titration
when applied to biodiesel production, titration is the act of determining the acidity or the amount of FFA of a sample of WVO. It is done to determine the extra amount of base (lye) needed to neutralise an amount of FFA in the WVO.

Transesterification
the process of making biodiesel by the separation of the three hydrocarbon chains from a lipid triglyceride to form glycerol, and biodiesel. It is the process of exchanging the alkyl group of an ester by another alcohol. These reactions are often catalyzed by the addition of an acid or base. Acids can catalyse the reaction by donating an electron to the alkyl group, thus making it more reactive, while bases can catalyse the reaction by removing an electron from the alcohol, thus making it more reactive.

Triglyceride
natural fats and oils composed of glycerine and three fatty acid chains.

ULSD
Ultra low Sulfur Diesel. A type of diesel fuel promoted in recent years in an attempt to reduce atmospheric pollution but at the expense of engine wear.

Unsaturated
a fat or fatty acid containing double or occasionally triple bonds. Many vegetable oils contain fatty acids with one of more double bonds in them. Fat molecules are monounsaturated if each contains one double bond, and polyunsaturated if each contain more than one.

Viscosity
the ability of a fluid to respond to movement. A high viscosity will resist movement, and a low viscosity will flow quickly. This is not necessarily the same as density. Viscosity is normally measured comparatively by the time a given volume of liquid will pass through a pipe of fixed diameter. Methanol has a low viscosity, while vegetable oil has a high viscosity.

WVO
Waste Vegetable Oil, the oil we collect from restaurant grease traps etc, which is the main constituent of making our biodiesel.

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BIODIESEL BASICS

Technical definition

Biodiesel, *n*—a fuel composed of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100, and meeting the requirements of ASTM (American Society for Testing & Materials) D 6751.

Biodiesel history

Rudolph Diesel (1858-1913) developed a theory that revolutionised the engines of his day. He envisioned an engine in which air is compressed to such a degree that there is an extreme rise in temperature. When fuel is injected into the piston chamber with this air, the fuel is ignited by the high temperature of the air, exploding it, forcing the piston down. Diesel designed his engine in response to the heavy resource consumption and inefficiency of the steam engine.

Diesel demonstrated his engine at the Exhibition Fair in Paris, France in 1898. This engine stood as an example of Diesel's vision because it was fueled by peanut oil - the "original" biofuel. He thought that the utilization of a biomass fuel was the real future of his engine. He hoped that it would provide a way for the smaller industries, farmers, and "commonfolk" a means of competing with the monopolizing industries, which controlled all energy production at that time, as well as serve as an alternative for the inefficient fuel consumption of the steam engine. As a result of Diesel's vision, compression ignited engines were powered by a biomass fuel, vegetable oil, until the 1920's when an alteration was made to the engine, enabling it to use a residue of petroleum - what is now known as diesel #2.

Diesel was not the only inventor to believe that biomass fuels would be the mainstay of the transportation industry. Henry Ford designed his automobiles to run on ethanol. Ford was so convinced that renewable resources were the key to the success of his automobiles that he built a plant to make ethanol in the Midwest and formed a partnership with Standard Oil to sell it in their distributing stations. During the 1920's, this biofuel was 25% of Standard Oil's sales in that area. With the growth of the petroleum industry Standard Oil cast its future with fossil fuels. Ford continued to promote the use of ethanol through the 1930's. The petroleum industry undercut the biofuel sales and by 1940 the plant was closed due to the low prices of petroleum.

Despite the fact that men such as Henry Ford, Rudolph Diesel, and subsequent manufacturers of diesel engines saw the future of renewable resource fuels, a political and economic struggle doomed the industry. Manufacturing industrialists made modifications to the diesel engines so they could take advantage of the extremely low prices of the residual, low-grade fuel now offered by the petroleum industry. The petroleum companies wanted control of the fuel supplies in the United States and, despite the benefits of biomass fuel versus the fossil fuels, they moved ahead to eliminate all competition.

At the beginning of World War II, the groundwork for putting biofuels out was in place. First, the diesel engine had been modified, enabling it to use Diesel #2. Second, the petroleum industry had established a market with very low prices for a residual product. Third, a major biomass industry was being shut down. Corn farmers were unable to organise at that time to provide a biomass resource. Finally, industries with immense wealth behind them were acting in concert to push forward their own agenda. It is interesting to note that, during World War II, both the Allies and Nazi Germany utilised biomass fuels in their machines. Despite its use during World War II, biofuels remained in the obscurity to which they had been forced.

By the 1970's, most countries were dependent on fossil fuel. The supply of crude oil, as are all supplies of fossil fuels, was limited. 1973 experienced the first of two crises. OPEC, the Middle

Eastern organization controlling the majority of the oil in the world, reduced supplies and increased prices. The second one came five years later in 1978. As was noted in the Diesel Engine section, automobile purchasers began to seriously consider the diesel car as an option. What is more, people began making their own biofuel. The potential of biofuels reentered the public consciousness. Biodiesel remains in the political and economic arena but is gaining momentum as the awareness of alternative fuel spreads through the consciousness of the general public.

What is biodiesel?

As the name implies Biodiesel is a kind of diesel fuel produced from domestic, renewable resources. It can be used as a better alternative to fossil fuel. It can also be blended at any level with petroleum diesel to create a biodiesel blend.

As with diesel, it can be used in compression ignition (diesel) engines with no major modifications to the engine or the fuel system. Pure biodiesel is simple to use, biodegradable, nontoxic, and essentially free of sulfur and aromatics. It can be used as a pure fuel or blended with petroleum in any percentage. B20 means a blend of 20 percent biodiesel with 80 percent petroleum diesel.

The biodiesel process involves breaking down the oil. This is done by adding it with lye and alcohol, turning oils and fats into esters, separating out the glycerine. The glycerine sinks to the bottom and the biodiesel or ester floats on top and can be siphoned off. Ester is the product which we commonly called biodiesel. The process is called transesterification, which substitutes alcohol for the glycerine in a chemical reaction, using lye as a catalyst. We can use either methanol or ethanol as reactant. The end product will be called “Methyl Esters” if we use methanol, otherwise, it will be called “Ethyl Esters”.

Biodiesel has a solvent effect that may release deposits accumulated on tank walls and pipes from previous diesel fuel usage. The released deposits may end up in fuel filters initially, so fuel filters should be checked more frequently at first.

Biodiesel Characteristics

Biodiesel has physical and chemical properties similar to petroleum diesel and can be used in existing diesel applications with little or no modification to the engine or fueling system. Table 3.1 compares the most common characteristics of No. 2 diesel and biodiesel.

Calorific Value, Heat of Combustion – sometimes called Energy Content, is the amount of heating energy released by the combustion of a unit value of fuels.

Kenimatic viscosity – refers to the thickness of the oil, and is determined by measuring the amount of time taken for a given measure of oil to pass through an orifice of a specified size. Viscosity affects injector lubrication and fuel atomization. Fuels with low viscosity may not provide sufficient lubrication for the precision fit of fuel injection pumps, resulting in leakage or increased wear. Fuel atomization is also affected by fuel viscosity. Diesel fuels with high viscosity tend to form larger droplets on injection which can cause poor combustion, increased exhaust smoke and emissions.

Density – or Specific Gravity is the weight of fuel per unit volume. Oils that are denser contain more energy. For example, petrol and diesel fuels give comparable energy by weight, but diesel is denser and hence gives more energy per litre.

Iodine Value (IV) - is a value of the amount of iodine, measured in grams, absorbed by 100ml of a given oil. The higher the IV the greater potential the oil has to polymerise.

Table 3.1 : Selected Properties of Typical No. 2 Diesel Fuel and B100 Biodiesel

Source: Biodiesel Handling and Use by US Department of Energy (EERE)

Fuel Property	Diesel	Biodiesel
Fuel Standard	**ASTM D975**	**ASTM D6751**
Lower Heating Value, BTU/gal	~129,050	~118,170
Kenimatic Viscosity @ 40°C	1.3 - 4.1	4.0 - 6.0
Specific Gravity (density), kg/l @ 15°C	0.85	0.88
Iodine Value (IV)		60 - 135
Water and Sediment, vol %	0.05 max	0.05 max
Carbon Residue, mass %		0.05 max
Hydrogen, wt %	13	12
Sulfur,wt %	0.05 max	0.0 to 0.0024
Boiling Point, °C	180 to 340	315 to 350
Flash Point, °C	60 to 80	100 to 170
Cloud Point, °C	-15 to 5	-3 to 12
Pour Point, °C	-35 to -15	-15 to 10
Cetane Number	40 - 55	48 - 65
Lubricity SLBOCLE, grams	2,000 - 5,000	>7,000
Lubricity HFRR, microns	300 - 600	<300

Water and Sediment – refers to the presence of free water droplets and sediment particles in the fuel. Poor drying techniques during manufacturing or contact with excessive water during transport or storage can cause water contamination. Excess water can lead to corrosion and provides an environment for microorganisms. Fuel oxidation can also raise sediment levels.

Carbon residue - gives a measure of the carbon-depositing tendency of a fuel and is an approximation of the tendency for carbon deposits to form in an engine.

Sulfur – the percentage by weight of sulfur in the fuel. Sulfur content in biodiesel is limited to reduce sulfate and sulfuric acid pollutant emissions and to protect exhaust catalyst systems on diesel engines. Biodiesel generally contains less than 15 ppm sulfur.

Boiling Point - refers to the temperature at which the oil in liquid form starts to boil and evaporate.

Flash Point - refers the minimum temperature at which the fuel will ignite (flash) on application of an ignition source. Flash point varies inversely with the fuel's volatility. Minimum flash point temperatures are required for proper safety and handling of diesel fuel.

Cloud Point - the temperature at which oil starts to solidify is known as the cloud point. While operating an engine at temperatures below an oil's cloud point, heating will be necessary in order to avoid waxing of the fuel.

Melt Point or Pour Point - Melt or pour point refers to the temperature at which the oil in solid form starts to melt or pour. In cases where the temperatures fall below the melt point, the entire fuel system including all fuel lines and fuel tank will need to be heated.

Cetane Number (CN) - is a relative measure of the interval between the beginning of injection and auto ignition of the fuel. The higher the cetane number, the shorter the delay interval and

the greater its combustibility. Fuels with low Cetane Numbers will result in difficult starting, noise and exhaust smoke. In general, diesel engines will operate better on fuels with Cetane Numbers above 50.

Biodiesel and the environment

The use of biodiesel in a conventional diesel engine results in a substantial reduction of unburned hydrocarbons, carbon monoxide, and particulate matter compared to emissions from diesel fuel. In addition, the exhaust emissions of sulfur oxides and sulfates are essentially eliminated. Of the major exhaust pollutants, both unburned hydrocarbons and nitrogen oxides are ozone or smog forming precursors. The use of biodiesel results in a substantial reduction of unburned hydrocarbons. Emissions of nitrogen oxides are either slightly reduced or slightly increased depending on the duty cycle of the engine and testing methods used.

Biodiesel is biodegradable and non-toxic. B100 biodiesel is as biodegradable as sugar and less toxic than table salt. It biodegrades up-to four times faster than petroleum diesel fuel with up-to 98% biodegradation in three weeks. Compared to fossil diesel fuel, biodiesel has the following emissions characteristics:

- 100% reduction of net carbon dioxide
- 100% reduction of sulfur dioxide
- 40-60% reduction of soot emissions
- 10-50% reduction of carbon monoxide
- a reduction of all polycyclic aromatic hydrocarbons (PAHs) and specifically the reduction of the following carcinogenic PAHs:

For every one ton of fossil fuel burnt, 3 tons of CO^2 is released into the atmosphere, biodiesel only releases the CO^2 that it has taken in while the plants it is made from were growing, and therefore there is no negative impact on the carbon cycle.

Table 3.2: Average Biodiesel Emissions Compared to Conventional Diesel According to EPA

Emission Type	B100	B20
Total Unburned Hydrocarbons	-67%	-20%
Carbon Monoxide	-48%	-12%
Particulate Matter	-47%	-12%
NOx	+10%	+2% to -2%
Sulfates	-100%	-20%
PAH (Polycyclic Aromatic Hydrocarbons)	-80%	-13%
nPAH (nitrated PAH's)	-90%	-50%
Ozone potential of speciated HC	-50%	-10%

Biodiesel and your health

Scientific research confirms that biodiesel exhaust has a less harmful impact on human health than petroleum diesel fuel. Pure biodiesel emissions have decreased levels of polycyclic aromatic hydrocarbons (PAH) and nitrited PAH compounds that have been identified as potential cancer causing compounds. Also, particulate matter, an emission linked to asthma and

other diseases, is reduced by about 47 percent, and carbon monoxide, a poisonous gas, is reduced by about 10 to 50 percent.

Biodiesel blends

Biodiesel can be blended with petroleum diesel in any percentage. The percentages are designated as B20 for a blend containing 20% biodiesel and 80% petroleum diesel, B100 for 100% biodiesel, and so forth.

B20: Twenty percent biodiesel and 80% petroleum diesel—B20—is the most common biodiesel blend. Using B20 provides substantial benefits and avoids many of the cold-weather performance and material compatibility concerns associated with B100.

B20 can be used in nearly all diesel equipment and is compatible with most storage and distribution equipment. B20 and lower-level blends generally do not require engine modifications. Not all diesel engine manufacturers cover biodiesel use in their warranties, however. Because diesel engines are expensive, users should consult their vehicle and engine warranty statements before using biodiesel. It is similarly important to use biodiesel that meets prescribed quality standards—ASTM D6751-07b.

Biodiesel contains about 8% less energy per gallon than petroleum diesel. For B20, this could mean a 1 to 2% difference, but most B20 users report no noticeable difference in performance or fuel economy. Greenhouse gas and air-quality benefits of biodiesel are roughly commensurate with the blend; B20 use provides about 20% of the benefit of B100 use and so forth.

B100: B100 or other high-level biodiesel blends can be used in some engines built since 1994 with biodiesel-compatible material for parts such as hoses and gaskets. However, as biodiesel blend levels increase significantly beyond B20, a number of concerns come into play. Users must be aware of lower energy content per gallon and potential issues on engine warranties, low-temperature gelling, solvency/cleaning effect if regular diesel was previously used, and microbial contamination.

B100 use could also increase nitrogen oxides emissions, although it greatly reduces other toxic emissions.

Biodiesel and your vehicle

Vehicles using biodiesel have similar horsepower and torque as using conventional diesel. Biodiesel has a higher cetane number, but slightly lower energy content than diesel. To the average driver, this means better engine performance and lubrication, but a small decrease in fuel economy (2-8 percent). Biodiesel vehicles can also have problems starting at very cold temperatures, but this is more of an issue for higher percentage blends such as B100.

You don't have to convert the engine to run it on biodiesel, but you might need to make some adjustments and you should check a few things.

Petroleum diesel is basically a waste product in making gasoline. It leaves a lot of dirt in the tank and the fuel system. Biodiesel is a good solvent and it tends to free the dirt and clean it out. You will need to check the fuel filters regularly at first. You might need to change filter after first or second tank full.

If a car has been left standing for a long time with petroleum diesel fuel in the tank the inside of the tank may have rusted. Water content is a common problem with petro-diesel fuel. Biodiesel will free up the rust and the dirt will end up in the fuel filter and clog it.

Having a solvent property, biodiesel, especially 100% biodiesel, will rot any natural or butyl rubber parts in the fuel system, whether fuel lines or injector pump seals. You will need to be aware of this and check these parts regularly. You don't have to replace them at once. It will take some time for this to happen, and if it happens you'll only have to replace them with resistant parts. But rubber parts in diesel engine fuel systems have been rare or non-existent since the early 1980s. It seldom happens, and when it does happen it's not catastrophic, you have plenty of warning and it's easily fixed.

Biodiesel's superior lubricating properties can reduce wear in diesel engines. Bench scale tests have shown that 1% biodiesel can improve the lubricity of diesel fuel by as much as 65%.

Biodiesel also has a higher cetane number than most diesel fuel. Cetane measures the tendency of diesel to autoignite in an engine, and is comparable to the octane number for gasoline. Higher cetane fuels have shorter ignition delay periods than lower cetane fuels. Fuels with a cetane number lower than the engine's minimum requirements can cause rough engine operation and may be more difficult to start, especially in cold weather or at high altitudes. Low cetane fuels may increase engine deposits resulting in more smoke, increased exhaust emissions, and greater engine wear.

Storage

Biodiesel will oxidise readily than petrol diesel. So if left in an open container the shelf life is very short. It will still burn after being oxidised but not nearly as well. When oxidised biodiesel dries it becomes polymerised into a tough, insoluble plastic-like solid. At the high temperatures in internal combustion engines the process is accelerated, steadily accumulating films of tough, insoluble plastic-like solids in the engine and injector pump.

Biodiesel is also hygroscopic. If stored in an open container it will absorb water from the air which will then cause rusting in the engine system

Storing biodiesel is best done in a sealed container, which limits the oxidation to the amount of air and surface area. The container should be full as practically possible. The storage temperature should be less than 30^{o}C. The US industry group, the National Biodiesel Board (NBB), recommends using biodiesel within six months. But it stores indefinitely in completely full, cool and air-tight containers.

Contamination by water: Biodiesel may contain small but problematic quantities of water. Although it is hydrophobic (non-miscible with water molecules), it is said to be, at the same time, hygroscopic to the point of attracting water molecules from atmospheric moisture. One of the reasons biodiesel can absorb water is the persistence of mono and diglycerides left over from an incomplete reaction. These molecules can act as an emulsifier, allowing water to mix with the biodiesel. In addition, there may be water that is residual to processing or resulting from storage tank condensation. The presence of water is a problem because:

- Water reduces the heat of combustion of the bulk fuel. This means more smoke, harder starting, less power.
- Water causes corrosion of vital fuel system components: fuel pumps, injector pumps, fuel lines, etc.
- Water & microbes cause the paper element filters in the system to fail (rot), which in turn results in premature failure of the fuel pump due to ingestion of large particles.
- Water freezes to form ice crystals near 0°C. These crystals provide sites for nucleation and accelerate the gelling of the residual fuel.
- Water accelerates the growth of microbe colonies, which can plug up a fuel system. Biodiesel users who have heated fuel tanks therefore face a year-round microbe problem.

- Additionally, water can cause pitting in the pistons on a diesel engine.

Recommendations for Handling and Using Biodiesel

Avoid spilling Biodiesel into bilges, onto engine or on painted surfaces. Clean spills up immediately with absorbent pads. Rinse surfaces with mild detergent and water. Biodiesel has been shown to degrade polysulphide rubber compounds used on decks. Spilling biodiesel on teak decks utilizing polysulphide caulking has resulted in the deterioration of deck seams. Spills which are immediately cleaned up with soapy water have not had the same impact on deck caulking compounds.

Summary of advantages & benefits of using biodiesel

- Biodiesels are biodegradable.
- They are non-toxic.
- They have significantly fewer noxious emissions than petroleum-based diesel, when burned.
- They are renewable.
- With a much higher flash point than it is for petro-diesel (biodiesels have a flash point of about 170 °C), biodiesel is classified as a non-flammable liquid. This property makes a vehicle fueled by pure biodiesel far safer in an accident than one powered by petroleum diesel or the explosively combustible gasoline.
- Biodiesel is the only alternative fuel that runs in any conventional, unmodified diesel engine.
- Biodiesel can be used alone or mixed in any ratio with petroleum diesel fuel.
- Biodiesel is about 10% oxygen by weight and contains no sulfur. The lifecycle production and use of biodiesel produces approximately 80% less carbon dioxide emissions, and almost 100% less sulfur dioxide.
- Combustion of biodiesel alone provides over 90% reduction in total unburned hydrocarbons, and a 75-90% reduction in aromatic hydrocarbons. When burned in a diesel engine, biodiesel replaces the exhaust odor of petroleum diesel with the pleasant smell of popcorn or french fries. Biodiesel further provides significant reductions in particulates and carbon monoxide than petroleum diesel fuel. Thus, biodiesel provides a 90% reduction in cancer risks.
- The use of biodiesel can extend the life of diesel engines because it is more lubricating than petroleum diesel fuel, while fuel consumption, auto ignition, power output, and engine torque are relatively unaffected by biodiesel.
- Biodiesel is safe to handle and transport because it is as biodegradable as sugar, 10 times less toxic than table salt.
- Biodiesel has almost the same MPG rating as petrodiesel
- Biodiesel readily blends and stays blended with petrodiesel.
- Biodiesel boasts of a zero total emissions production facility

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SAFETY

While the finished products of the reaction are nontoxic, some of the chemicals used to make biodiesel are dangerous. Always practice standard laboratory safety procedures when making biodiesel. Wear protective apparel: chemical proof gloves, eye protection, apron, and close-toed shoes. Tie back long hair, remove jewelry, and work safely. Avoid breathing fumes by not putting your face above any chemicals and use a fume hood if available. Your workspace should be well ventilated, and water and vinegar (to neutralise basic substances like lye and methoxide) should be on hand in case of spills.

Handle all substances carefully. Methanol is toxic to inhale, ingest, or come in contact with. Sodium Hydroxide (lye) and Potassium Hydroxide are both caustic, contact can cause chemical burns. Methoxide and Ethoxide are caustic and produce toxic vapours. Keep all substances away from children and pets, and handle only with adult supervision.

Making sodium methoxide is the most dangerous step when making biodiesel. Carefully consider the safety of the design of your equipment and workspace before mixing methoxide. Always wear protective clothing and a respirator.

On the other hand, nobody has yet burned/maimed/blinded/killed themselves or anyone else making homebrewed biodiesel. Large numbers of ordinary people all over the world are making their own biodiesel. It's been going on for years, and so far there have been no serious accidents. It's safe if you're careful and sensible. As a comparison, food could even be more dangerous. A lot of people died every year due to high blood pressure which can be linked directly to eating habit. If you always use your common sense in curbing your eating habit you could avoid it. The same thing is true with biodiesel home brewing. So don’t be scared. Making biodiesel is safe if you're careful and sensible. Nothing about life is safe if you're not careful and sensible!

Methanol

Wear proper protective gloves, apron, and eye protection and do not inhale any vapour when dealing with methanol. Methanol can cause blindness and death, and you don't even have to drink it, it's absorbed through the skin. Organic vapour cartridge respirators are more or less useless against methanol vapours. The best advice is not to expose yourselves to the fumes in the first place. The main danger is when the methanol is hot. When it's cold or at "room temperature" it fumes very little if at all and it's easily avoided, just keep it at arm's length whenever you open the container. Avoid using "open" reactors. Biodiesel processors should be closed to the atmosphere, with no fumes escaping. All methanol containers should be kept tightly closed anyway to prevent water absorption from the air. Refer to Chapter 14 for more information about methanol.

Sodium hydroxide

Sodium hydroxide can cause severe burns and death. Handle it with caution.

Sodium methoxide

Together methanol and sodium hydroxide form sodium methoxide, an extremely caustic chemical. This is a dangerous chemical; treat it as such, the same with methanol.

Sulfuric acid

Sulfuric acid is a very corrosive chemical. Take full safety precautions; wear safety goggles, gloves and apron and eye protection. Have running water nearby. Don't inhale fumes.

The corrosive properties of sulfuric acid are accentuated by its highly exothermic reaction with water. Hence burns from sulfuric acid are potentially more serious than those of comparable strong acids (e.g. hydrochloric acid), as there is additional tissue damage due to dehydration and particularly due to the heat liberated by the reaction with water; i.e. secondary thermal damage. The danger is obviously greater with more concentrated preparations of sulfuric acid.

The standard first aid treatment for acid spills on the skin is, as for other corrosive agents, irrigation with large quantities of water. However, the acid should be neutralised first by rinsing with a base (e.g. calcium hydroxide solution), because the water used in washing will react with the acid and increase the chance of secondary damage. Washing should be continued for at least ten to fifteen minutes in order to cool the tissue surrounding the acid burn and to prevent secondary damage. Contaminated clothing must be removed immediately and the underlying skin washed thoroughly.

Preparation of the diluted acid can also be dangerous due to the heat released in the dilution process. It is essential that the concentrated acid is added to water and not the other way round, to take advantage of the relatively high heat capacity of water. Addition of water to concentrated sulfuric acid leads at best to the dispersal of a sulfuric acid aerosol, at worst to an explosion.

Although sulfuric acid is non-flammable, contact with metals in the event of a spillage can lead to the liberation of hydrogen gas. The dispersal of acid aerosols and gaseous sulfur dioxide is an additional hazard of fires involving sulfuric acid.

Sulfuric acid is not considered toxic besides its obvious corrosive hazard, and the main occupational risks are skin contact leading to burns (see above) and the inhalation of aerosols. Exposure to aerosols at high concentrations leads to immediate and severe irritation of the eyes, respiratory tract and mucous membranes: this ceases rapidly after exposure, although there is a risk of subsequent pulmonary edema if tissue damage has been more severe.

Dress code:

Making biodiesel requires chemicals which are said to dangerous. To make it safe, you need to formulate and implement a strict dress code in your work area. Requirements include:

- gloves - should be chemical-proof with cuffs that can be pulled up over long sleeves.
- no shorts or sandals.

Always have running water handy when working with them. The workspace must be thoroughly ventilated. No children or pets allowed.

Fire Hazards

The most flammable chemical used in making biodiesel is methanol. It is flammable in liquid as will as in gas form. Read more about methanol in Chapter 14.

The main fire hazard is using an open reactor and poor ventilation in the presence of an ignition source. Ignition sources can be:

- Using combustible fuels as a direct heat source (propane, natural gas, wvo, wood, etc.), rather than a heat exchange system where the flame is far removed from the processing area.
- Open electric motor housings, rather than TEFC motors (Totally Enclosed Fan Cooled AC Motors) or explosion proof motors.
- Disconnecting an electrical appliance by pulling out the wall plug, rather than using enclosed switches.
- Any open flame.

Other fire sources can be faulty electrical connections, over-taxed pumps and motors in close proximity to combustible materials (garage walls, plastic barrels, almost anything), oversised breakers and/or fuses, undersised wiring for higher amperage draws.

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BIODIESEL PRODUCTION

Biodiesel can be made from new or used vegetable oils and animal fats, which are nontoxic, biodegradable, and renewable. Fats and oils are chemically reacted with an alcohol (methanol or ethanol) to produce chemical compounds known as fatty acid methyl (or ethyl) esters. Biodiesel is the name given to these esters when they are intended for use as fuel. Glycerine (used in pharmaceuticals and cosmetics, among other markets) is produced as a co-product.

Vegetable or animal oils and fats are triglycerides, composed of three chains of fatty acids bound by a glycerine molecule. Free fatty acids (FFAs) are fatty acids that have become separated from the triglycerides. This is caused by heat and water in the foods cooked in the oil. The hotter the oil gets and the longer it's cooked, the more FFAs it will contain.

As glycerine is an alcohol (glycerol), a fatty acid attached to it (a glyceride) forms an ester. A "transesterification" is the conversion (switching) of one ester into another (a glyceride ester into an alkyl ester) in the case of biodiesel, where methanol (or ethanol) replaces the glycerine.

An "esterification" is the conversion of a non-ester into an ester. FFAs are non-esters. FFAs are converted into esters by acid esterification in the first stage of the two-stage acid-base biodiesel process. The second stage will be basically transesterification.

In a single-stage process the FFAs must be removed from the process, or they will dissolve in the biodiesel being formed, yielding an acidic, poor-quality fuel that will not meet quality standards. In transesterification, using a single-stage process, extra lye is used to neutralise the FFA content of the oil, turning it into soaps. These soaps drop out of the process as a by-product, joining the so-called "glycerine layer" at the bottom.

The basic lye quantity used in transesterification acts as a catalyst, not a neutraliser. Lye attacks ester bonds, breaking the bond, and the alcohol drops off, leaving an open-ended fatty acid chain. With glycerides the alcohol that drops off is glycerine. The affinity of the replacement methanol or ethanol for the resulting open bond is strong enough to prevent the glycerine reattaching to the fatty acid.

This is also why it is critical that a minimal amount of lye is used, as lye will continue to attack ester bonds, even those of biodiesel. Too much lye will break the biodiesel ester bonds; some of the broken bonds will mate with the lye and form excess soap, and others will match up with a water molecule to form FFAs, which dissolve back into the biodiesel resulting in a poor quality biodiesel. FFAs can corrode fuel injection equipment, cause filter plugging and the build-up of sediments on fuel injection parts.

Production methods

Biodiesel can be produced using a variety of production methods or esterification technologies. The list includes the batch process; supercritical process; the McGyan process; ultra- and high-shear in-line reactors and the ultrasonic-reactor method and many others. Of these methods, only the batch process is applicable to home brewing of biodiesel.

With the batch process, the WVO is filtered and preprocessed to remove water and contaminants. If free fatty acids are present, they can be treated in two ways. Titration could be done to determine the extra amount of catalyst needed or it could be transformed into ester using the esterification process. There are two types of batch process employed in biodiesel home processing, the single-stage process and the two-stage process. The single stage employs titration to determine the extra amount of lye needed to neutralise the FFA while the

two-stage process employs esterification wherein the FFAs are transformed into ester and then converted with the other esters into biodiesel using transesterification process. The batch process could be summarised as follows:

- Preparation: WVO may contain some food debris, water and FFAs. Food debris can be removed by filtering. Care must be taken to monitor the amount of water and FFAs in the incoming biolipid (oil or fat). If the free fatty acid level or water level is too high it may cause problems with soap formation (saponification) and the separation of the glycerine by-product downstream. In the case of single stage process, this problem could be solved by titration. In the case of two-stage process, this could be solved by esterification which is actually the stage 1 of the process.

- Catalyst is dissolved in the alcohol using a standard agitator or mixer. Take note that the catalyst is hygroscopic, hence, care must be taken to ensure that it does not absorb moisture from the air.

- The alcohol/catalyst mix is then charged into a closed reaction vessel and the biolipid (vegetable or animal oil or fat) is added. The system from here on is totally closed to the atmosphere to prevent the loss of alcohol.

  The reaction mix is kept at 45 to 55^{o}C to speed up the reaction. The alcohol boils at around 65^{o}C. The solution is stirred for 15 to 20 minutes then let it settle for 12 to 24 hours.

- After settling, the biodiesel will stay at the top while the glycerine settles at the bottom. The glycerine phase is much more dense than biodiesel phase and the two can be gravity separated with glycerine simply drawn off the bottom of the settling vessel.

- Once the glycerine and biodiesel phases have been separated, the excess alcohol in each phase is removed with a flash evaporation process or by distillation and is re-used. Care must be taken to ensure no water accumulates in the recovered alcohol stream.

- The glycerine by-product contains unused catalyst and soaps that are neutralised with an acid and sent to storage as crude glycerine.

- Once separated from the glycerine, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps, dried, and sent to storage.

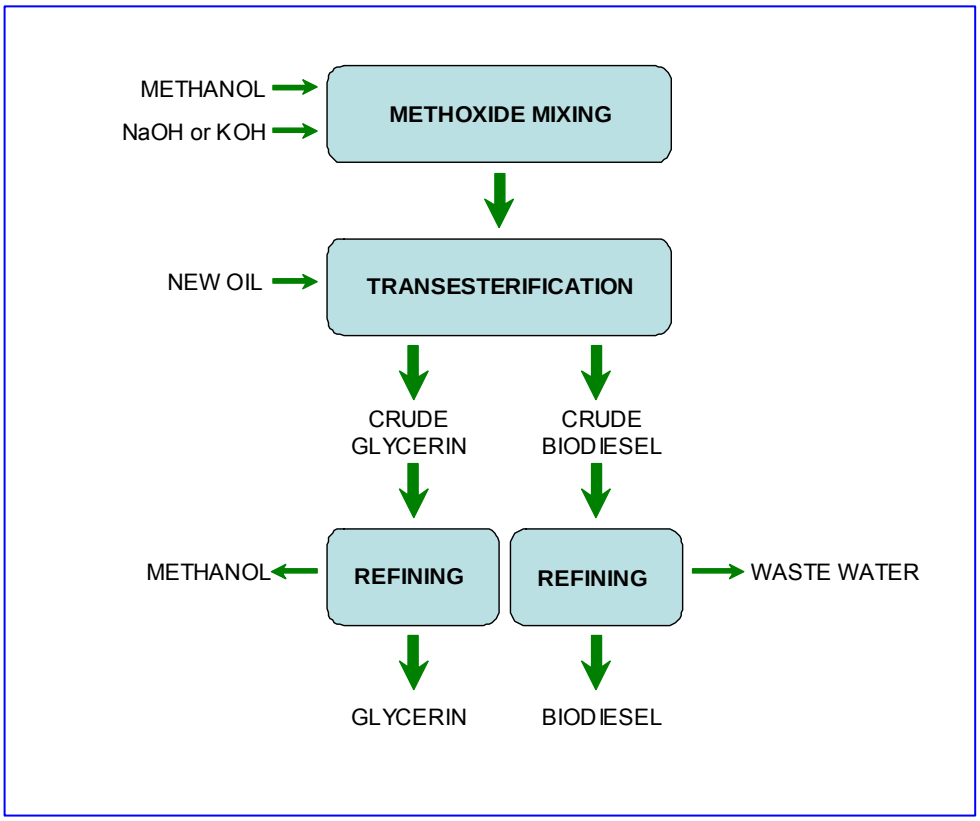

Figure 5.1: Schematic diagram of biodiesel production using new oil, single stage process

Figure 5.1 illustrates biodiesel production using new oil, single stage process. Because new oil is used, it is assumed that the oil is clean with no food debris to filter. Water content and FFAs are negligible; hence, titration is not necessary.

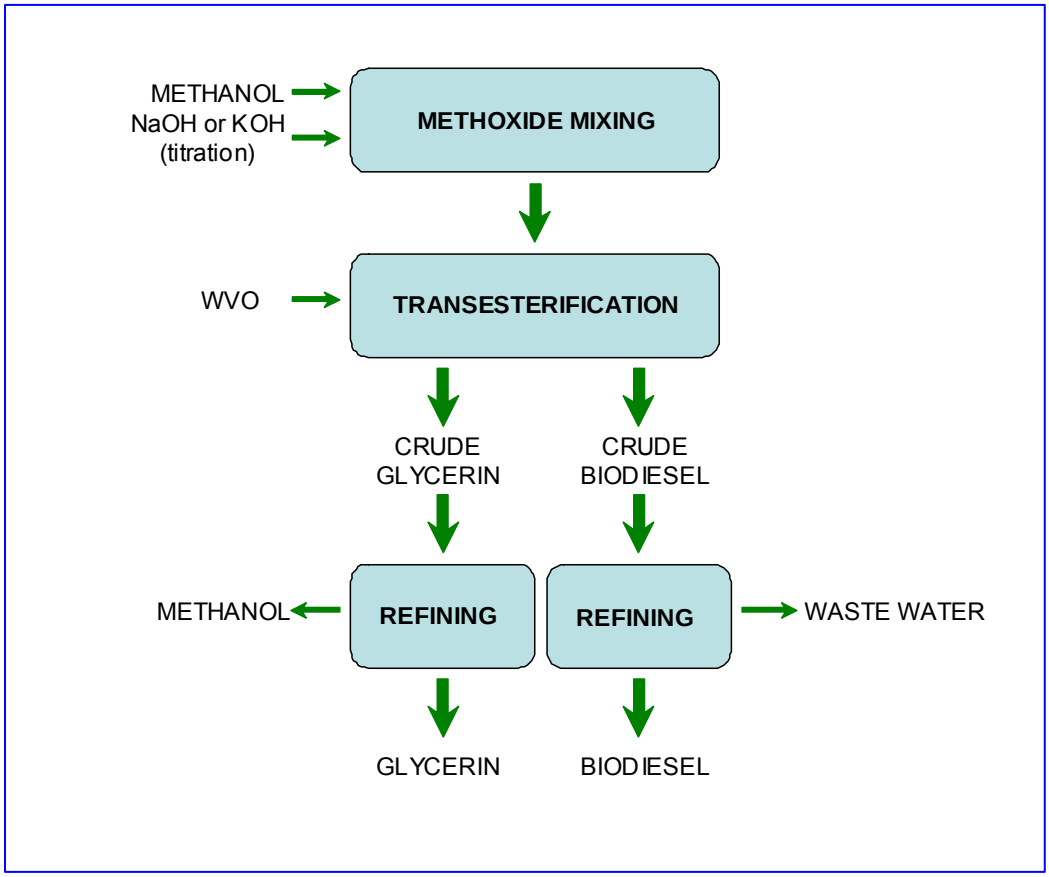

Figure 5.2: Schematic diagram of biodiesel production using WVO, single stage process

Figure 5.2 illustrates biodiesel production using waste vegetable oil, single stage process. WVO contains food debris, some water and FFAs. Pre-processing of WVO is needed to remove some food debris and water. Titration is also needed to determine the amount of extra lye needed to neutralise FFAs. Refer to Chapter 7 for more information in preparing WVO.

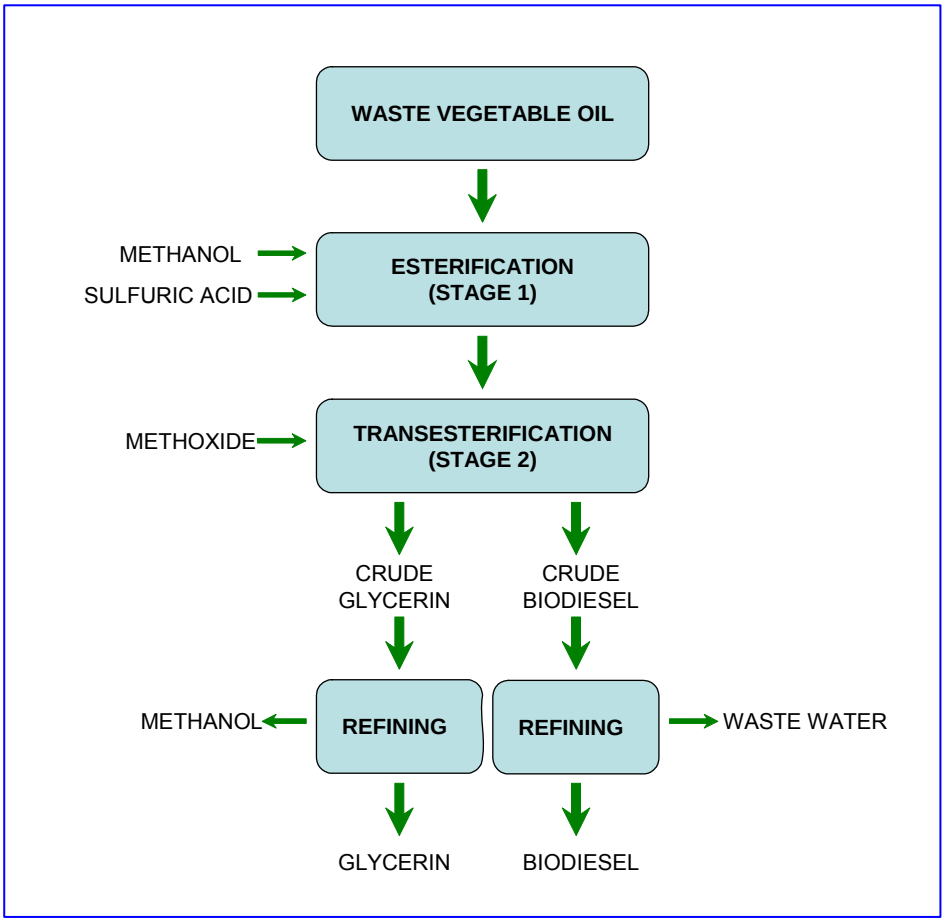

Figure 5.3: Schematic diagram of biodiesel production using WVO, 2 stage process

Figure 5.3 illustrates biodiesel production using waste vegetable oil, 2-stage process. Just like Figure 5.2 above, pre-processing of WVO is needed to ensure proper reaction of the reactants. In stage 1, sulfuric acid is added to neutralise FFAs and then methanol is added to transform neutralised FFAs to esters. Stage 2 downwards follows the process as shown in Figure 5.1 and 5.2, but with reduced amounts of methoxide. It should be noted that titration is not needed in this process. However, titration might be needed if you wish to refine the glycerine byproduct. Glycerine byproduct contains lye which needs to be neutralise. To neutralise it you need to know how much lye was used in the process, and determining this amount will not be a problem if titration was done in the first place.

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CHEMICALS FOR BIODIESEL PRODUCTION

The breaking up of vegetable oil and transforming it to esters and glycerine needs chemicals. It should be noted that these chemicals are highly dangerous. Read Chapter 4 (Safety) first before attempting to make your first biodiesel. Though these chemicals are dangerous, the resulting biodiesel is non-toxic and biodegradable. These chemicals are dangerous, but with proper handling and following strictly the safety precautions, the process could be simple and safe.

Alcohol

Alcohol breaks down the vegetable oil into ester and glycerine. It is needed whether you use new vegetable oil or waste vegetable oil. You can use either methanol, which makes methyl esters, or ethanol which makes ethyl esters.

Methanol: Methanol is sometimes called methyl alcohol. Methanol is a poisonous chemical that can blind you or kill you. It is more poisonous than ethanol. You don't even have to drink it because you can absorb it through the skin and you can breathe in the fumes. However, human susceptibility to the acute effects of methanol intoxication is extremely variable. The minimum dose of methanol causing permanent visual defects is unknown.

Authorities advise that swallowing up to 1.3 grams or 1.7 ml of methanol or inhaling methanol vapour concentrations below 200 ppm should be harmless for most people. No severe effects have been reported in humans of methanol vapour exposures well above 200 ppm.

Out of 1,601 methanol poisonings reported in the US in 1987 the death rate was 0.375%, or 1 in 267 cases. It might have been only 1 in more than a thousand cases because most cases weren't reported. Most cases were caused by drinking badly made home-made spirits and this has nothing to do with biodiesel production.

Methanol is readily biodegradable in the environment under both aerobic and anaerobic conditions (with and without oxygen) in a wide variety of conditions. Generally 80% of methanol in sewage systems is biodegraded within 5 days.

Methanol is of low toxicity to aquatic and terrestrial organisms and it is not bioaccumulated. (It's toxic mainly to humans and monkeys.)

Environmental effects due to exposure to methanol are unlikely. Unless released in high concentrations, methanol would not be expected to persist or bioaccumulate in the environment. Low levels of release would not be expected to result in adverse environmental effects.

There are no reports of carcinogenic, genotoxic, reproductive or developmental effects in humans due to methanol exposure. Its environmental effects if any are minimal and short-lived.

You will need 200ml of methanol per litre of vegetable oil (waste or new). Most methanol come from fossil fuels.

You can usually get methanol from bulk liquid fuels distributors. With a bit of patience, most people in most countries can manage to track down a source of methanol for a much lesser price. For small amounts, you can use "DriGas" fuel antifreeze. Methanol is also sold in supermarkets and chain stores as "stove fuel" for barbecues and fondues.

There is no free methanol in washed biodiesel. All the national standards require washing. According to US EPA studies methyl esters biodiesel is less toxic than table salt and more biodegradable than sugar. It has none of the toxic or environmental hazards of fossil diesel fuel.

Most of the excess methanol used in the biodiesel process ends up in the glycerine by-product layer, and the rest stays in the biodiesel. If you don't reclaim it for re-use the portion that's in the biodiesel gets washed out when you wash the fuel, mostly with the first wash. The first wash-water probably won't contain more than 5-6% methanol (as well as some sodium or potassium lye and some soap).

Ethanol: Ethanol is sometimes called ethyl alcohol. Like methanol, it is also poisonous, with methanol being more poisonous. But they are not dangerous if you work carefully and safely.

Most ethanol is plant-based and you can distill it yourself if you want to. But the biodiesel process using ethanol is more difficult than with methanol.

Lye

Lye is a type of chemical compound that propels the transesterification process faster. It is a catalyst that helps alcohol does its job. You will need lye catalyst whether you use new or waste vegetable oil. The lye can be either potassium hydroxide (KOH) or sodium hydroxide (caustic soda, NaOH).

NaOH is often easier to get and cheaper but more difficult to use because it takes longer time to dissolve it in alcohol.

KOH is easier to use, and it does a better job. Experienced biodieselers making good-quality fuel usually use KOH, and so do the commercial producers. KOH will produce potash fertiliser as a by-product of the biodiesel process. You will need 1.4 times more KOH compared to NaOH.

You can get both KOH and NaOH from soapmakers suppliers and from chemicals suppliers or from hardware stores.

NaOH is used as drain-cleaner and you can get it from hardware stores. It has to be pure NaOH.

Lye (both NaOH and KOH) is dangerous, don't get it on your skin or in your eyes, don't breathe any fumes, keep the whole process away from food, and right away from children. Lye reacts with aluminium, tin and zinc. Use HDPE (High-Density Polyethylene), glass, enamel or stainless steel containers.

Lye is very hygroscopic. It will easily absorb water moisture from the air. Water interferes with the biodiesel reaction, producing a poor quality biodiesel. You need to be quick when measuring out the lye. If possible make the measuring and weighing inside a closed plastic bag. You need to improvise some ways and means to the weighing and measuring weather-tight.

Lye also absorbs carbon dioxide from the atmosphere and becomes carbonated if not stored properly. Carbonated lye is chalky white, fresh lye is almost translucent.

NaOH must be at least 97% pure, use exactly 3.5 grams per litre of new oil. KOH must be at least 85% pure to give satisfactory result. If you are using KOH, follow the following:

99% pure KOH	4.91 grams per litre of new oil
92% pure KOH	5.33 grams per litre of new oil
90% pure KOH	5.45 grams per litre of new oil
85% pure KOH	5.78 grams per litre of new oil

These amounts of NaOH and KOH are in case you are using new vegetable oil. In the case of waste vegetable oil, you will need extra amount of lye to take care of more free fatty acids. This extra amount of lye will be determined by titration. Just add the titration amount to the above figures (don’t confuse yourself adding ml to grams, that’s the way it works, just follow it).

You can use either KOH solution or NaOH solution for titration. Unlike NaOH, your KOH probably won't be pure, but as long as you use the same strength of KOH for both the titration solution and the reaction it won't make any difference.

Fresh cooking oil contains a standardised amount of Free Fatty Acids (FFAs), which can slow or stop the transesterification process. The lye is alkaline and neutralises the FFA by turning it into soap, which drops out with the glycerine layer, and with 3.5 grams of NaOH per litre of oil there's enough left over to catalyse the biodiesel reaction. Used cooking oil (WVO) needs more lye than new oil, not to serve as the catalyst but to neutralise the extra FFAs formed in cooking the oil.

However, too much lye will make extra soap, with very alkaline biodiesel that's difficult to wash, with loss of production, or it can ruin the reaction when the ratio of soap to biodiesel reaches a point where the whole batch turns into "glop soap". Too little lye will mean some of the oil is left unreacted.

Isopropanol

Isopropanol is needed for titration. Isopropanol is available from chemicals suppliers.

Phenolphthalein

Titration involves checking pH. In the absence of a pH metre or litmus paper, you can use phenolphthalein indicator. Phenolphthalein is an acid-base indicator that's colourless in acid and red in base. Phenolphthalein lasts about a year. It's sensitive to light so store it in a cool, dark place. You can get it from chemicals suppliers.

Methoxide

Methoxide is what you will get if you mix methanol and sodium hydroxide or potassium hydroxide. Mixing lye with methanol creates an exothermic reaction, generating heat. It must be thoroughly mixed before you use it, with all the lye dissolved.

Take full safety precautions when working with methanol and lye. Methanol and lye are two dangerous substances. When mixed together they will create an even more dangerous stuff. You have to improvise some ways and means to make the process more simple and safe.

Use a tough, thick, container made of HDPE (High-density Polyethylene), with a screw-on lid. Measure out the methanol into the container. Add the required amount of lye. If you're doing large quantities, add it bit by bit rather than all at once, give the container a swirl in between (replace stopper and lid first). Swirl it about from side to side rather than shaking it up and down. Once it's all added, replace the stopper and the lid, and swirl the mixture about for a few seconds. Then let it stand. Do that a few more times, every few hours or so until all lye is dissolved. It will be thoroughly dissolved in 24 hours, or maybe a bit longer. Don’t use it until all the lye is thoroughly dissolved. If you use a white translucent HDPE container you can see any undissolved lye at the bottom of the container.

Potassium hydroxide (KOH) dissolves in methanol much faster than NaOH, sodium hydroxide. It can be ready in as little as 10 minutes.

Stock methoxide solution is very useful for making test batches. You also have the option of buying Sodium Methoxide from the supplier if this available in your area. Rather than measuring tiny amounts of lye for each test batch, make a stock solution using one litre of methanol and 50 grams of lye. Then you can dilute quantities of the stock solution to whatever strength each test batch requires.

Example:

If titration was, say, 3 ml, you'll need 3 + 3.5 grams of NaOH lye per litre of WVO. You will also need 200 ml of methanol at 20%.

Your stock methoxide solution contains 1 gram of lye for every 20 ml (1000/50). Since you need 6.5 grams of lye per litre of WVO, measure 130 ml (20x6.5) of stock solution to cover all your lye requirements per litre of WVO. Since you need 200ml of methanol per litre of WVO, you'll only have to top it up with 70 ml of methanol.

Once mixed, methoxide won't last forever, but it's good for a few weeks.

Sulfuric Acid (H_2SO_4)

Sulfuric acid, is a strong mineral acid. It is soluble in water at all concentrations. Sulfuric acid has many applications, and is one of the top products of the chemical industry. Principal uses include ore processing, fertilizer manufacturing, oil refining, wastewater processing, and chemical synthesis.

Although nearly 100% sulfuric acid can be made, this loses SO_3 at the boiling point to produce 98.3% acid. The 98% grade (18M) is more stable in storage, and is the usual form of what is described as *concentrated sulfuric acid*. Other concentrations are used for different purposes. Some common concentrations are

- 10%, *dilute sulfuric acid* for laboratory use,
- 33.5%, *battery acid* (used in lead-acid batteries),
- 62.18%, *chamber* or *fertilizer acid*,
- 77.67%, *tower* or *Glover acid*,
- 98%, *concentrated acid*.

The hydration reaction of sulfuric acid is highly exothermic. If water is added to the concentrated sulfuric acid, it can react, boil and spit dangerously. One should always add the acid to the water rather than the water to the acid. The necessity for this safety precaution is due to the relative densities of these two liquids. Water is less dense than sulfuric acid, meaning water will tend to float on top of this acid.

With biodiesel production, always add sulfuric acid to the oil rather than the other way around. Use at least 95% pure sulfuric acid.

Phosphoric acid (H3PO4)

Phosphoric acid is a mineral (inorganic) acid having the chemical formula H_3PO_4. Pure 75-85% aqueous solutions (the most common) are clear, colourless, odourless, non-volatile, rather viscous, syrupy liquids, but still pourable. Phosphoric acid is very commonly used as an aqueous solution of 85% phosphoric acid. Because it is a concentrated acid, an 85% solution can be corrosive, although nontoxic when diluted.

Melting point – 41 ^{o}C
Boiling point – 158 ^{o}C
Specific gravity – 1.69

Vapour density – 3.4
Non-flamable

Phosphoric acid is essential in the body organism as the constituent of bones and teeth as well as in many metabolic processes of carbohydrate, fat and protein. Phosphoric acid is abundant in natural foods as the form of free phosphoric acid itself or as the mineral salts (potassium, sodium or calcium). Phosphoric acid is used to acidify foods and beverages. But the continuous and excessive absorption of beverages particularly Coca Cola and Pepsi Cola which contain large amount of phosphoric acid should be limited. Phosphate excretion takes place in the form of calcium phosphate. The excessive amounts of phosphoric acid in the body may cause calcium deficiency which causes poor teeth and weak bone density (osteoporosis). Phosphoric acid is used in pharmaceutical preparations as a solvent and as a gastric acidifier orally.

Most phosphoric acid is used in the production of fertilizers. Phosphorus is one of the elements essential for plant growth. Organic phosphates are the compounds which provide the energy for most of the chemical reactions that occur in living cells. Therefore, enriching soils with phosphate fertilizers enhances plant growth.

Increasing the phosphate concentration in surface waters also enhances the growth of aquatic plant life. Run-off from fertilized farm lands can stimulate plant growth in lakes and streams. Waste water that contains phosphates from detergents can have the same effect.

It is also used in making electrolytes, electroplating and derusting solutions. It is used in the manufacture of industrial cleaning products, other inorganic and organic phosphoric chemicals, foundry resins, paints, enamels and refractory, antifreeze productions, and textile process materials. It is used in water treatment. Food grade phosphoric acid is used; as a acidulation in soft drink (particularly cola); ph control in imitation jellies; nutrient in production of yeast; bacteria growth control in selected processed foods; flocculating agent for clarification of sugar juices after liming process.

Phosphoric acid is also used in dentistry and orthodontics as an etching solution, to clean and roughen the surfaces of teeth where dental appliances or fillings will be placed. This acid is also used in teeth whiteners to eliminate any plaque that may be on your teeth.

In biodiesel production, adding phosphoric acid (H_3PO_4) converts the soap back to FFAs and separates it all into three distinct layers, with catalyst-phosphorus on the bottom, glycerine-methanol in the middle, and FFAs on the top.

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PREPARING WVO

Waste vegetable oil (WVO) contains variable amounts of charred food debris, suspended water and free fatty acids. If you are using WVO instead of new oil, it is necessary to have it undergone pre-processing to remove these impurities. The presence of water causes the triglycerides to hydrolyze to give salts of the fatty acids instead of undergoing transesterification to give biodiesel. Free fatty acids also will react with lye, thus reducing the amount of lye available for transesterification.

Different restaurants have different cooking methods, hence, different impurities, different water contents and different amount of FFAs. To save time and resources, it is necessary that you don't combine them until you do preliminary testing to determine the amounts of these impurities. You may then combine those WVOs not needing pre-treatment and vice versa.

Removing suspended water

Before you mix WVO from different sources you should filter them first and checked them for water content. As stated above different restaurants have different cooking methods and food requirements. Some WVO might contain water, some might not. You will save time and energy if you treat only those WVO which need treatment. You may combine those needing treatment and those that are not. Here's how it is done:

1. Let the WVO from different sources settle for at least 12 hours before testing for water content. To test for water content; heat half a litre or so of the oil in a saucepan on the stove. Monitor the temperature with a thermometer. If there's water in it, it will start to produce crackling or popping sounds by 50^{o}C or so. If it's still not crackling by 60^{o}C there's no need to dewater it. Test the WVO from different sources before combining them.
2. Make sure you didn't disturb the WVO in their respective containers. For those WVO which does not need water content treatment, transfer the upper 90% (filtering at the same time) to your WVO storage, ready for further processing.
3. Combine the remaining 10% from step 2 to the batches of WVO which need water content treatment. Heat the WVO to 60%, maintain the temperature for 15 minutes. Remove from heat, then let it settle for 12 hours. Then remove the top 90% (filtering at the same time) and combine it to the tested WVO in step 2, ready for further processing.
4. For the remaining 10% of WVO from step 3, raise the temperature to 100^{o}C, hold it there and allow any water to boil off. Stir the WVO occasionally to avoid steam pockets forming below the oil and exploding, splashing hot oil out of the container. When boiling slows, raise the temperature to 130^{o}C for 10 minutes. Remove heat, allow it to cool and combine it to the 90% in step 3, ready for further processing.

Titration

Make sure you are wearing all of your protective gear: goggles, gloves, apron and shoes. See Chapter 4 "Safety".

For processing used oil, it's essential to titrate the oil to determine the Free Fatty Acid (FFA) content and calculate how much extra lye will be required to neutralise it.

If you are souring WVO from many different restaurants, you may mix them together in a large container so that you will only make single titration and single trial batch. This could be done after you have tested them for water content and de-water them. You may make several titrations and several trial batches but you only use one reliable result.

Titration involves determining the pH, the acid-alkaline level of the oil. PH7 is neutral, lower values are increasingly acidic while higher than 7 is alkaline. An electronic pH metre is best, but you can also use pH test strips (or litmus paper), or, better than test strips, phenolphthalein solution. Phenolphthalein lasts about a year. It's sensitive to light so store it in a cool, dark place.

Titration is the most difficult step in the process of making biodiesel, and the most critical. Make your titration as accurate as possible. Here's how it is done:

a. Dissolve 1 gm of lye (KOH or NaOH) in 1 litre of distilled water to make 0.1% w/v lye solution (weight-to-volume). Make sure it dissolves completely. This sample is then used as a reference tester for the titration process. It's important not to let the sample get contaminated. It can be used for many titrations.

b. In a smaller beaker, dissolve 1 ml of dewatered WVO with 10 ml of pure isopropyl alcohol. Warm the beaker gently by standing it in hot water, stir until all the oil dissolves in the alcohol and turns clear.

c. Add 2 drops of phenolphthalein solution. Ignore this step if you are using pH metre.

d. Using a graduated syringe add 0.1% lye solution drop by drop to the oil-alcohol-phenolphthalein mixture, stirring all the time. It might turn a bit cloudy, keep stirring. Keep on carefully adding the lye solution until the mixture just starts to turn pink (magenta) and stays that way for 15 seconds or if the pH metre reads 8 to 9.

e. Take the number of millilitres of 0.1% lye solution you used and add the basic amount of lye needed to process fresh oil, 3.50 grams for NaOH or 4.91 grams for (pure) KOH. This is the number of grams of lye you'll need per litre of used oil. Don't worry that you seem to be adding millilitres to grams, that's the way it works. For example if you are using NaOH lye, the result of your titration is 3.5ml and you want to process 150 litres of WVO, you will need 1050 grams (150 x 7) of NaOH lye.

What has been outlined above is the basic process of titration. It looks so simple but measuring 1 gram of lye or 1ml of WVO is not very simple. The lesser the amount you are measuring, the bigger the error. But there's a way of doing it more accurate. In the case of lye solution, mix 5 grams of lye with 500ml of distilled water to make a stock solution. Before titration measure out 5 ml of the stock solution, add 45ml of distilled water. This makes a 0.1% w/v lye solution.

In the case of oil-isopropyl alcohol solution, mix 4 ml of oil in 40 ml of isopropyl alcohol in a glass beaker. Warm the mixture gently by standing the beaker in hot water, stir until all the oil dissolves and it becomes a clear mixture. Then titrate as usual, measuring millilitres of stock solution used. When it reaches pH8.5 count up the number of millilitres used as usual; and then divide it by 4. This will give a much more precise result.

To facilitate the learning process, you will have to start with waste vegetable oil of better quality first. You should avoid poor-quality oil for early test-batches. You should use oil that titrates at 2 to 3ml of 0.1% lye solution. Leave overcooked oils with high titration levels for later when you have gained more experience.

Satisfactory results mean that you get a good, clear separation, that it settles well, leaving a clear product with not too much soap formation, a good production rate, and, most important, that it washes easily without frothing.

Deacidifying WVO

WVO contains variable amounts of free fatty acids (FFAs). FFAs interfere with the biodiesel process by reacting with the lye to form soap. We do titration to determine the amount of FFAs

present in the WVO. If the WVO contains too much FFAs, say, more than 4ml titration, we may deacidify it. The process simply involves removing or neutralizing the FFAs in advance of transesterification.

There are two ways to deacidify WVO. The first involves using an acid catalyst and the second uses a base catalyst. The base catalyst converts FFAs into soap while the acid catalyst coverts FFAs into esters which can further be converted into biodiesel.

1. Acid Catalysis

Measure the WVO to be processed and put in the reactor. Pre-heat it to 35°C. Measure 0.08 litre of methanol per litre of WVO and add it to the heated oil. Use only 99%+ methanol. Mix it for 5 minutes. The mixture will become murky because of solvent change. For each litre of WVO, add 1ml of 95% sulfuric acid. Use a graduated eye dropper, a graduated syringe or a pipette. Take extra care when handling concentrated sulfuric acid. Mix gently while keeping the temperature at 35°C. Maintain the temperature for 1 hour then stop heating but continue stirring. Stir the mixture for another hour (or a total of 2 hours). Then let it set for at least 8 hours.

The process transforms FFAs into esters which is then transformed into biodiesel in the succeeding stages, hence no need to separate them.

2. Base Catalysis

Mix NaOH with 40ml of water per litre of WVO. Use the amount of NaOH according to your titration result. For example, if titration result is 4.5ml, you measure 8 grams (3.5+4.5) of NaOH for every litre of WVO. It gets hot. Use a stainless steel or HDPE container. Mix it by stirring gently, not shaking up and down. This is very corrosive stuff, take full safety precautions, have running water handy.

When the NaOH is fully dissolved add the solution to the oil at room temperature. Stir gently and thoroughly using a mechanical mixer until fully mixed. Let it settle overnight. This leaves soapstock at the bottom. The water stays with the soapstock. Filter to remove the soapstock.

You may now process the pre-treated WVO as if it is new oil, using 3.5 grams NaOH per litre of oil, and 20% methanol. No need for titration.

You can add the soapstock to the glycerine layer after separation for further treatment. See Chapter 12 “Glycerine”.

CONTENTS

METHYL ESTERS FOR BEGINNERS

Making biodiesel at home is easy, everybody can do it. Your diesel engine will run better and last longer on your home-made fuel, plus you don't have to worry about high fuel prices. It's much cleaner, better for the environment and your health. You can make it from fresh vegetable oil, or from waste vegetable oil (WVO) you can obtain from restaurants. If you make it from WVO it's not only cheap but you'll be recycling a troublesome waste product.

To make biodiesel fuel efficiently from used vegetable oils and animal fats we have to avoid one major problem: soap formation. Soap is formed during base-catalyzed transesterification (using lye) when sodium ions combine with free fatty acids present in waste (and some fresh) vegetable oils and animal fats. The soaps diminish the yield because they bond the methyl esters to water. The bonded esters get washed out at the washing stage but make water separation more difficult and increase water consumption.

The process

Vegetable oil and animal fats are triglycerides, which means, containing glycerol and fatty acid radicals. The biodiesel process involves breaking down the oil. This is done by adding it with lye and alcohol, turning oils and fats into esters, separating out the glycerine. The glycerine sinks to the bottom and the biodiesel or ester floats on top and can be siphoned off. Ester is the product which we commonly called biodiesel. The process is called transesterification, which substitutes alcohol for the glycerine in a chemical reaction, using lye as a catalyst.

We can use either methanol or ethanol as reactant. The end product will be called "Methyl Esters" if we use methanol, otherwise, it will be called "Ethyl Esters". This chapter will deal mainly with methyl esters.

Making biodiesel at home is simple. To make good quality biodiesel we need to understand exactly how the process works. Waste vegetable oils contain free fatty acids (FFA) of varying quantity, depending on the cooking temperatures and how long it's been heated. It is different from fatty acids in the sense that it is free, not bonded. It will react with lye to form soap. The amount of lye needed for complete reaction is therefore become deficient, resulting to incomplete reaction. The presence of un-reacted fatty acids in the finished product degrades its quality. The process therefore involves mainly of converting as much as possible free fatty acids into glycerine, and proper washing of biodiesel product. The first is done by determining the exact amount of lye needed in the reaction.

To gain solid understanding of how the process works, it is necessary to start with fresh unused oil**,** not with WVO. This will form part of your basic knowledge in biodiesel making. Using waste vegetable oil will be similar, except that you will need more lye. It will be the determination of the amount of the additional lye that will pose a challenge. But once you've mastered it, everything will just be fine. The process of mastering things always starts from the basics. Start by making a small, 1-litre test batch of biodiesel using fresh new oil before turning to waste vegetable oil and big batches.

Learn one step at a time. It's all quite simple really, very few biodiesel home brewers are chemists or technicians, there's nothing a layman can't understand, and do. But there is a lot to learn. You'll find everything you need to know right in this book. We've tried to make it easy for you. You start off with the simplest process that has the best chance of success and move on step by step in a logical progression, adding more advanced features as you go on.

Know how it works

Alcohol: Alcohol breaks down the vegetable oil into ester and glycerine. It is needed whether you use new vegetable oil or waste vegetable oil. You can use either methanol, which makes methyl esters, or ethanol which makes ethyl esters. In this chapter we will be using methanol. You will need 200ml of methanol per litre of vegetable oil (waste or new).

Lye: Lye is a type of chemical compound that propels the transesterification process faster. It is a catalyst that helps alcohol does its job. You will need lye catalyst whether you use new or waste vegetable oil. The lye can be either potassium hydroxide (KOH) or sodium hydroxide (caustic soda, NaOH).

NaOH is often easier to get and cheaper but more difficult to use.

KOH is easier to use, and it does a better job. Experienced biodieselers making good-quality fuel usually use KOH, and so do the commercial producers. KOH will produce potash fertilizer as a by-product of the biodiesel process. You will need 1.4 times more KOH compared to NaOH.

Refer to Chapter 6 for more information.

Titration: Titration is the process of determining the amount of additional lye needed in transesterification.

Refer to Chapter 7 for more information.

Experiment #1

This will be your first ever attempt to make biodiesel. You may repeat this step several times to get yourself acquainted with the process. Make everything according to the book. Make a journal handy and record everything you intend to do including what you have observed. Since this is your first biodiesel, you will be using new vegetable oil; hence, you will not be doing any titration.

The purpose of this step is for you to understand and familiarise with the basic process of transesterification. You will also learn where to get the chemicals needed. Notice that we specify the type of containers. The process involves chemical reaction which gives off heat. Some materials (aluminum, low density plastics, etc.) are just not practical.

Here are the ingredients and tools you need:

- 1 litre of new vegetable oil from the supermarket
- 200 ml of methanol, 99+% pure
- lye catalyst -- either potassium hydroxide (KOH) or sodium hydroxide (NaOH) . 3.5 grams NaOH or 5.78 grams KOH.
- scale accurate to 0.1 grams, preferably less -- 0.01 grams is best
- measuring beakers for methanol and oil
- 1/2 - litre translucent white HDPE container with screw-on cap
- 2 funnels to fit the HDPE container
- 2-litre PET bottle (water or soft-drinks bottle) for settling
- 2-litre PET bottles for washing
- 1/2-litre PET bottle for quality testing
- thermometer
- Blender
- pH metre

Make sure that all equipment is clean and dry. Do the entire process properly and safely. Read and understand “Safety” (Chapter 4) first before proceeding.

Mixing the methoxide: Measure out 200ml of methanol and pour it into the half-litre HDPE container via the funnel. Make sure you minimise exposure of methanol to the air as it quickly absorbs water. Do it quickly and replace the lid of the methanol container tightly. Work at an ordinary room temperature and keep it at arm's length. Don’t expose yourself to fumes. Make sure there is ready running water.

Carefully add the lye to the HDPE container via the second funnel. Replace the cap and screw it tightly. Shake the container a few times. Swirl it round gently, rather than shaking it up and down vigorously. The mixture gets hot from the reaction. Swirl it thoroughly until the lye is completely dissolved. KOH dissolves faster than NaOH. The resulting solution will be sodium methoxide or potassium methoxide. As soon as the liquid is clear with no undissolved particles you can begin the process.

The more you swirl the container the faster the lye will dissolve. With NaOH it can take from overnight to as little as half-an-hour with lots of swirling. Wait for all the lye to dissolve. Mixing KOH is much faster; it dissolves in the methanol more easily than NaOH and can be ready for use in 10 minutes.

Treat methoxide with extreme caution. Avoid inhaling any vapours. If any methoxide gets splashed on your skin, it will burn you without your feeling it. Wash immediately with lots of water. Always have a hose running when working with methoxide. Methoxide is also very corrosive to paints. Lye reacts with aluminum, tin and zinc. Use glass, enamel or stainless steel containers.

The part of the process where it's vital to keep all water out of the reaction is when making the methoxide. Keep the blender and all utensils the lye comes in contact with as dry as possible. The chances of a good clean splitting of esters from glycerine with little soap by-product are much better if water is completely kept out.

Mixing the ingredients: Make sure the blender you use in biodiesel processing will not be used in food processing again. Check that the blender seals are in good order. Make sure all parts of the blender are clean and dry and that the blender components are tightly fitted.

Pre-heat the oil to 55^{o}C and pour it into the blender. With the blender still switched off, carefully pour the prepared methoxide from the HDPE container into the oil. Secure the blender lid tightly and switch on. Lower speeds should be enough. Mix for 15 to 20 minutes.

Settling: As soon as the process is completed, pour the mixture from the blender into the 2-litre PET bottle for settling and screw on the lid tightly. Take note that biodiesel is also hygroscopic. Cover it tightly and allow to settle for 12-24 hours.

Darker-coloured glycerine by-product will collect at the bottom of the bottle, with a clear line of separation from the pale liquid above, which is the biodiesel. The biodiesel varies somewhat in colour according to the oil used (and so does the by-product layer at the bottom) but usually it's pale and yellowish.

Carefully decant the top layer of biodiesel into a clean jar or PET bottle, taking care not to get any of the glycerine layer mixed up with the biodiesel. If you do, re-settle and try again.

Quality test: Put 100 ml of unwashed biodiesel in a half-litre glass jar or PET bottle. Add 150 ml of water screw the lid on tight and shake it up and down vigorously for 10 seconds or more. Then let it settle. The biodiesel should separate from the water in half an hour or less, with

amber biodiesel on top and milky water below. This is quality fuel, a completed product with minimal contaminants. Proceed to washing the biodiesel.

But if it turns into something that looks milky and won't separate, or if it only separates very slowly, with a creamy white layer sandwiched between water and biodiesel, it's not quality fuel and your process needs improvement.

Possible sources of error include:

1. You might have used too much lye and made excess soap. You should be very careful in measuring the lye next time.

2. Poor conversion has left you with half-processed mono and diglycerides, fuel contaminants that act as emulsifiers. Try either using more methanol, better agitation, longer processing time or better temperature control

3. Or it could be both too much lye and poor conversion.

Whichever, you're headed for washing problems. You might get away with washing but you'll still be left with poor-quality fuel with contaminants that can cause engine damage in the long run.

The best remedy will be to reprocess your biodiesel. Restart from the beginning as if using new oil, but use only 100ml of methanol per litre of failed fuel.

Washing: If it passes the quality test then wash the rest of the biodiesel using the 2-litre PET bottle. Put biodiesel in the PET bottle, including the one used in quality testing (including the water), add 350ml of tap water, and then shake it up and down vigorously for ten seconds or more. Allow to settle for two hours between washing. Check the pH of the water. Remove the water, doing it carefully not to include the biodiesel. Add 500ml of tap water and repeat the process above, until the pH of water reaches 7 or the same with that of tap water.

Drying: When it's clear (not colourless but translucent) it's dry and ready to use. It might clear quickly, or it might take a few days or up to a week. If you're in a hurry, heat it gently to 48^{o}C and allow to cool. Your biodiesel is now ready to use.

You have just made high-quality diesel fuel, but it’s only 1 litre. Considering that you will be using waste vegetable oil, this will just be your starting point for bigger things. However, you might consider extracting oil yourself from some raw materials, say, jatrupha or coconut. Then you can use exactly the same process as detailed here.

Experiment #2

This will be your first ever attempt to make biodiesel using waste vegetable oil. Again, you may repeat this step several times to gain mastery of the process. Even if your experience has already built up, you will still be doing this step from time to time as a trial or test batch. As in experiment number 1, make everything according to the book. Make a journal handy and record everything you intend to do including what you have observed. You will be doing titration for the first time. Make sure to do it right the first time.

You’ve learned in the previous experiment where to get the chemicals. Here you will learn where to get waste vegetable oil. It is a waste matter. Restaurants are paying a fortune to dispose it. You should be able to get it for free. Best sources are fast food restaurants.

Aside from the ones needed for titration, you will be using the same tools and materials as in experiment number 1. Learn how to improvise to make the process more simple and safe. Here are the ingredients and tools you need:

- 1 litre of waste vegetable oil
- 200ml of methanol, 99+% pure
- lye catalyst -- either potassium hydroxide (KOH) or sodium hydroxide (NaOH)
- scale accurate to 0.1 grams, preferably less -- 0.01 grams is best
- measuring beakers for methanol and oil
- 1/2 -litre translucent white HDPE containers (with screw-on cap)
- 2 funnels to fit the HDPE container
- 2-litre PET bottle (water or soft-drinks bottle) for settling
- 2-litre PET bottles for washing
- 1/2-litre PET bottle for quality testing
- thermometer
- Blender
- Syringes
- pH metre or phenolphthalein solution
- Filter
- Pure isopropyl alcohol for titration

Make sure that all equipment is clean and dry. Do the entire process properly and safely. Read and understand “Safety” (Chapter 4) first before proceeding.

Preparing the oil: WVO contains bits of pieces of food. It could also contain water. Water in the oil will interfere with the lye, especially if you use too much lye. It will slow down the reaction and cause saponication, and you'll end up with jelly. The less water in the WVO the better.

Test first for water content, heat the WVO in a saucepan on the stove and monitor the temperature with a thermometer. If there's water in it, it will start to produce crackling or popping sounds by 50^{o}C or so. If it's still not crackling by 60^{o}C there's no need to dewater it.

If it crackles, raise the temperature to 100^{o}C, hold it there and allow any water to boil off. Stir the WVO occasionally to avoid steam pockets forming below the oil and exploding, splashing hot oil out of the container. When boiling slows, raise the temperature to 130^{o}C for 10 minutes. Remove heat and allow to cool. Then filter it through a fine mesh filter to remove bits of pieces of food. Use a double layer of cheesecloth in a funnel, or a restaurant or canteen-type coffee filter. Measure 1 litre for the process.

Take note that this WVO preparation does not consider neutralizing the FFAs.

Titration: Waste vegetable oils contain Free Fatty Acid (FFA). FFAs are formed in cooking the oil, the longer and hotter the oil has been cooked the more FFAs it will contain. FFAs interfere with the transesterification process for making biodiesel. With waste oil you have to use more lye catalyst to neutralise the FFAs. The extra lye turns the FFAs into soap which drops out of the reaction along with the glycerine by-product.

Refer to Chapter 7 for further details of doing it.

Mixing methoxide: Proceed as with new oil with quantity of lye as measured according to the results of titration. Mix the lye with the 200ml of methanol in a strong, heatproof glass bottle with a narrow neck to prevent splashing. Use closed containers for mixing methoxide. Take full safety precautions when working with methoxide, have a source of running water handy. It will get slightly hotter and take a little longer to mix as there's more lye this time. Make sure the lye

is completely dissolved in the methanol. Treat methoxide with extreme caution. See experiment number 1.

Mixing the ingredients: Use the same blender you used in experiment number 1.

Check the temperature of your 1 litre WVO. The temperature should be around 45 to 55^{o}C so it will mix better. Don't let it get too hot or the methanol will evaporate. Methanol boils at 65^{o}C. Blenders are very thorough at mixing the ingredients so heating is not as critical.

Add carefully the methoxide to the heated oil then start the blender. Blender batches need only be run for about 15-20 minutes for mixing to be completed before switching off. The settling takes some time to complete. The solution can be poured from the blender into another container right after switching off the blender.

Settling, quality test, washing and drying: Let it settle for 12 to 24 hours. Remove the newly-made biodiesel, leaving the glycerine by-product. Make sure no glycerine is included in the processed biodiesel.

Proceed with quality test, washing and drying as with new vegetable oil (experiment number 1). Problems do usually occur but mostly can be solved by making more accurate measurements. Do the titration two to three times to get better results. Do everything you can to improve the accuracy of your measurements so you get consistent results.

It is a good idea to do a few batches with varying amounts of lye. Later when checking results one can choose the lye quantity that did the best job. When too much lye is used the result can be some soap formation. When not enough lye is used the reaction does not go far enough so some unreacted WVO will be mixed with the biodiesel and glycerine. This will form three levels with biodiesel on top above unreacted WVO with glycerine on the bottom. If there is too much water in the WVO it will form soaps and settle right above the glycerine forming a fourth level in the container. This layer is not too easy to separate from the unreacted WVO and glycerine layers.

Experiment # 3 (the same with experiment number 2 but using 20 litres WVO)

This will be your first ever attempt to do the real thing. As in experiments number 1 and 2, make everything according to the book. Make a journal handy and record everything you intend to do including what you have observed.

You've learned in experiment number 1 the basic process of making biodiesel. Then you made your trial batch in experiments number 2. You will be using the results you obtained in experiment number 2 here.

Preparing the oil: When you do your trial batch (experiment #2) you must have secured enough quantity of WVO to include your requirements for experiment #3. Different restaurants have different cooking methods, hence, different amounts of FFAs. If you are souring WVO from many different restaurants, you may mix them together in a large container so that you will only make single titration and single trial batch. You may make several titrations and several trial batches but you only use one respective reliable result. You should be using the same batch of WVO as in experiment #2 to avoid performing another titration.

Before you mix WVO from different sources you should filter them first and checked them for water content. As stated above different restaurants have different cooking methods and food requirements. Some WVO might contain water, some might not. You will save time and energy if you treat only those WVO which need treatment. You may combine those needing treatment and those that are not.

Take note that WVO preparation for this experiment does not consider neutralising the FFAs.

Refer to Chapter 7 for further information in preparing the WVO.

Ingredients and Tools: You will be using similar tools as in experiment experiment #2, but much bigger. Learn how to improvise to make the process more simple and safe. Here are the ingredients and tools you need:

- 20 litres of prepared WVO
- 4 litres of methanol, 99+% pure
- lye catalyst -- either potassium hydroxide (KOH) or sodium hydroxide (NaOH) quantity depends on the result of titration
- scale accurate to 0.1 grams, preferably less -- 0.01 grams is best
- measuring equipment for methanol and oil
- 1 10-litre HDPE container, preferable with narrow neck for mixing methoxide
- 1 40-litre steel drum for processing and settling
- 2 40-litre HDPE drum for washing
- 1/2-litre PET bottle for quality testing
- thermometer
- Filter

Make sure that all equipment is clean and dry. Do the entire process properly and safely. Read and understand “Safety” (Chapter 4) first before proceeding.

Titration: You will not be doing titration here. It is already done in experiment number 2.

Mixing methoxide: Proceed as with new oil with quantity of lye as measured according to the results of titration. Mix the lye with the 4 litres of methanol (200ml per litre of WVO) in a 10-litre strong, heatproof container with a narrow neck to prevent splashing. Use closed containers for mixing methoxide. Take full safety precautions when working with methoxide, have a source of running water handy. It will get slightly hotter and take a little longer to mix as there's more lye this time. Make sure the lye is completely dissolved in the methanol.

Mixing the ingredients: The blender you used in experiment #2 and 3 might be too small for this project. You need to improvise your own blender using a variable speed electric drill. Or you may use a small pump (aquarium air pump, etc.) to let the mixture circulate by draining it from the bottom of the reactor and then pumping it back to the reactor through the top.

Warm the 20 litres of prepared WVO in a 40-litre steel drum to about 45 to 55^{o}C to thin it so it will mix better. Don't let it get too hot or the methanol will evaporate. Methanol boils at 65^{o}C.

Improvise a jig (wood or steel) with a portable vice clamped to it holding a power drill fitted with a paint mixer to stir the contents of the drum. Make sure that the drum can be closed. You can do this by making a hole in the drum cover in such a way that the rod of the stirrer can pass through.

Add carefully the methoxide to the heated oil then start stirring. Keep stirring for an hour keeping the temperature within the range of 45 to 55^{o}C then let it settle for 12 to 24 hours.

Settling and separation: The methyl esters (biodiesel) will be floating on top while the denser glycerine will have congealed on the bottom of the container forming a gelatinous mass. Siphon off newly processed biodiesel using a clear hose, leaving glycerine by-product in the bottom of the drum. Make sure no glycerine is included in the processed biodiesel.

An alternative method is to reheat the reactants to around 40^{o}C making sure they don’t re-mix. Drain the reactants out of the bottom of the container through a transparent hose. The semi-

liquid glycerine has a dark brown colour; the biodiesel is honey-coloured. Keep watch on what flows through the tube. When the lighter-coloured biodiesel appears divert it to a separate container. The little amount of biodiesel that goes with the glycerine can be retrieved later once the glycerine has hardened or thickened. Sometimes it will solidify, sometimes not.

Quality test: Proceed with quality test as in experiment #1. Problems do usually occur but mostly can be solved by making more accurate measurements. Do the titration two to three times to get better results. Do everything you can to improve the accuracy of your measurements so you get consistent results. The production rate is less than with new oil, usually 8-9 litres of biodiesel per 10 litres of WVO. With care and growing experience the production rate will improve.

Washing and drying: Washing and drying the biodiesel will be different in experiment #1, simply because we are now dealing with much larger quantity. Refer to Chapter 11 for the methods of washing you may use.

Congratulations. You have just made your own high-quality biodiesel. Fill it in your car and go. Remember, you don't have to visit the gas station again. With your own effort you don't have to worry about ever increasing fuel prices plus you have contributed much to improving our environment. You eased the burden of restaurant operators of treating their wastewater, you help the government in reducing dependency on foreign oil plus you helped eliminate hydrocarbon emissions.

CONTENTS

Experiments #1 to 3 in Chapter 8 where arranged to provide you with step by step learning process of making biodiesel at home. It emphasizes with properly doing the titration. As your knowledge increases and you have become more familiar with the qualities of the ingredients in your area, you can now start experimenting making biodiesel without doing titration. Take note that experiments #1 to 3 did not consider deacidifying the WVO, or neutralizing or transforming the FFAs.

The process will be more or less the same but this is more simplified and used more alcohol. This process involves with the pre-treatment of FFAs hence, titration is no longer needed. This process is specifically useful when dealing with WVO with high FFA content but can also be used with WVO of less FFA content.

Please take note that the methods presented in this book were patented. You may use any of these methods for education purposes or for your own personal consumption. If you decide to produce biodiesel of commercial quantity, you should contact the owner of the patent or improvise your own process.

Test batches

Whenever you're trying a new method, it's always a good idea to make small test batches of a litre or less first to familiarise yourself with the process before moving on to bigger batches. Most people use kitchen blenders for this but don't use it for food again afterwards.

WVO preparation

Filter the used cooking oil first as usual. For a successful reaction the oil must be free of water. Refer to Chapter 7 for more information regarding pre-processing of WVO.

There are several methods of pre-treating FFAs but this book will consider only the two simplest ones. The first method uses acid catalyst to transform FFAs into esters and have it converted to biodiesel. The second method uses base catalyst to convert FFAs to soap and let it settle down and separated before proceeding to transesterification. The first method obviously produces more yield than the second one, but the second method will be handy if you don’t have acid.

Safety

Wear proper chemical-proof gloves, apron, and eye protection and do not inhale any vapours. Methanol can cause blindness and death, and you don't even have to drink it, it's absorbed through the skin. Sodium hydroxide can cause severe burns and death. Together these two chemicals form sodium methoxide. This is an extremely caustic chemical. Always have a hose running when working with them. The workspace must be thoroughly ventilated. No children or pets allowed. See “Safety” (Chapter 4) for further information.

Experiment #4

This experiment will use acid catalysis in dealing with FFAs. The alcohol is still methanol, but instead of using lye (sodium hydroxide), the catalyst in this reaction is sulfuric acid. It needs at least 95% sulfuric acid. Do not use battery acid because it is only around 50% pure.

The transesterification stage uses lye, as usual, but it only uses about half as much as experiments #1 to 3. The sulfate ion in the sulfuric acid combines with the sodium ion in the lye

during the second-stage reaction to form sodium sulphate, which is a water-soluble salt and is removed in the wash. No sulfur remains in the biodiesel fuel product.

You don't need a special processor for this method but you can design your own after you have thoroughly understood the process. Generally, a bottom-drained reactor vessel is best, closed on top. Tall, narrow containers work better than wide, shallow ones. Use a circulating pump for mixing rather than a mechanical stirrer. The pump should take the mixture from near the bottom of the reactor and return it via the top, to splash down on the surface.

You will also need an immersion heater for heating the solution. You could use a thermostat to control the temperature, but they're expensive: just use a thermometer and switch on the heater as required.

Ordinary iron and steel implements and containers will eventually corrode because of the acid used in this process. However, the proportion of acid used in this process is very low. You should be able to use an uncoated drum for a year or more before the rust gets out of control. Any plastic container that won't deform at 100^{o}C or a bit more is fine. Stainless steel is also fine. Use an immersion heater with plastic containers. With steel containers you can use propane heaters to heat the oil, and then switch to an immersion heater before adding the methanol.

Here's how it is done:

1. Measure 20 litres of WVO to be processed and put it in the reactor.
2. Heat the oil to 35^{o}C.
3. Methanol: use only 99%+ pure methanol. Measure out 1600 ml methanol (8% by volume). Add the methanol to the heated oil.
4. Mix for five minutes. The mixture will become murky because of solvent change. A suspension will form.
5. Add 20 ml of 95% sulfuric acid (H_2SO_4), 1 ml per litre of WVO. Use a graduated eyedropper, a graduated syringe or a pipette. Take care when handling the concentrated sulfuric acid.
6. Mix gently (low speed if using a mechanical stirrer) while keeping the temperature at 35^{o}C.
7. Maintain the temperature at 35^{o}C for one hour then stop heating. Continue stirring.
8. Stir the mixture for another hour, a total of two hours, then stop mixing. Let the mixture sit for at least eight hours, overnight is better.
9. In the meantime prepare the sodium methoxide: measure 2.4 litre of methanol (12% by volume) and weigh 70 grams of sodium hydroxide (NaOH), 3.5 grams per litre of WVO. Weigh the lye carefully. Using too much will complicate the washing process later. Mix the lye into the methanol until the lye is completely dissolved. Take note that sodium methoxide is a dangerous chemical. Take full safety precautions when working with methanol, lye and sodium methoxide, wear safety goggles, protective gloves and clothing, have running water nearby. Refer Chapter 4 for safety requirements.
10. After settling for eight hours, or the next morning, pour half of the prepared methoxide into the unheated mixture and mix for five minutes. This will neutralise the sulfuric acid and boost the base catalysis. If you've used solid fat, it probably solidified during settling, gently melt the mixture first.
11. Heat the mixture to 55^{o}C and maintain for the whole reaction.
12. Add the second half of the prepared sodium methoxide to the heated mixture and start mixing at the same low speed. Take care when handling the sodium methoxide.
13. Take regular samples in a glass container. Watch for a straw yellow colour of the ester portion. When this colour is reached (usually in 1.5-2.5 hours) turn the heat and mixer off. Instead of taking out samples to check the colour you could use translucent braided tubing for the pump.
14. Allow to settle for at least 12 hours. Glycerine (brown and sticky) will settle at the bottom of the jar.
15. Drain off the glycerine and put in a sealed container.

Washing:
Refer to Chapter 11 “Washing” for further details about washing biodiesel. Just add a little 10% phosphoric acid (H_3PO_4) to the washing water first, (2-3 ml/litre). Checking the pH is not necessary but if you are curious about the results of your wash, the end result should be neutral (pH7) or just below neutral.

Drying:
Let the biodiesel stand for about three weeks and use only when it has completely cleared. If you want quicker results, heat the biodiesel to 45^{o}C, and let it cool. A deposit may form in the bottom during settling. Don't let it get in your fuel tank.

You’ve just made a good quality biodiesel. In steps 1 to 10, free fatty acids were esterified and some triglycerids were transesterified. Steps 11 to 14 do only transesterification, but it's much quicker and more complete. A quick note for steps 1 to 10: do not mix methanol directly with sulfuric acid before adding the WVO. Two major reasons: (a) the reaction between methanol and concentrated H_2SO_4 is quite violent and it could splash, which doesn't happen if you mix it as described; and (b) dimethyl ether can form. Dimethyl ether is a colourless gas and highly explosive.

The product in steps 11 to 15 should be quite murky. This is no problem, as it will wash out. After the processed WVO has turned straw-yellow and you've let it settle for an hour and drained the glycerine, you should have a total of about 120 ml of glycerine per litre of WVO used. If it's less than about 100 ml/litre of WVO, something's wrong, even if the colour is right. The process hasn't gone far enough.

This will almost certainly be due to carbonated lye. Lye has a really limited shelf life. CO^2 from the air neutralises it and forms sodium carbonate. Carbonated lye is much whiter than pure lye, which is almost translucent. The carbonate in the lye won't harm the reaction, but you'll have to use more lye.

The solution is to repeat steps 11 to 15 process. Prepare a fresh batch of methoxide with 0.03 litres of methanol and 0.75 grams of lye for each litre of WVO. Reheat the biodiesel to 55^{o}C, add the fresh methoxide and mix as before. Don't worry about the colour this time. Mix for one hour, settle, drain off the extra glycerine, and proceed to washing and drying.

If you plan to continue using the carbonated lye in your next batches, make sure to increase the amount by 25%. Store lye at room temperature, in dry conditions if possible, with the container lid really tightly closed.

Experiment #5

This experiment will use base catalyst in dealing with FFAs. The base catalyst will transform FFAs into soap and be separated before transesterification.

As with experiment #4, you don't need a special processor but you can design your own after you have thoroughly understood the process. Generally, a bottom-drained reactor vessel is best, closed on top. Tall, narrow containers work better than wide, shallow ones. Use a circulating pump for mixing rather than a mechanical stirrer. The pump should take the mixture from near the bottom of the reactor and return it via the top, to splash down on the surface. However, mechanical stirrer works just fine.

You will also need an immersion heater for heating the solution.

Any plastic container that won't deform at 100^{o}C or a bit more is fine. Stainless steel is also fine. Use an immersion heater with plastic containers. With steel containers you can use propane heaters to heat the oil, and then switch to an immersion heater before adding the methanol.

Here's how it is done:

1. Measure 20 litres of WVO to be processed and put it in the reactor.
2. Prepare your methoxide: mix 5 litres (25% by volume of WVO) of pure methanol and 135 grams (6.25g/litre of WVO) of sodium lye (NaOH).
3. Heat the WVO to 48-52^{o}C.
4. Add 3/4 of the prepared methoxide (save the rest in a sealed container out of reach of children and flames or sparks).
5. Mix for 50-60 minutes holding the initial temperature.
6. Let the mixture rest or settle for 12 hours.
7. Separate the glycerine from the FAME.
8. Heat the FAME to 55^{o}C and maintain for the whole reaction.
9. Add the remaining 1/4 methoxide.
10. Mix for 50-60 minutes holding the initial temperature.
11. Let the mixture rest or settle for 12 hours.
12. Drain off the glycerine and combine it with glycerine in step 7.
13. Proceed to washing and drying as described in the acid catalysis.

You will notice that the conversion using the base catalysis is lesser when compared to acid catalysis.

Methanol recovery from FAME:
Methanol recovery is optional. But to keep costs down, even amateur biodiesel producers try to salvage the unreacted methanol. You may do this after you drain off the glycerine and before washing. You can do this using the heat extraction method. Heat the second-stage product to 70^{o}C in a sealed boiler/vessel and lead the fumes into a condenser. Intercept the condensed methanol in a liquid trap. Take great care because methanol is highly flammable and the fumes are explosive. The temperature will need to be increased as the amount of unreacted methanol in the mixture decreases. Another way of doing this is by vacuum/heat extraction method. This is basically the same as heat extraction, but it requires less energy. The drawback of this method is that you need a special vessel and equipment to do this.

Quality:
Diesel engines require quality fuel. Poor quality biodiesel could contain free glycerine, poorly converted oils/fats and lye; three very dangerous enemies of a diesel engine. Free glycerine and poorly converted oils/fats will form gum-like deposits around injector tips and valve heads. Lye can damage the injector pump. The key to good fuel is to just do it right and finish it. Use pure chemicals (sulfuric acid, sodium lye and methanol) and measure them accurately. Follow the instructions carefully to avoid poor conversions. Proper washing will get rid of the glycerine and neutralise any remaining lye.

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ETHYL ESTERS

Making ethyl-esters biodiesel using ethanol is a tricky process, not as simple as making methyl esters with methanol. Ethyl esters process doesn't work well with oils with more than about 2 ml titration. It's not for beginners. Learn how to make biodiesel with methanol first and get yourself acquainted with titration.

Ethanol provides the advantage of making a Biodiesel fuel produced entirely from renewable resources. But the use of ethanol in Biodiesel production has not been studied as extensively as has methanol.

A method developed by Peterson et al 1996, was used to produce the ethyl esters with slight modifications adopted from other methods. Peterson's experiment used rapseed oil. The quantities used were:

EtOH = 0.2738 x Oil
KOH = Oil/85

Where:

Oil represents the desired amount of WVO in litres,
EtOH is the amount of ethanol in litres and
KOH is the amount of potassium hydroxide require in Kg.

These equations are based on the theoretical requirements. The quantity of KOH catalyst must be increased according the quantity of FFAs present in the WVO. Alternatively, you may do pre-treatment of the WVO to reduce or neutralise the FFA present.

Waste vegetable oil

In using ethanol, the WVO will have to be fairly low in FFAs. Anything over 2 ml titration (using 0.1% NaOH solution) can cause failure of the glycerine to separate. Titration under 1 ml is a good idea. Most WVO contains too much FFAs for ethanol to work well. Pre-treatment of WVO to reduce the FFA content might be necessary. Treating it with acid catalyst to transform FFAs into esters might be worth a try. Pre-treatment using a base catalyst might also work, but the base catalyst converts the FFAs into soap, thereby reducing the conversion efficiency.

The original oil must be water-free, because every molecule of water destroys a molecule of catalyst, thus decreasing its concentration. To prepare the oil, heat the oil to 120^{o}C and hold it there until you can turn off the flame and see the bubbling stop almost immediately. Sometimes that's still not dry enough. Any more than 0.5% water can kill the reaction.

Ethanol

Ethanol will have to be very dry, 199-proof or higher. The nearer to absolute (200 proof), the better. Ethanol that is very dry is difficult to find and it is not cheap. Gasoline present in the alcohol as a denaturant appears to do no harm. The reaction proceeds satisfactorily in mixtures of 200-proof ethanol with 10%(v/v) or more gasoline present. However, even small quantities of water (less than 1%) can decrease the extent of the conversion reaction enough to prevent the separation of glycerol from the reaction mixture.

Potassium Hydroxide Catalyst

KOH should be at least 85% pure. Even the best grades of KOH have 14 to 15% water (which cannot be removed). It should be low in carbonate, because potassium carbonate does not serve as a satisfactory catalyst, and may cause cloudiness in the final ester. Try to find a source of KOH (potassium hydroxide) to use instead of Sodium Hydroxide with ethanol. Sodium hydroxide (NAOH) is not a suitable catalyst because it is not sufficiently soluble in ethanol and it tends to promote undesirable gel and emulsion formation during transesterification.

KOH must be protected as much as possible from atmospheric moisture and carbon dioxide contamination.

Procedure

1. Measure WVO and heat it to 40 to 50^{o}C.

2. Do the titration and determine the amount of extra KOH needed.

3. Prepare the ethoxide. Dissolve the catalyst with alcohol by vigorous stirring in a small reactor. Make sure that the catalyst is fully dissolved.

4. Transfer the heated WVO into the Biodiesel reactor and then add the catalyst/alcohol mixture.

5. Stir the mixture vigorously for 6 hours.

6. Initial settling: In a good completed reaction, the glycerol begins to separate immediately upon cessation of stirring, and the settling mostly complete in one hour.

7. After initial settling, the entire contents of the reaction vessel are again mixed together and stirred vigorously for 40 minutes. After the first 20 minutes of re-stirring, water is added at 15% of the initial volume of oil used in the reaction. Stirring should continue an additional 20 minutes after the water is added or a total of 40 minutes of re-stirring. Remixing the glycerol layer with the ester layer while adding water has the effect of collecting and removing impurities and products of incomplete reaction from the ester. The washing phase can then proceed at a more rapid pace than if the remixing stage were left out.

8. This mixture is then allowed to settle for 10 hours or more. A longer separation time facilitates the washing process.

   In batches where poorer quality (moisture-containing) ethanol is used the reaction will not go to completion and requires much longer for the glycerol to separate. If separation does not occur, the addition of a small amount (perhaps 10% of the original volume) of alcoholic KOH with stirring may tip the reaction balance in favor of separation. It is also sometimes possible with the addition of a small amount of water (0.5% of the total volume) after the reaction is supposedly completed, to effect the separation of the glycerol from the ester. If the original ethanol contains as much as 1% water, the reaction may be so incomplete that the glycerol may never separate, and the entire batch must be discarded.

9. After remixing the glycerol and 15% water addition, and completion of the separation, the lower, heavier glycerol/water layer is drained off, leaving the ester in the reactor.

10. Finally, in order to remove the remaining alcohol and trace amounts of potassium, glycerol or soap, the ester is washed with water at about 30% of the ester volume. The water is stirred into the ester with a combination of mechanical stirring and air agitation.

    For mechanical stirring, use a mechanical stirrer whose rotation can be strictly controlled. The best speed for the equipment used has been about 50 to 70 RPM. The stirrer shaft should have two blades with one in the water phase and one in the ester phase rotating to lift the solution upward. This orientation, along with aeration develops maximum contact between ester and water.

    Air agitation is accomplished by introducing deep into the water layer numerous air bubbles. These numerous water coated bubbles rise through the liquid interface into the ester, carrying large amounts of water in the film, and accomplishing washing as they rise up through the ester. Upon reaching the surface, the bubbles burst and form droplets of water which fall back down through the ester, further washing it.

11. After a few hours the stirring/aeration is stopped and the water is allowed to settle out for two to three days. At this point the process is complete and the crystal clear product can be pumped into fuel tanks for storage or immediate use.

Take note however that the procedure presented here is based on Peterson's process which used rapseed oil. Using the WVO might be different and it might be necessary to vary the quantities of ingredients. Using ethanol in producing biodiesel is not widely used, but it is worth experimenting because ethanol can be produced using renewable resources and it is not as toxic as methanol.

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WASHING

There are a number of water-soluble impurities left in biodiesel after the reaction and initial settling is complete. They are found in everyone's homebrew biodiesel, regardless of the quality of your reaction or of your oil. Biodiesel must be washed before use to remove soaps, excess methanol, residual lye, free glycerine and other contaminants. Quality biodiesel is well-washed biodiesel. Filtering it is no use, and letting it settle for a few weeks won't help much either. Washing the fuel with water removes these contaminants. Washing also has two additional advantages: (a) it stops the very slow remaining reaction that sometimes occurs (in unwashed fuel you can sometimes see glycerine settle out over a period of weeks or months, and washing and removing methanol or lye will stop this); and (b) it provides you with some quality control feedback.

All washes can be done after the initial settling time (at least 12 hours) and after draining the glycerine layer, but you can get better results by waiting at least 36-48 hours or longer before the first wash, so that soaps and free glycerine have more of a chance to settle out.

Quality check

Before washing the biodiesel, it is best to check first if it has undergone complete reaction. If you wash biodiesel which reaction has been stopped pre-maturely or when the reaction has not been completed, you are heading to emulsion problems. When this happens, you are going to have trouble washing.

To do this put 100 ml of unwashed biodiesel in a half-litre glass jar or PET bottle. Add 150 ml of water screw the lid on tight and shake it up and down vigorously for 10 seconds or more. Then let it settle. The biodiesel should separate from the water in half an hour or less, with amber biodiesel on top and milky water below. This is quality fuel, a completed product with minimal contaminants. Proceed to washing the biodiesel.

But if it turns into something that looks milky and won't separate, or if it only separates very slowly, with a creamy white layer sandwiched between water and biodiesel, it's not quality fuel and your process needs improvement.

Possible sources of error include:

- You might have used too much lye and made excess soap. You should be very careful in measuring the lye next time.
- Poor conversion has left you with half-processed mono and diglycerides, fuel contaminants that act as emulsifiers. Try either using more methanol, better agitation, longer processing time or better temperature control
- Or it could be both too much lye and poor conversion.

Whichever, you're headed for washing problems. You might get away with washing but you'll still be left with poor-quality fuel with contaminants that can cause engine damage in the long run. The best remedy will be to reprocess your biodiesel. Restart from the beginning as if using new oil, but use only 100ml of methanol per litre of failed fuel.

Washing with water

One method is to wash the soaps out of the fuel with water, one or more times. When washing biodiesel the first time it's best to add 3ml of acetic acid (vinegar) for every 10 litres of biodiesel

before adding the water. The acetic acid brings the pH of the solution closer to neutral because it neutralises and drops out any lye suspended in the biodiesel.

Put the unwashed biodiesel in the washing tank. Add 500ml of water for every litre of biodiesel. Using a mechanical stirrer or your improvised stirrer, mix the water/fuel mixture to the point of appearing homogenous for about 10 to 20 minutes, followed by 1 to 2 hours of settling. The biodiesel and water will separate. Remove the water from the bottom, and then check the pH of the water.

This process might have to be repeated two or three times to remove close to 100% of soaps. The succeeding washings can be done with water alone. During the last washing, let it settle for at least 12 hours, then drain out the water. After the last washing, let any remaining water gets removed by re-heating the oil slowly. The water and other impurities sink to the bottom. Washing is done when the pH reading reaches 7 or very close to 7.

The water from the third and succeeding washes can be used for the first or second washes for the next batch. Used water from biodiesel washing can be allowed to settle down. Left-over biodiesel can be re-claimed, and the soaps can be processed further into something useful, e.g., industrial-type degreasers, etc.

Transesterified and washed biodiesel will become clearer over time as any remaining soaps drop out of the solution.

Bubble washing

Bubble washing technique was invented by Idaho University. It takes longer, but uses less water. It is very effective, giving a clean, polished product.

It is done by using an aerator usually used in aquariums. The bubbles are formed by compressed air passing through an air stone. Add about 3ml of vinegar (acetic acid) per 10 litres of biodiesel and then add about 50% water. Make sure the water and the ester have roughly the same temperature. Let the air stone sink to the bottom of the tank. When you switch on the aerator, the air bubbles rise through the biodiesel, carrying a film of water which washes the biodiesel as it passes through. At the surface, the bubble bursts, leaving a small drop of water which sinks back down through the biodiesel, washing again. If the mixture is still cloudy after a couple of hours, add a little more vinegar.

Bubble-wash for 12 hours or longer (up to 24), then drain off the washing water, skim off any wax floating on top. Check the pH of the water. Repeat the bubble wash two more times until the pH reaches 7 or very close to 7. You may keep the water from the 2nd and succeeding washes for washing the next batch.

The drawback of using bubble wash is that it can cause a more complicated problem. Aquarium air pump is designed to replenish the oxygen in the water in the aquarium by introducing tiny air bubbles through the bottom of the aquarium. By introducing the same tiny air bubbles to the biodiesel, we are inviting a problem about oxidation. When oxidised biodiesel dries it becomes polymerised into a tough, insoluble plastic-like solid. At the high temperatures in internal combustion engines the process is accelerated. Steadily accumulating films of tough, insoluble plastic-like solids in your engine and injector pump.

Saturated oils don't polymerise, unsaturated oils do. The level of unsaturation is called the Iodine Value (IV). The higher the IV the more unsaturated the oil, the faster it will oxidise and the more it will polymerise. Coconut oil has an IV of 10 and won't polymerise.

Bubble washing is preferred by many biodiesel home brewers because it uses “gentle stirring” which will likely produce less emulsification problems. However, gentle stirring does not solve any problem about incomplete reaction. It just hides it.

Mist washing

Mist washing was developed as a way to address emulsification issues. It uses more water and more complicated equipment. This system uses a very fine mist head from a garden supply store, suspended over a container of biodiesel, with a way to drain the water after it falls through the fuel. The mist stirs up the fuel less than in bubblewashing, and removes soaps gradually. The gentler agitation gives less of an opportunity for the soaps and any mono and diglycerides to form emulsion.

The disadvantages of misting are higher water consumption, more complicated equipment, and the masking of potential problems. You won’t know if your biodiesel was the result of an incomplete reaction.

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GLYCERINE

In the transesterification process, oils and/or fats rich in triglycerides are mixed with an alcohol such as methanol and base such as potassium or sodium hydroxide, resulting in a methyl ester biodiesel main product and a glycerine byproduct. This glycerine byproduct typically contains a mixture of glycerine, methanol, water, inorganic salts (catalyst residue) free fatty acids, unreacted fatty acids, methyl esters, and a variety of other organic matters in varying quantities. The methanol is typically stripped from this byproduct and reused, leaving behind, after neutralization, what is known as crude glycerine. In raw form, this crude glycerine has high salt and free fatty acid content and substantial colour (yellow to dark brown). Consequently, crude glycerine has few direct uses due to the presence of the salts and other species, and its fuel value is also marginal.

The name glycerol is preferred for the chemical, but commercially the product is usually called glycerine, sometimes spelled glycerine, which are interchangeably used. The name comes from the Greek word *glykys* meaning sweet. Pure glycerine is a colourless, odourless, viscous, nontoxic liquid with a very sweet taste and has literally thousands of uses. The biodiesel byproduct is crude and it's not colourless.

Why isn't it solid?

It's mainly the soaps combined with the glycerine that can cause it to solidify. Soaps made from saturated fats are harder than those made from unsaturated fats, so the type of oil used can sometimes makes a difference. More important is how much soap there is. The more soap, the more likely the by-product layer will solidify, no matter what oil you used.

Other factors:

- Excess methanol makes the by-product layer thinner
- Too much lye creates excess soap
- Using potassium hydroxide (KOH) as the catalyst instead of sodium hydroxide (NaOH) produces a liquid by-product that won't solidify.

The glycerine from WVO is brown and usually turns to a solid below about 38^{o}C. Glycerine from fresh oil often stays liquid at lower temperatures.

Reclaiming excess methanol

Depending on the kind of oil you're using, it takes from 110-160 millilitres of methanol per litre of oil to form the methyl esters molecule. But you also need to use an excess of methanol to push the conversion process towards completion. The total used is usually 20% or more of the volume of oil used.

Much of the excess methanol can be recovered after the process for reuse, simply by boiling it off in a closed container with an outlet leading to a simple condenser. Methanol boils at 64.7^{o}C, though it starts vapourizing well before it reaches boiling point.

Unlike ethanol, methanol does not form an azeotrope with water and relatively pure methanol can be recovered, pure enough to reuse in the next batch. The methanol can be recovered at the end of the process, or just from the glycerine by-product layer, since at least 70% of the excess methanol collects in the by-product.

It's best to recover the methanol from the glycerine by-product. To do this, heat the glycerine to 65-70 ^{o}C in a close container connected to a condenser. As the methanol evaporates, leaving

an ever-lower proportion of methanol in the mixture, the boiling point will increase, so you'll have to keep raising the temperature to keep the methanol vapourizing. It goes up to 100 ^{o}C or more, and then it starts to froth and you have to stop or you'll get frothy brown by-product in your methanol condensate. But the bulk of the methanol should have been recovered by then.

Other biodiesel home-brewers try to recover methanol from the unwashed biodiesel. This is done by heating the unwashed biodiesel in a close container connected to a condenser. The disadvantage of this process is that you will be using much more energy to heat a much larger volume and yet recover only a marginal quantity of methanol. Others try to solve this problem by trying to recover the methanol just after the reaction is completed, while the solution is still hot. However, doing this process (while the glycerine containing the catalyst is still not separated) could push the reaction backwards, creating more problems. It will still work though, using more sophisticated processes, but for the home-brewers, it's just too risky.

Separating glycerine

Adding phosphoric acid (H_3PO_4) converts the soap back to FFAs and separates it all into three distinct layers, with catalyst-phosphorus on the bottom, glycerine-methanol in the middle, and FFAs on the top. But it won't happen without the methanol, so this should be done when the methanol is not yet separated.

The methanol can then be recovered from the glycerine in the middle layer by heating to above 65^{o}C in a closed container fitted with an outlet into a simple condenser as outlined in the procedure above.

Add the phosphoric acid and mix it thoroughly. The mixture gets a bit hot from the reaction. A translucent glycerine layer will form under a dark FFA layer, and not long afterwards the catalyst precipitates out at the bottom, sodium phosphates if you used NaOH as the catalyst, or potassium phosphates with KOH (in which case separation takes a bit longer).

The catalyst layer is yellowish, the glycerine layer is translucent while the FFA layer much darker. There's a clear separation between each. Then the three layers can be separated and the methanol can be removed from the glycerine.

The amount of phosphoric acid to use could be determined by trial and error. Some home brewers can make some calculations to determine the ideal amount of acid needed based on the initial amount ingredients used in the biodiesel production but the process could be too complicated for the average home brewers.

Start by making a trial batch, and use at least 85% pure phosphoric acid. Put 500 ml of glycerine in a pet bottle. Add 5 ml of phosphoric acid, close the pet bottle and shake vigorously. The first hint of glycerine separation should be visible within 5 minutes. If not, then add another 5 ml, shake vigorously then wait for another 5 minutes. Work your way up in 5 ml increments until it starts to separate.

Use the result in computing the total amount of acid needed in the batch of glycerine you want to separate. Stir the mixture vigorously and let it settle for at least 24 hours before separating them.

The crude glycerine layer in the middle contains methanol. The methanol can be recovered from the glycerine by heating to above 65^{o}C in a closed container fitted with an outlet into a simple condenser as outlined in the procedure above. The glycerine was acidified in the process and should be neutralised, with a dilute lye solution. This leaves you with industrial-grade glycerine of about 90% purity. It's a much more attractive product for glycerine refiners than prior to FFA recovery, and you might be able to sell it to a refiner, if not for much, or at least give it away rather than having to dispose of it.

The FFA on the top layer burns well, but not very well. It can be used as heating fuel. Like the by-product cocktail it can be composted. It also makes an effective weedkiller, and should biodegrade quickly once it hits the soil.

The lye catalyst reacts with the phosphoric acid to form sodium phosphates (with NaOH) or potassium phosphates (with KOH) and settles at the bottom. Either can safely be added to the compost pile. Industrial-scale biodiesel operations use potassium hydroxide, KOH, as the catalyst, and after separating the by-product with phosphoric acid the potassium phosphate left is sold as a fertilizer.

Soap

The crude glycerine by-product from homemade biodiesel makes a good degreaser. Remove the residual methanol first as outlined in “Reclaiming excess methanol” above. The raw by-product still contains most of the lye catalyst used in the processing, which makes it very caustic and it can burn the skin.

Saponifying the by-product makes it good cleaner, and it won't harm the skin. It can be used in all cleaning jobs, whether to clean old machine parts filthy with dirt and grease, or as an effective and economical dishwasher, laundry soap, or an excellent hand-cleaner.

Saponifying simply means mixing extra lye with water, add it to the heated by-product and mix for 10 or 15 minutes while maintaining the temperature. Then you have to cure it for a couple of weeks (up to 4 weeks). The difficulty with this process is in calculating how much extra lye to use. It depends how much lye is already in the mixture. The results also depend on which catalyst you used. Sodium hydroxide (NaOH) will give a solid bar soap and potassium hydroxide (KOH) makes liquid soap.

You have to stay with the catalyst you started with. If you used NaOH in the biodiesel process you can't then use KOH to make soap from the by-product, it has to be NaOH, and vice versa.

Use 100 ml of water per litre of by-product. You may also try using less water. The lye quantity is more critical. It depends on the titration of the oil you used. Common recommendations are that if you used say 5-7 grams of NaOH per litre of oil in the biodiesel process, then you need another 30 to 40 grams of NaOH to saponify the by-product. The figure is not fixed. It could be more or it could be less. Make trial batches and adjust the amount of lye accordingly. If you don't use enough lye the soap tends to leave an oily film, and if you use too much it's too harsh.

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HOMEMADE BIODIESEL STANDARDS

International standards

Specifications for biodiesel have been implemented in several countries around the world, most notably in the U.S., through the American Society of Testing and Materials (ASTM), and in Europe through the European Committee for Standardization (CEN). The relevant committees of these groups that oversee fuel specification development, including standards for biodiesel, are comprised of automobile and engine manufacturers, refining companies, biofuel producers, government entities and other fuel users who agree on specifications to help ensure good quality fuels for safe and satisfactory operation of vehicles and engines.

In the U.S., the ASTM specification for biodiesel is ASTM D 6751. The ASTM specification defines biodiesel as a fuel comprised of mono-alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats. Raw vegetable oils and animal fats that have not been processed do not meet biodiesel specifications.

In Europe, EN 14214 establishes specifications for fatty acid methyl esters for diesel engines. EN 14214, is more restrictive and applies only to mono-alkyl esters made with methanol, fatty acid methyl esters (FAME). The addition of components that are not fatty acid methyl esters other than additives is not allowed. Table 13.1 shows ASTM specifications for biodiesel.

Other countries developed their own standards but these are basically based on either the ASTM D 6751 or EN 14214. ASTM D6751 is the most common standard referenced in the United States and Canada. In Germany, the requirements for biodiesel are fixed in the DIN EN 14214 standard and in the UK the requirements for biodiesel is fixed in the BS EN 14214 standard, although these last two standards are essentially the same as EN 14214 and are just prefixed with the respective national standards institution codes. Table 13.2 shows EN specifications for biodiesel.

The standards ensure that the following important factors in the fuel production process are satisfied:

- Complete reaction.
- Removal of glycerine.
- Removal of catalyst.
- Removal of alcohol.
- Absence of free fatty acids.
- Low sulfur content.

Table 13.1: ASTM D 6751 Requirements for Biodiesel (B100)

Source: Changes in Diesel Fuel, Service Technician's Guide to Compression Ignition Fuel Quality, Reynolds, et., al. 2007

Property	Units	Limits	Importance
Calcium and magnesium, combined	ppm (µg/g)	5 max	To protect against wear of injectors, pumps, pistons & ring and also engine deposits & premature failure of particulate filters
Flash point	ºC	93 min	Safety during fuel handling & storage
Alcohol control - One of following must be met:			To ensure alcohol from manufacturing process is properly removed
1. Methanol content	% mass	0.2 max	
2. Flash point	ºC	130 min	
Water and sediment	% volume	0.05 max	Filter plugging, injector wear, increased corrosion
Kinematic viscosity, 40ºC	mm2/s	1.9-6.0	Injector wear & spray pattern, pump wear, filter damage
Sulfated ash	% mass	0.020 max	Limits unremoved catalyst from fuel to protect against wear in injector pumps, pistons, rings & reduce engine deposit
Sulfur	% mass (ppm)	0.0015 max (15)	To protect emissions control equipment
Copper strip corrosion		No. 3 max	Protect copper, brass, bronze fuel system parts
Cetane number		47 min	Measure of ignitability (ignition quality)
Cloud point	ºC	Report	Low-temperature operability
Carbon residue, 100% sample	% mass	0.050 max	To reduce deposits in fuel system and engine
Acid number	mg KOH/g	0.50 max	Protect against fuel system deposits & corrosion
Free glycerine	% mass	0.02	Injector deposits & fuel system clogging
Total glycerine	% mass	0.24	Injector deposits, filter plugging & low-temperature operability
Phosphorus content	% mass	0.001 max	Protect catalysts in exhaust after-treatment devices
Distillation temperature, 90% recovered	ºC	360 max	Affects fuel economy and power under varying loads/speeds
Sodium and potassium, combined	ppm (µg/g)	5 max	Limits unremoved catalyst from fuel to protect against wear in injector pumps, pistons, rings & reduce engine deposit
Oxidation stability	hours	3 min	Storage stability, prevent degradation of fuel

Table 13.2: EN 14214 Requirements for Biodiesel (B100)

Source: Changes in Diesel Fuel, Service Technician's Guide to Compression Ignition Fuel Quality, Reynolds, et. al., 2007

Property	Units	Limits	Importance
Alkali Metals (Na+K)	mg/kg	5 max	
Flash point	ºC	101	Safety during fuel handling & storage
Methanol content	% mass	0.2 max	To ensure alcohol from manufacturing process is properly removed
Water content	mg/kg	500 max	Filter plugging, injector wear, increased corrosion
Kinematic viscosity, 40ºC	mm2/s	3.5 - 5	Injector wear & spray pattern, pump wear, filter damage
Sulfated ash	% mass	0.02 max	Limits unremoved catalyst from fuel to protect against wear in injector pumps, pistons, rings & reduce engine deposit
Sulfur content	mg/kg	10 max	To protect emissions control equipment
Copper strip corrosion, 3 hours at 50 °C		Class 1	Protect copper, brass, bronze fuel system parts
Cetane number		51	Measure of ignitability (ignition quality)
Acid number	mg KOH/g	0.50 max	Protect against fuel system deposits & corrosion
Free glycerine	% mass	0.02 max	Injector deposits & fuel system clogging
Total glycerine	% mass	0.25 max	Injector deposits, filter plugging & low-temperature operability
Phosphorus content	mg/kg	10 max	Protect catalysts in exhaust after-treatment devices
Oxidation stability, 110°C	hours	6 min	Storage stability, prevent degradation of fuel
Ester content	% (m/m)	96.5	
Density at 15°C	kg/m³	860 - 900	
Tar remnant (at 10% distillation remnant)	% (m/m)	0.30 max	
Total contamination	mg/kg	24 max	
Iodine value	-	120 max	
Linolenic Acid Methylester	% (m/m)	12 max	
Polyunsaturated (>= 4 Double bonds) Methylester	% (m/m)	1 max	
Monoglyceride content	% (m/m)	0.80 max	
Diglyceride content	% (m/m)	0.20 max	
Triglyceride content	% (m/m)	0.20 max	

Standards and the homebrewer

As noted in the first part of this chapter, standards and specifications were developed to help ensure good quality fuels for safe and satisfactory operation of vehicles and engines. But from a legal perspective, there is no need for fuel standards for homemade biodiesel if it is made for personal consumption. The issue of standards for homemade biodiesel should not even arise. Humans are not only creative but also protective of their own assets, especially their own vehicles. Homebrewers constantly push the envelope to see how much fuel they can create with little resources, something the government should encourage. Considering that biodiesel reduces and sometimes even eliminated most of the emission problems created by petroleum diesel usage, the only restriction that should be necessary in regards to homebrewed biodiesel is limiting the use of the fuel for the personal use of the homebrewer.

However, in pushing towards producing good quality fuel, homebrewers should aim at making their products comply with existing quality checks. Being an alternative fuel for petroleum diesel, biodiesel should meet the petroleum diesel specification. It is however unknown why biodiesel standards are so different from petroleum diesel standards. It is understandable that every country will have slightly different biodiesel characteristics because of differing feedstock available, but at least it should be close enough to petroleum diesel. Theoretically, petroleum diesel will not even qualify to any of the biodiesel standards. For example nobody knows exactly why ASTM D 6751 and EN 14214 set the cetane requirement of biodiesel to 47 and 51 respectively when the petroleum diesel cetane requirement (ASTM) is only 40. Both standards are also silent about the requirements regarding emissions, environmental and health effects which biodiesel is known to have tamed of.

On the other hand most of the standards can be met simply by preparing and washing the fuel well. Any homebrewer should not be concerned about any of these standards. As long as you have good separation, done proper washing and drying, your biodiesel should be of good quality. And even if your biodiesel didn't separate at all which means that you can not wash it, putting the whole thing in your fuel tank does not constitute any legal offense, so long as you do not pollute the planet.

CONTENTS

METHANOL

The information contained in this chapter was based mainly from the information provided in the Methanol Institute website (www.methanol.org).

Methanol, CH_3OH (Methyl Alcohol) is the second most important ingredient in making biodiesel. As repeated many times in this book, this material is also the most dangerous if handled carelessly and senselessly. This chapter was added mainly to provide as much information as possible about this material and how it is handled properly. It's entry into your body system could be fatal, and you don't even have to drink it because it can enter through your skin. It is also flammable (including its vapour), with almost invisible flame.

Safe handling and storage guidelines:

- Avoid prolonged or repeated breathing of methanol vapours. You should not breathe in the vapour. Ensure that the area in which you work is very well ventilated.
- Methanol should always be kept within closed systems or approved containers and stored in a secure space.
- Wear proper attire when handling methanol, including appropriate footwear, face shields or safety spectacles, gloves and respiratory protection.
- Do not smoke or permit smoking anywhere near the area where methanol is stored. Do not use torches or any ignition source near the area where methanol is stored, as even small amounts of lingering vapours in the area or in an apparently empty storage container can ignite and result in a dangerous blast and/or fire. Remove any source of ignition from the working area. Don't forget that a hot air gun, a hot plate or even a radiator may be sufficiently hot to ignite the vapour.
- If a methanol spill occurs, stop or reduce discharge of material if this can be done without risk. Isolate the spill or leak area immediately for at least 100 to 200 metres in all directions. Eliminate all sources of ignition, and stay upwind. Do not touch or walk through the spilled material.
- Prevent methanol from entering into waterways, sewers, basements or confined areas. Do not pour methanol down the drain. Methanol is a hazardous material and must be disposed of properly. Check with local environmental officials for instructions on how to safely dispose of methanol in your community.
- A vapour suppressing foam may be used to reduce vapours. For small spills (up to 55-gallon drum) absorb with earth, sand or other non-combustible material and transfer to containers for later disposal. For large spills, dike far ahead of liquid spill for later disposal, and follow local emergency protocol for handling.
- Spills into large natural bodies of water, such as rivers and oceans, cannot be contained. For releases into soil, surface water or groundwater, methanol has a half-life of just one to seven days, and given its high rate of biodegradation, methanol spills are not likely to persist. Again, methanol must be disposed of properly, do not attempt to dump methanol on the ground or in any body of water.

Methanol is used extensively in the wastewater treatment facilities to reverse the damaging effects of nitrate buildup in sensitive aquifers and waterways by accelerating biodegradation. As a flammable and toxic chemical, caution must be exercised to avoid contact with methanol.

Principal hazards

Methanol is toxic. Target organs include kidneys, central nervous system, eyes and skin. Aside from inhaling or swallowing it, it can also be absorbed through the skin.

Eye:
Produces irritation, characterised by a burning sensation, redness, tearing, inflammation, and possible corneal injury. Vapours may cause eye irritation and painful sensitization to light.

Skin:
May cause skin irritation.

Ingestion:
Ingestion of methanol may be fatal or cause blindness. It may cause irritation of the digestive tract, kidney damage. It may cause central nervous system depression, characterised by excitement, followed by headache, dizziness, drowsiness, and nausea. Advanced stages may cause collapse, unconsciousness, coma and possible death due to respiratory failure.

Inhalation:
Inhalation of methanol may cause respiratory tract irritation. It may cause visual impairment and possible permanent blindness. It may also cause effects similar to those described for ingestion.

Chronic:
Prolonged or repeated skin contact may cause dermatitis. Chronic inhalation and ingestion may cause effects similar to those of acute inhalation and ingestion.

Methanol is also very flammable and can cause fires and explosions. The pure liquid catches fire easily and aqueous solutions containing a significant amount of methanol can also catch fire. The flame above burning methanol is virtually invisible, so it is not always easy to tell whether a methanol flame is still alight.

First Aid Recommendations:

Eyes:
If methanol comes in direct contact with eyes, immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower lids. Get medical aid immediately. The patient should be taken to a health care facility and referral to an ophthalmologist considered.

Skin:
In case of methanol contact with skin, remove contaminated clothing immediately, wash with soap and water for 15 minutes, and seek medical attention if irritation occurs.

Ingestion:
Ingestion of methanol is life threatening. Onset of symptoms may be delayed for 18 to 24 hours after ingestion. If victim is conscious and alert, give 2-4 cupfuls of milk or water. Never give anything by mouth to an unconscious person. Due to the risks of aspiration into the lungs, do not induce vomiting. The decision to induce vomiting should be left to a medical professional attending the victim. Transport immediately to a health care facility where standard methanol ingestion treatment can be administered. Immediate medical attention is critical.

Inhalation:
In case of inhalation of methanol vapours, move individual from vapour exposure to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. It may require artificial respiration. Get immediate medical aid.

Physical properties

Form:	colourless liquid with a characteristic smell

Odor:	slight alcohol-like
Stability:	Stable, but very flammable
Melting point:	-98°C
Boiling point:	64.7°C
Flash point:	11°C
Explosion limits;	6% - 36%
Water solubility:	miscible in all proportions
Specific gravity:	0.79
Vapour density (Air = 1):	1.1
Autoignition Temperature:	455°C

Disposal

Trace amounts of methanol can be flushed down a sink with a large quantity of water, unless local rules prohibit this. Larger amounts should be collected in a non-chlorinated waste solvent container for disposal.

Fire Fighting Measures

General Information: Methanol is a flammable liquid. Vapours of methanol may form an explosive mixture with air. It can travel to a source of ignition and flash back. Vapours may be heavier than air and can spread along the ground and collect in low or confined areas. It may be ignited by heat, sparks, and flame. Containers may explode when heated.

Extinguishing Media: Putting off fires can be done by using dry chemical, carbon dioxide, water spray or alcohol-resistant foam. Use water spray to cool fire-exposed containers, but water in itself may be ineffective in putting off the fire.

Personal Protective Equipment

Eyes:
Wear appropriate protective eyeglasses or chemical safety goggles as described by OSHA's eye and face protection regulations in 29 CFR 1910.133 or European Standard EN166.

Skin:
Wear appropriate protective gloves to prevent skin exposure.

Clothing:
Wear appropriate protective clothing to prevent skin exposure.

Respirators:
Follow the OSHA respirator regulations found in 29CFR 1910.134 or European Standard EN 149. Always use a NIOSH or European Standard EN 149 approved respirator when necessary.

Stability and Reactivity

Chemical Stability:
Stable under normal temperatures and pressures.

Conditions to Avoid:
High temperatures, incompatible materials, ignition sources.

Incompatibilities with Other Materials:

Acids (mineral, non-oxidizing, e.g. hydrochloric acid, hydrofluoric acid, muriatic acid, phosphoric acid), acids (mineral, oxidizing, e.g. chromic acid, hypochlorous acid, nitric acid, sulfuric acid), acids (organic, e.g. acetic acid, benzoic acid, formic acid, methanoic acid, oxalic acid), azo, diazo, and hydrazines (e.g. dimethyl hydrazine, hydrazine, methyl hydrazine), isocyanates (e.g. methyl isocyanate), metals (alkali and alkaline, e.g. cesium, potassium, sodium), nitrides (e.g. potassium nitride, sodium nitride), peroxides and hydroperoxides (organic, e.g. acetyl peroxide, benzoyl peroxide, butyl peroxide, methyl ethyl ketone peroxide), epoxides (e.g. butyl glycidyl ether), oxidizing agents (strong, e.g. bromine, hydrogen peroxide, nitrogen dioxide, potassium nitrate), reducing agents (strong, e.g. aluminum carbide, chlorosilane, hydrogen phosphide, lithium hydride), water reactive substances (e.g. acetic anyhdride, alkyl aluminum chloride, calcium carbide, ethyl dichlorosilane).

Hazardous Decomposition Products:
Carbon monoxide, carbon dioxide, formaldehyde.

CONTENTS

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www.biodiesel.org

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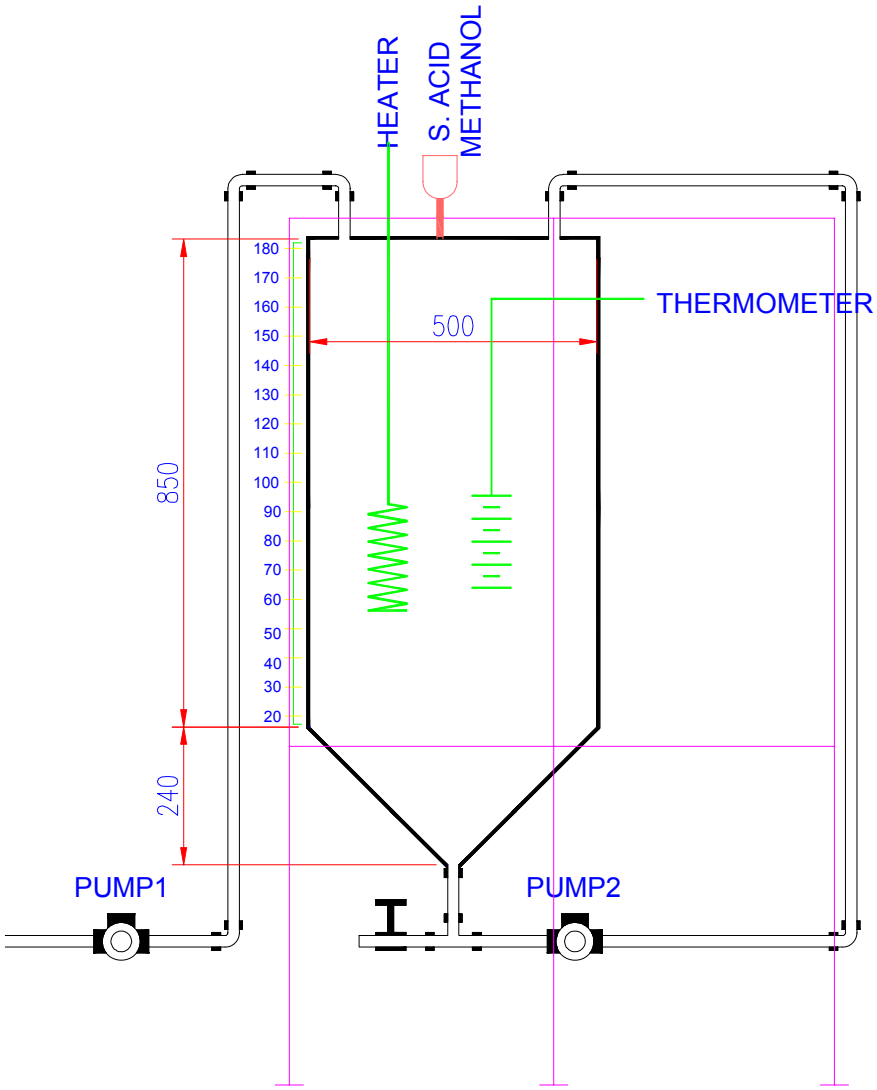
RELATED SITES:

Sydney Biodiesel Users Group: http://www.sydneybiodiesel.com

CONTENTS

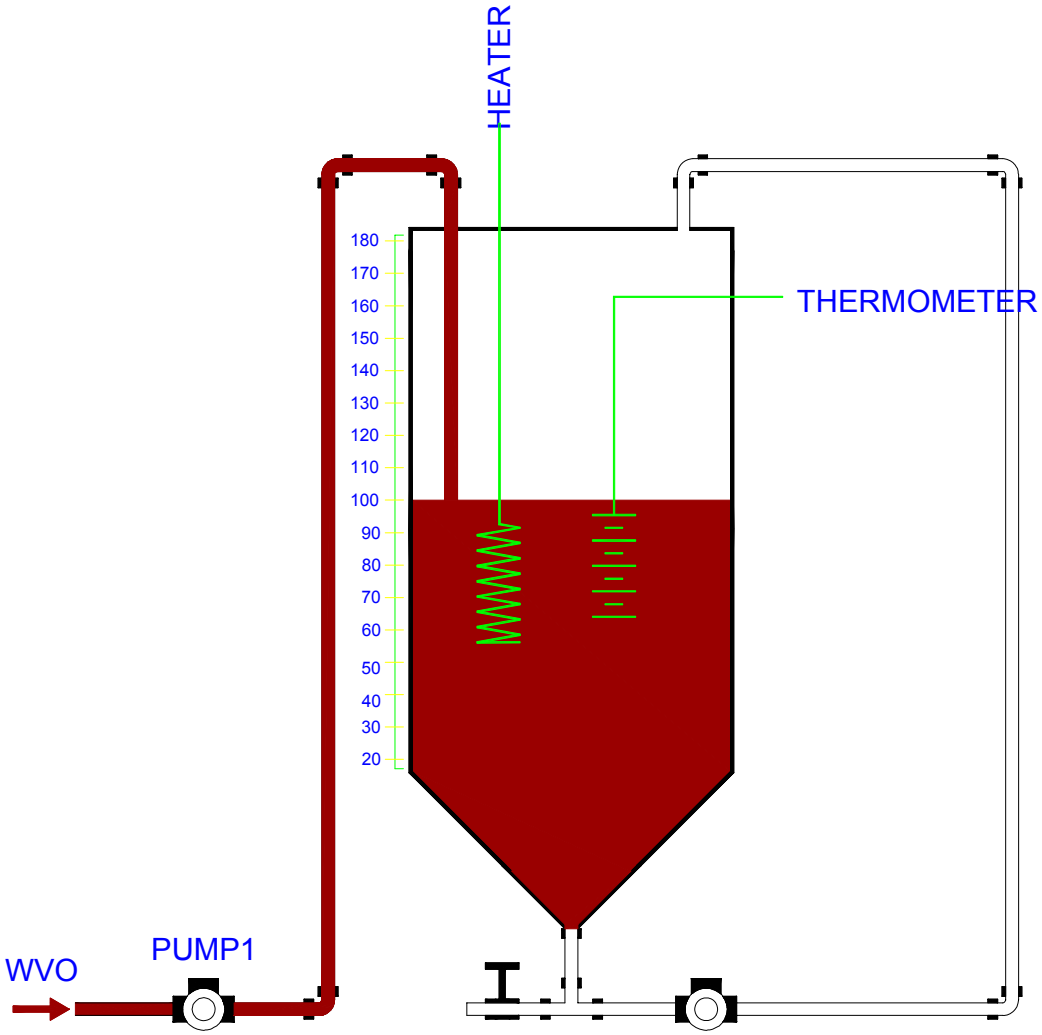
APPENDIX A
PROCESSOR AND PROCESSING

Presented in the succeeding sketches is a processor design with the suggested processing scheme.



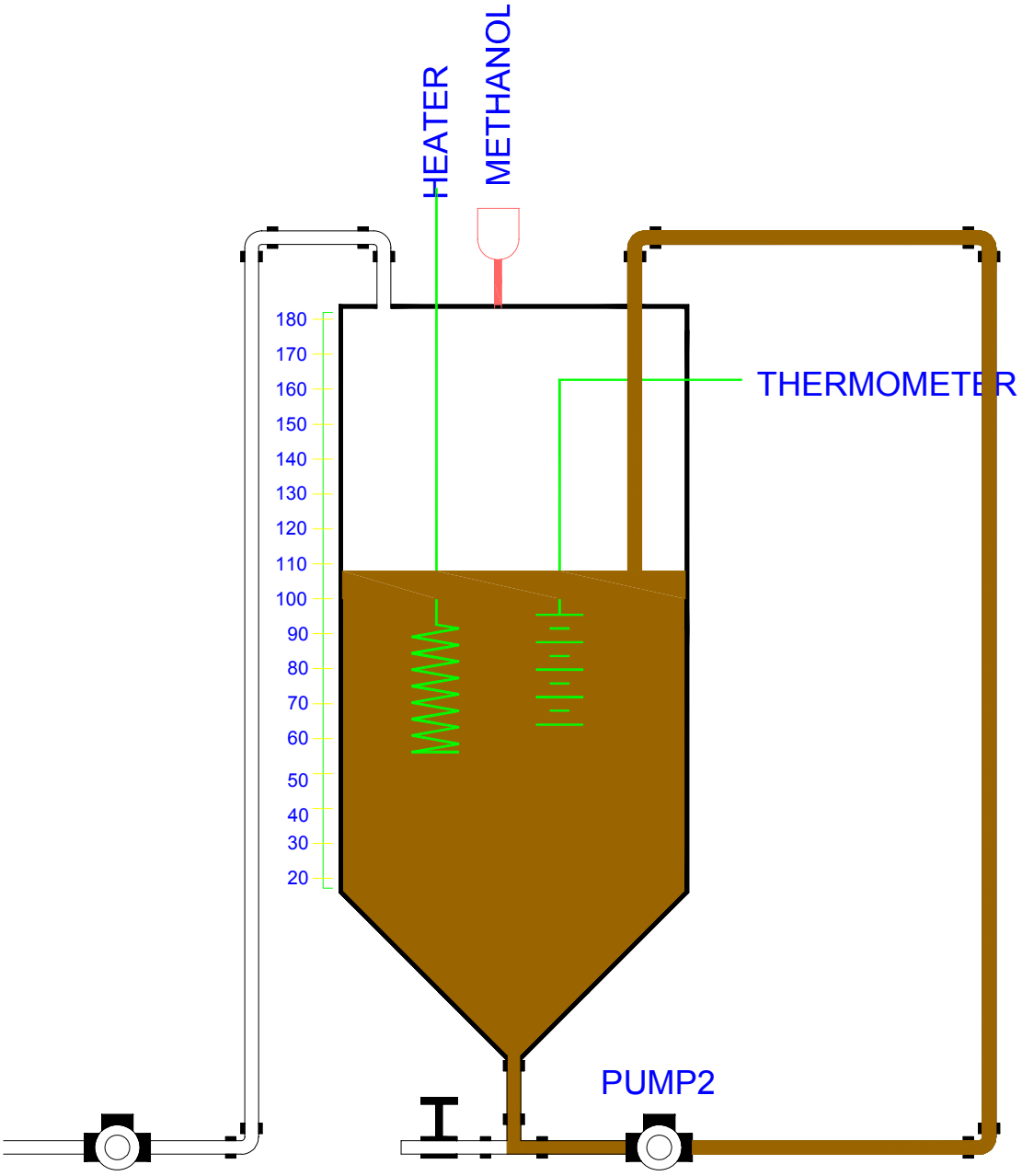
1. ALL FITTINGS AND PIPES, 10MM Ø. IT IS RECOMMENDED THAT YOU USE TRANSLUCENT BRAIDED TUBING SO YOU CAN SEE AND MONITOR THE COLOUR OF THE MIXTURE.
2. FRAME CAN BE MADE OF TIMBER OR STEEL.
3. PUMP 1 CAN BE MANUAL.
4. PUMP 2 AVERAGE CAPACITY: 10 LITRES PER MINUTE.
5. THE MAIN REACTOR CAN BE FITTED WITH A GRADUATED TRANSLUCENT TUBE (AS IN SKETCH) TO FACILITATE MEASURING OF WVO AND METHANOL.

Figure 1: Suggested Processor Design: 100 Litres



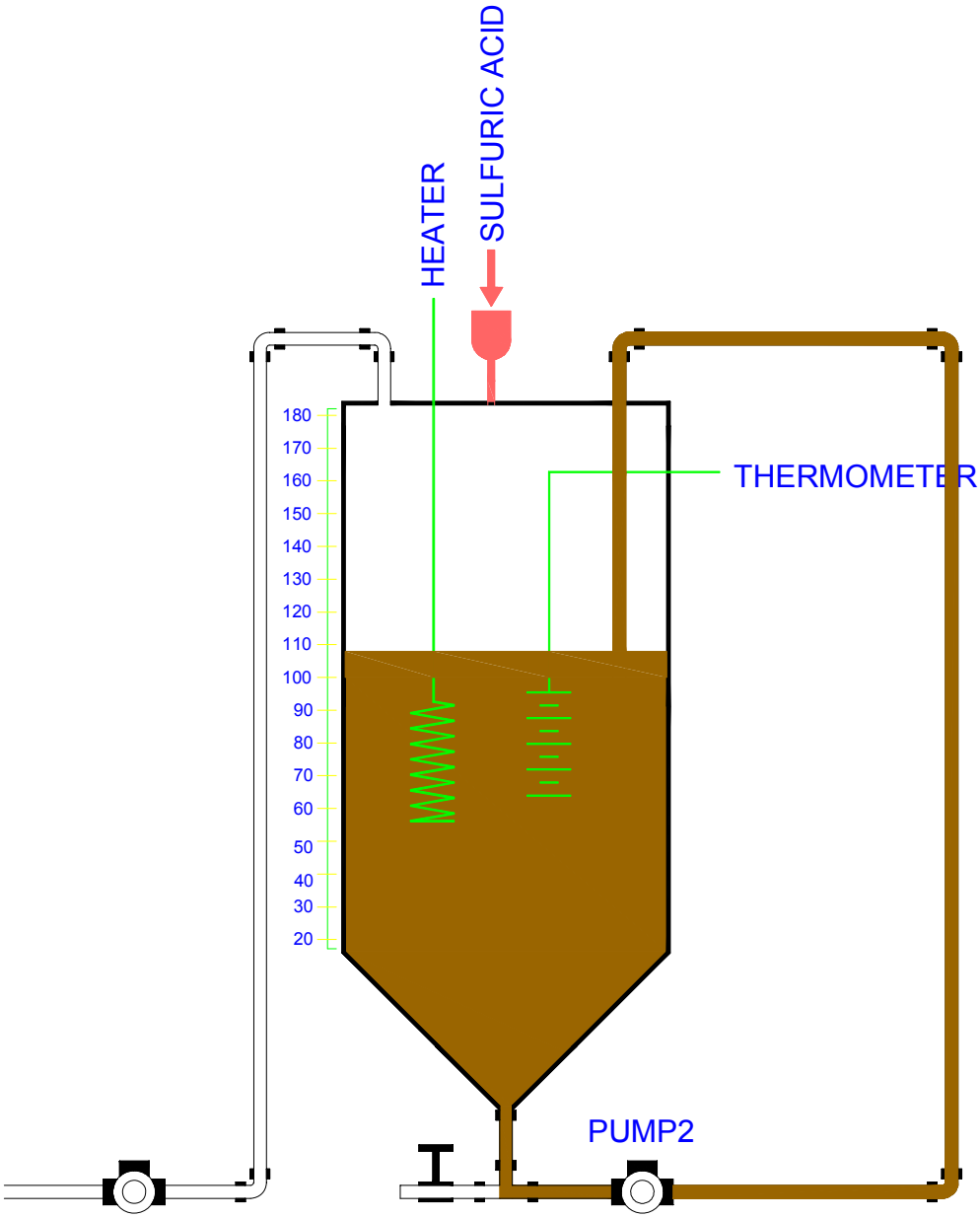
1. PUMP IN 100 LITRES OF DEWATERED WVO TO THE REACTOR, THEN HEAT IT TO 35°C.

Figure 2: Step 1 of Biodiesel Production



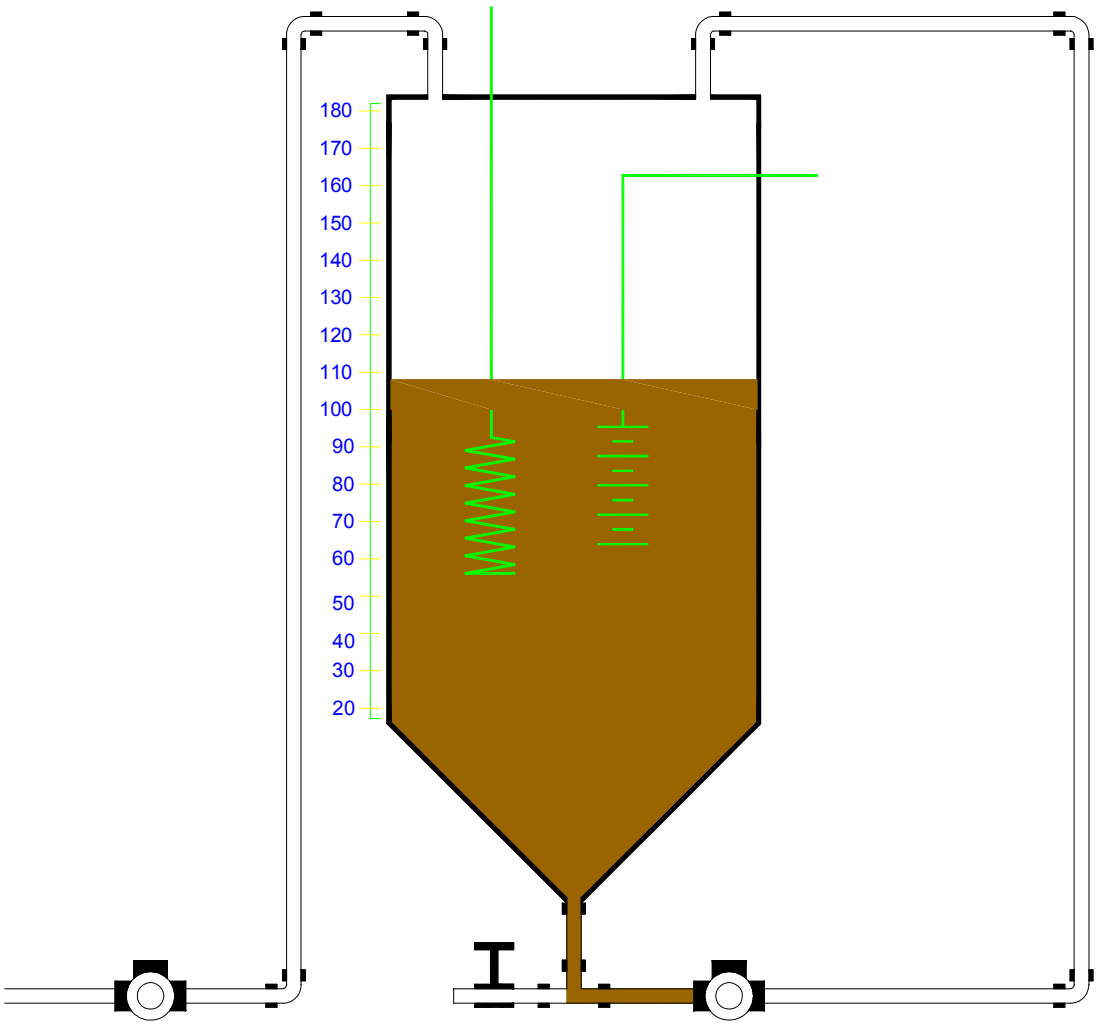
2. MEASURE 8 LITRES OF METHANOL AND ADD IT TO THE WVO IN THE REACTOR, THEN TURN ON PUMP 2. MIX FOR 10 MINUTES. NOTE, MIXING IS DONE BY TURNING ON PUMP 2.

Figure 3: Step 2 of Biodiesel Production



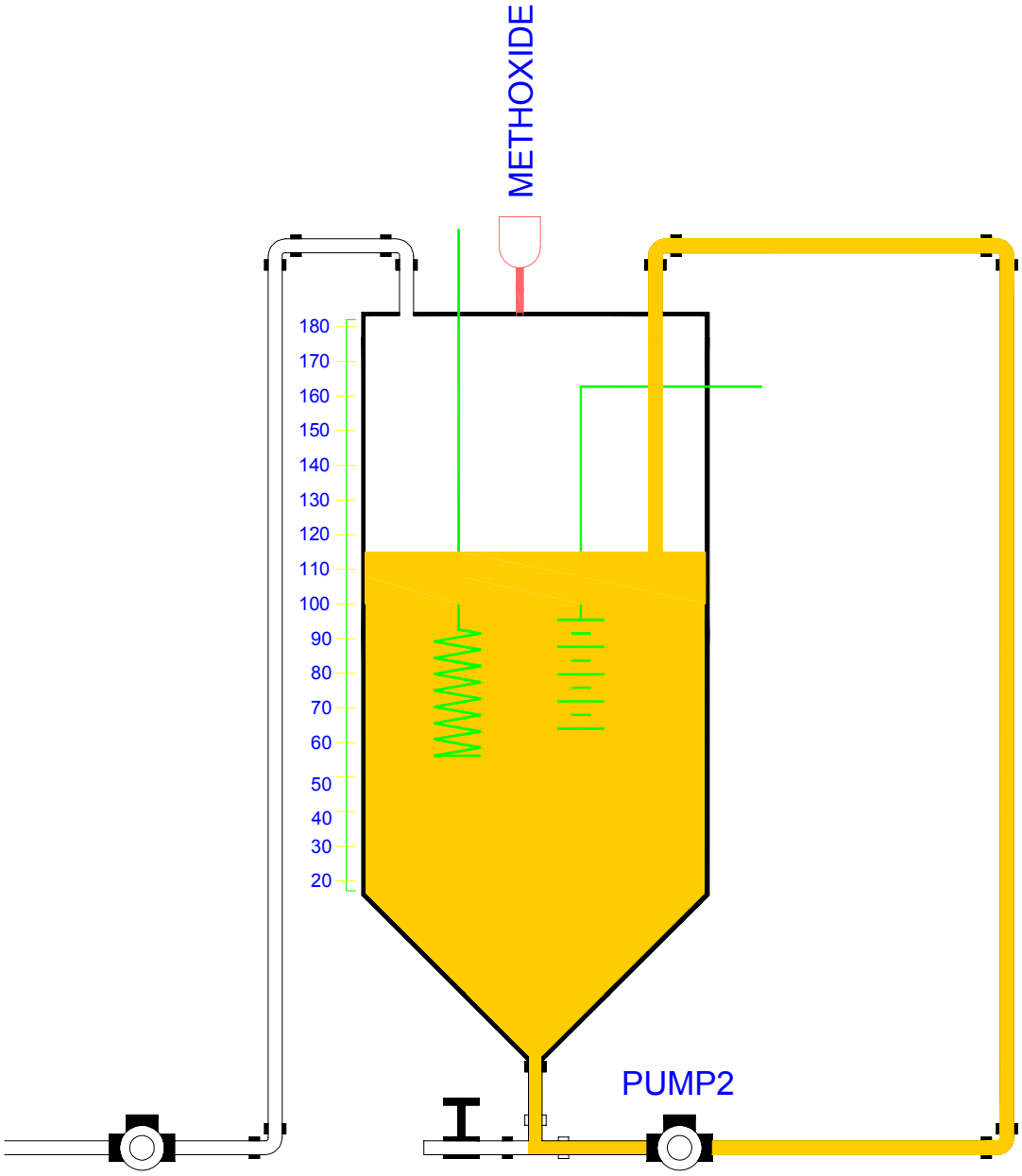
3. ADD 100ML 95% SULFURIC ACID. CONTINUE MIXING WHILE MAINTANING THE TEMPERATURE AT 35°C. TURN OFF THE HEATER AFTER 1 HOUR, THEN CONTINUE MIXING FOR ANOTHER 1 HOUR (TOTAL 2 HOURS).

Figure 4: Step 3 of Biodiesel Production



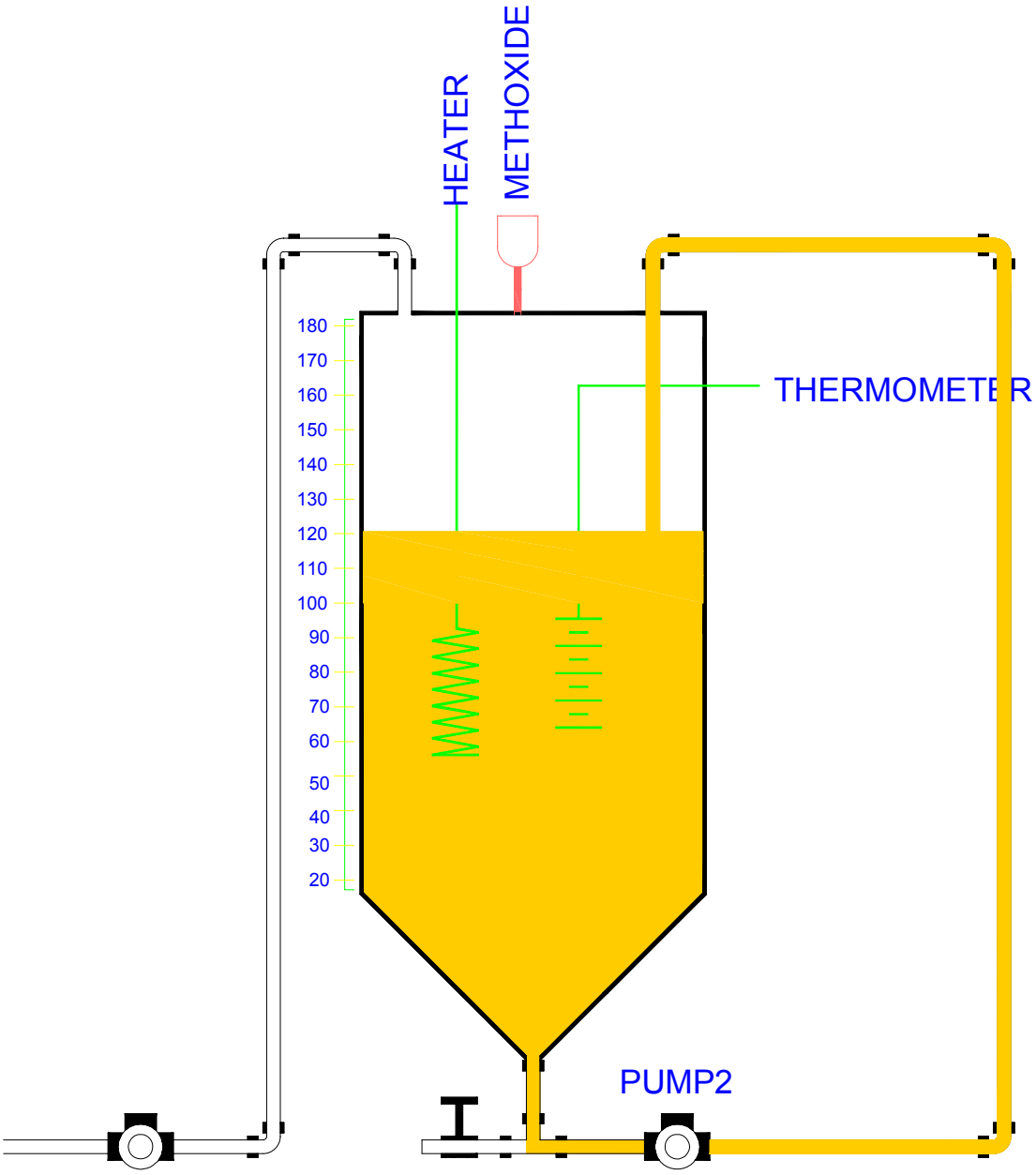
4. STOP MIXING THEN LET THE MIXTURE REST FOR AT LEAST 8 HOURS. MEANWHILE, PREPARE THE METHOXIDE IN A SEPARATE CONTAINER. DISSOLVE 350 GRAMS OF SODIUM HYDROXIDE IN 12 LITRES OF METHANOL.

Figure 5 : Step 4 of Biodiesel Prduction



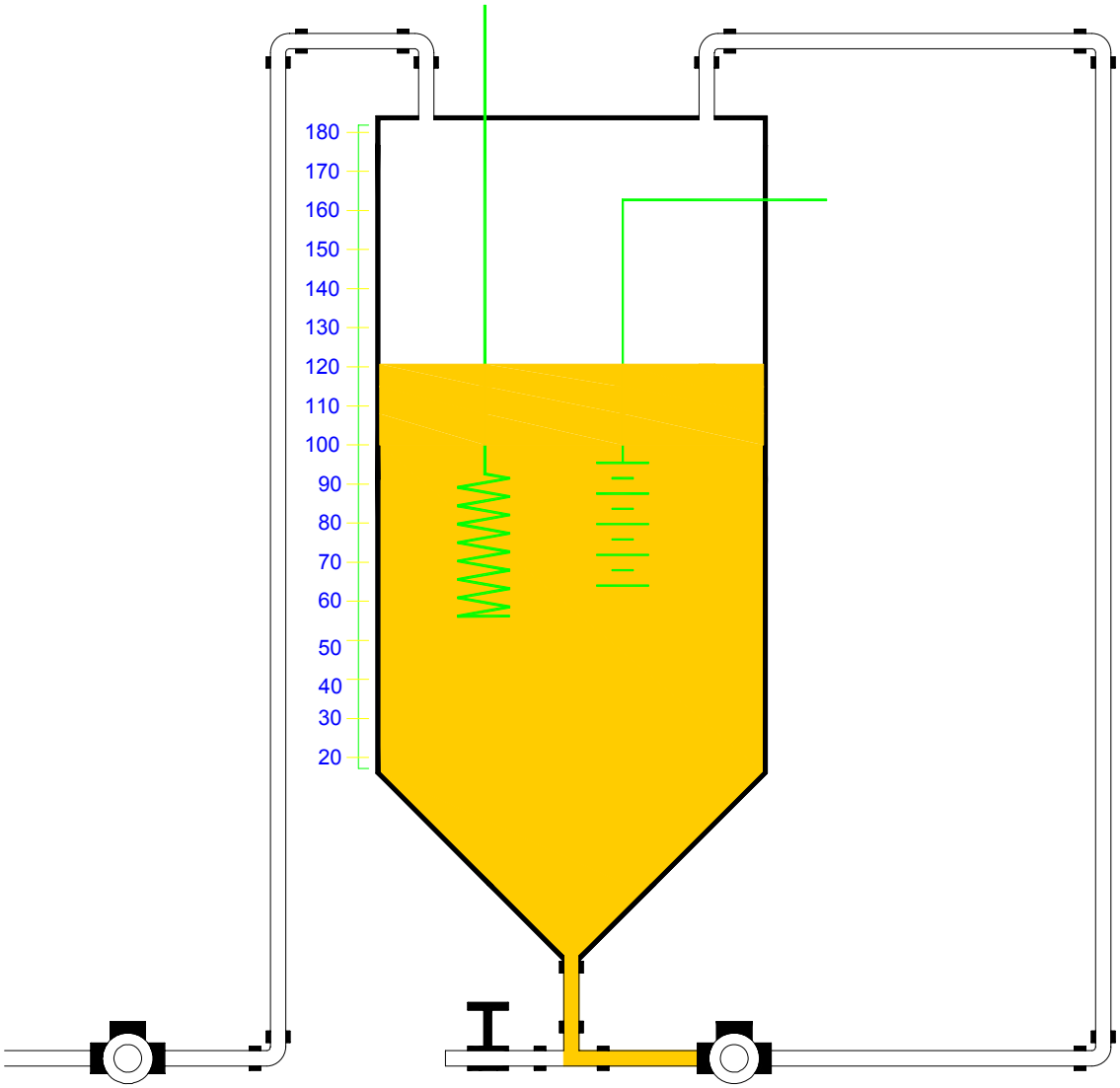
5. ADD HALF (6 LITRES) OF THE PREPARED METHOXIDE INTO THE REACTOR, THEN MIX FOR 10 MINUTES.

Figure 6: Step 5 of Biodiesel Production



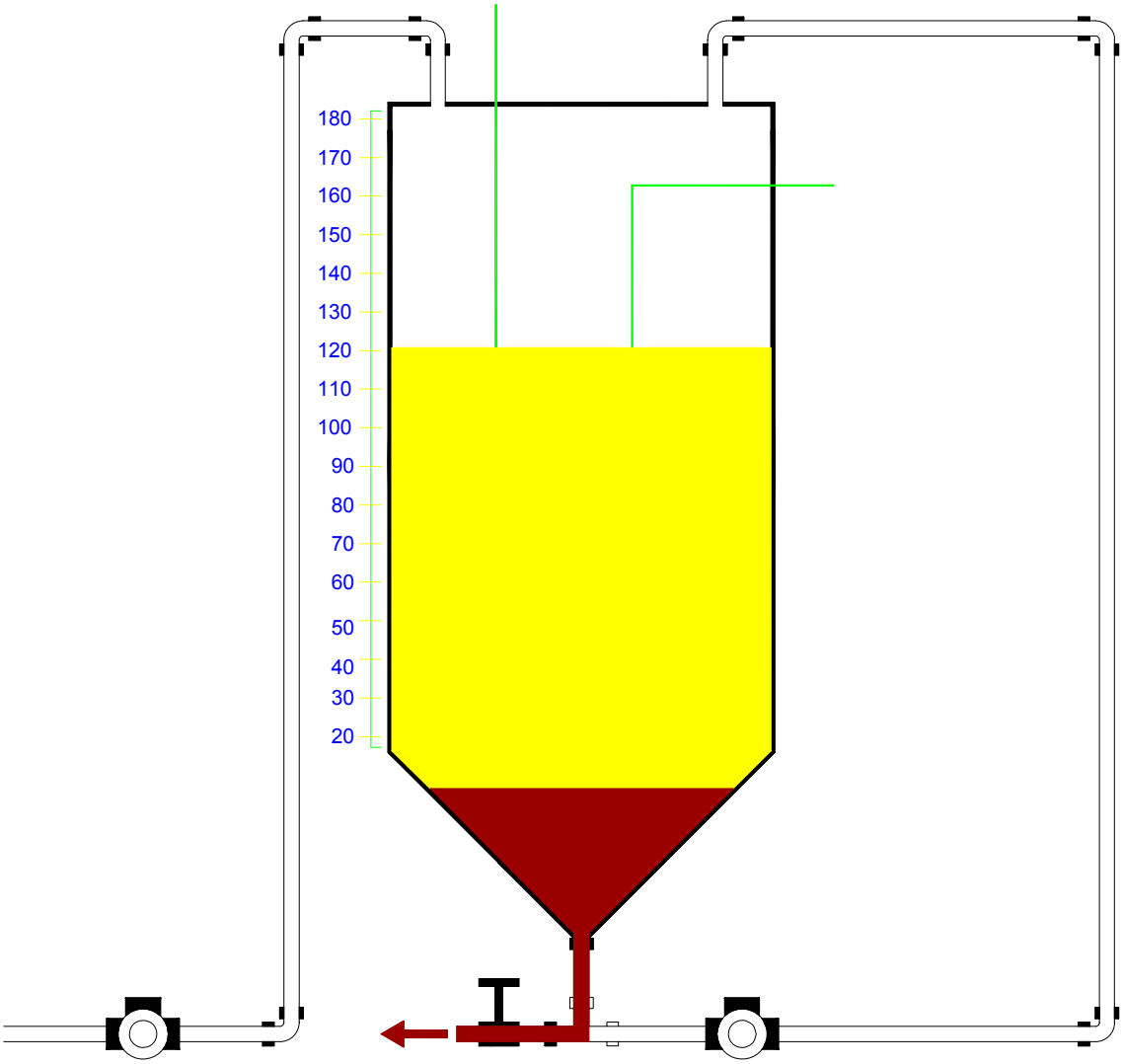
6. HEAT THE MIXTURE TO 55°C. MAINTAIN THIS TEMPERATURE FOR THE WHOLE REACTION. ADD THE OTHER HALF OF THE METHOXIDE. CONTINUE MIXING UNTIL THE COLOUR TURNS STRAW YELLOW (ABOUT 1.5 TO 2.5 HOURS).

Figure 7: Step 6 of Biodiesel Production



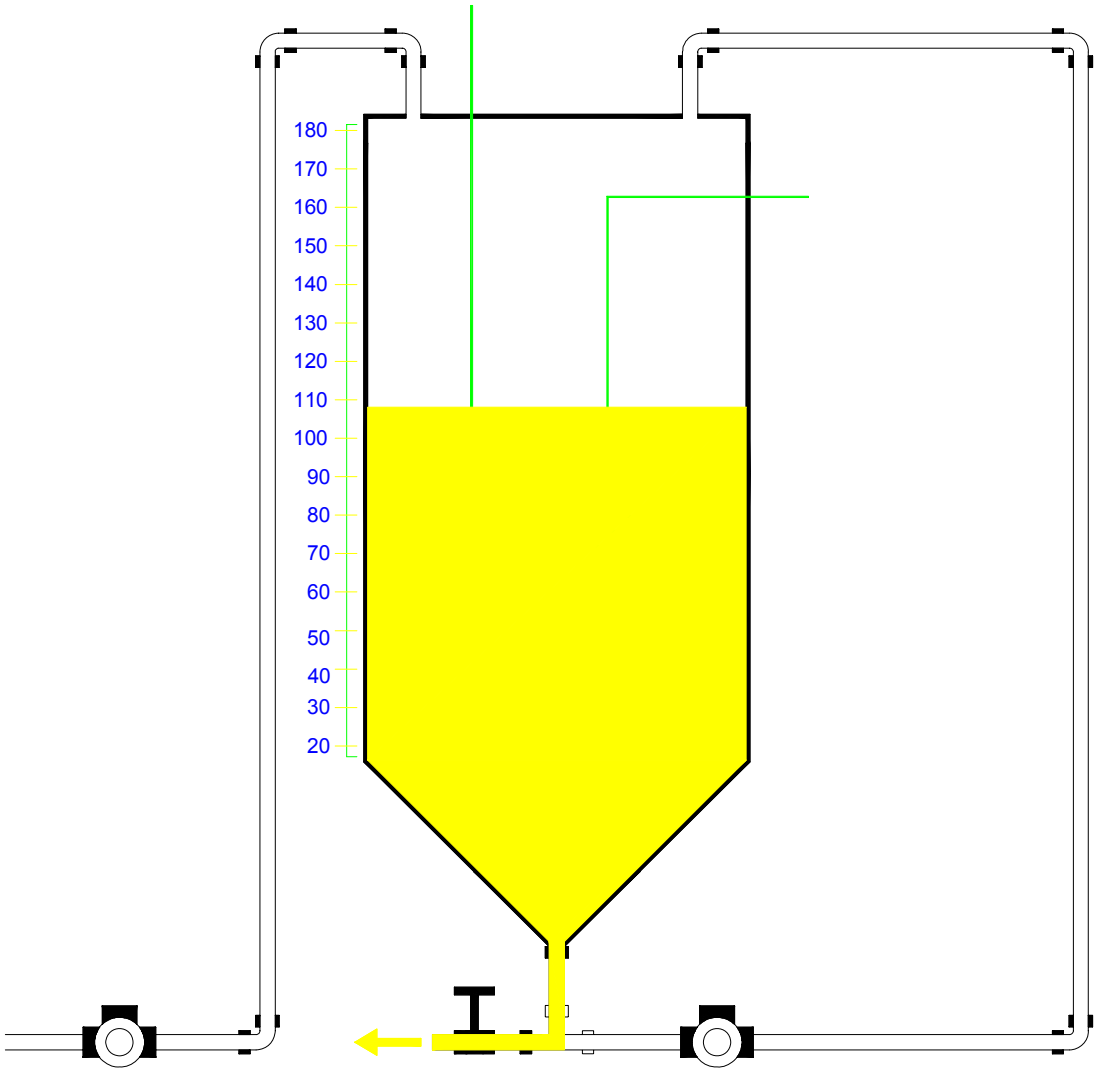
7. TURN OFF HEAT AND PUMP, THEN ALLOW TO SETTLE FOR AT LEAST 12 HOURS.

Figure 8: Step 7 of Biodiesel Production



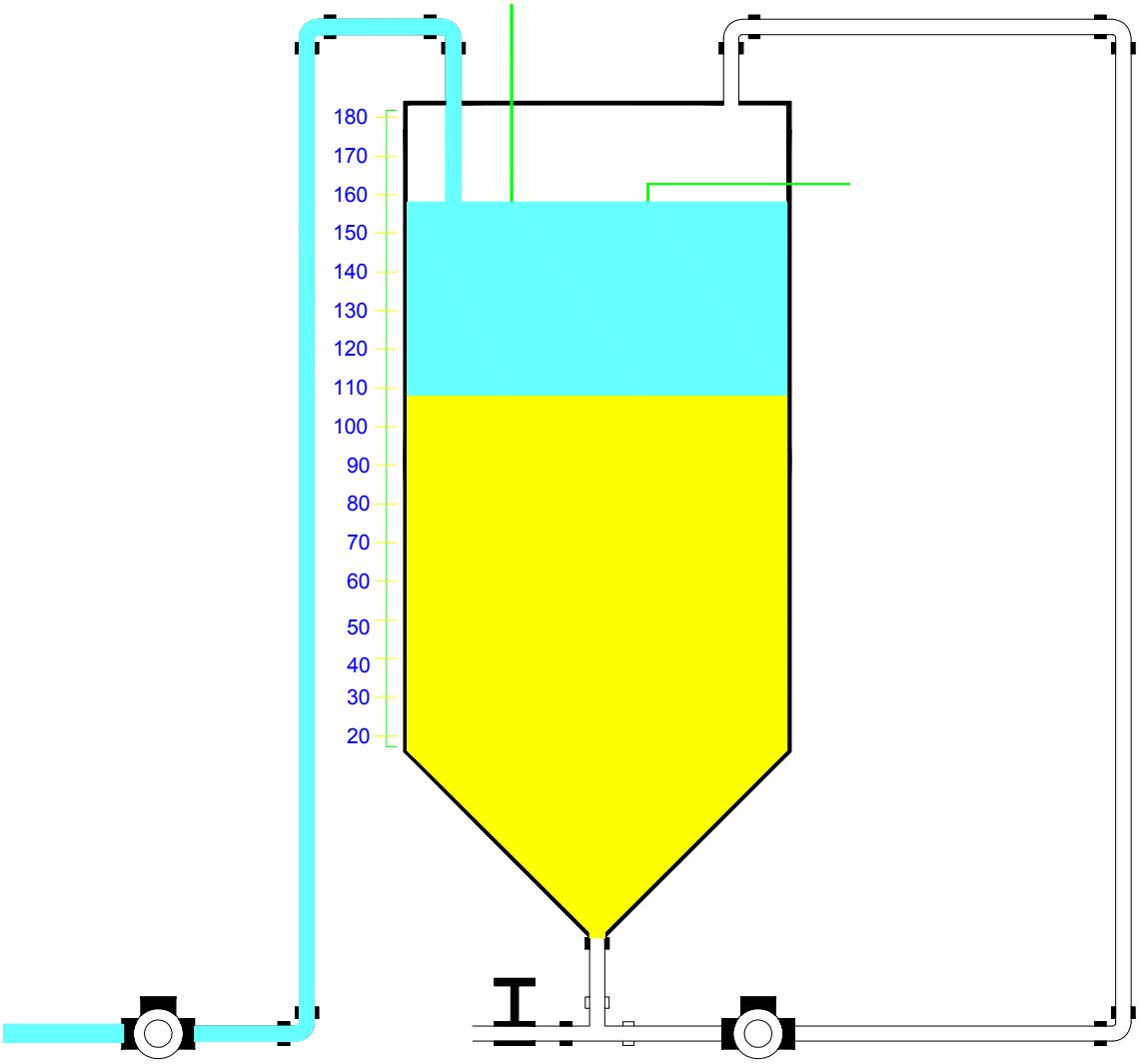
8. DRAIN OFF GLYCERINE AND PUT IN A SEALED CONTAINER. EXPECT AROUND 12 LITRES OF GLYCERINE.

Figure 9: Step 8 of Biodiesel Production



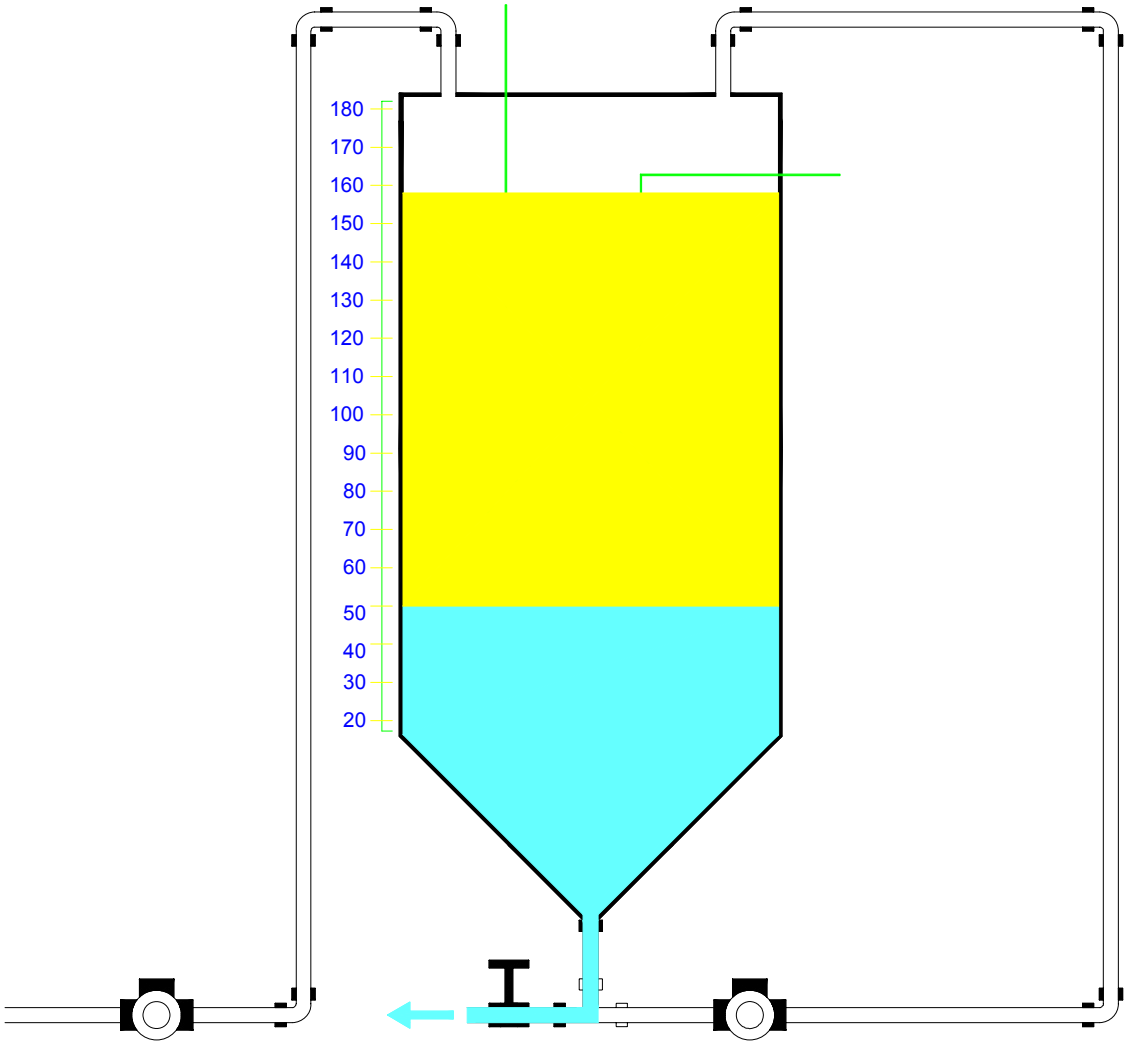
9. QUALITY CHECK: DRAIN OFF 100 ML OF BIODIESEL AND PUT IN A 2 LITRE PET BOTTLE. ADD 150 ML OF WATER, SCREW THE LID TIGHT, THEN SHAKE VIGOROUSLY FOR 10 SECONDS. THE MIXTURE SHOULD SEPARATE IN 30 MINUTES TO 1 HOUR. IF NOT, READ PAGE 70 FOR TROUBLESHOOTING.

Figure 10: Step 9 of Biodiesel Production



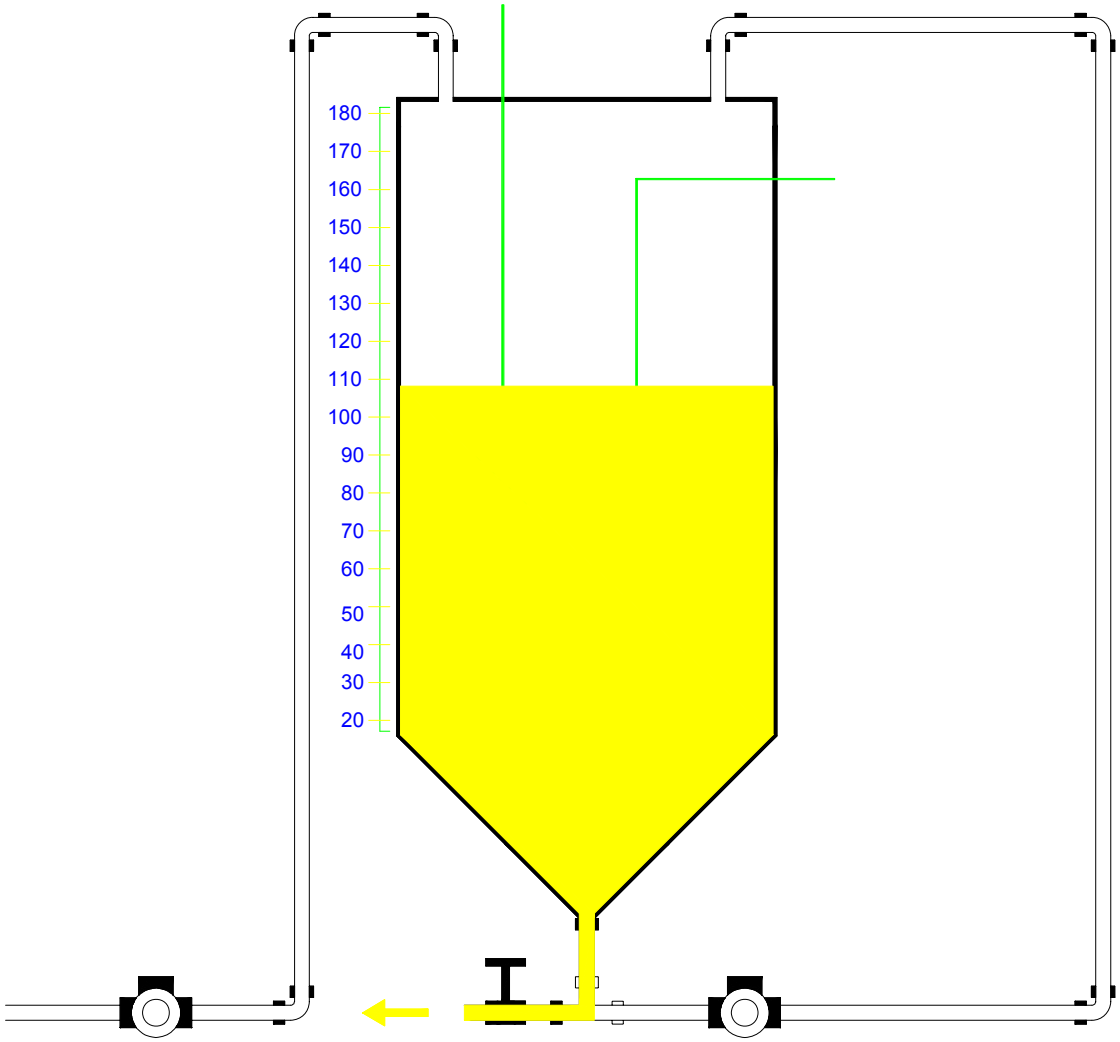
10. WASHING: ADD 30 ML OF ACETIC ACID TO THE BIODIESEL, THEN ADD 50 LITRES OF WATER. TURN ON PUMP 2 THEN MIX FOR 30 MINUTES.

Figure 11: Step 10 of Biodiesel Production



11. WASHING: LET IT SETTLE FOR 2 HOURS, THEN REMOVE THE WATER. CHECK THE PH OF WATER. REPEAT STEPS 10 TO 11 (BUT WITHOUT ADDING ACETIC ACID), UNTIL THE WATER PH REACHES 7 OR VERY CLOSE TO IT.

Figure 12: Step 11 of Biodiesel Production



12. DRYING: TRANSFER THE BIODIESEL TO YOUR STORAGE TANK, THEN LET IT STAND FOR 3 TO 4 WEEKS BEFORE USING. IF YOU CAN NOT WAIT, YOU CAN HEAT IT TO 45°C, THEN LET IT COOL. YOU CAN THEN USE IT TO YOUR CAR WITH CONFIDENCE.

Figure 13: Step 12 of Biodiesel Production

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3. **ELECTRICITY – MAKE IT, DON'T BUY IT**: This fantastic eBook will show you how to set up your own electric company running on biodiesel fuel in your back yard.

4. **HOW TO START AND OPERATE A DAYCARE CENTRE**: A step by step guide on how to start and operate a successful daycare centre. Everything You Ever Wanted to Know About Starting a Daycare.

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21. **THE STUDENT'S WINNING EDGE:** Remembering information is a skill many students struggle with. It is not because they were born with or without the ability to remember. The fact of the matter is, no one has ever taught you HOW to memorize what you need for tests. The Students Winning Edge is a proven, easy, fun and guaranteed way to learn and retain information taught in school. It is designed to teach you how to remember the information that is taught in your classes.

22. **HEARTS AND MINDS MUSIK TRACKS® FOR STUDENTS**: WARNING!!! Students who don't start lowering their stress levels immediately, allow stress to reach maximum levels – resulting in sleep loss, ruined test scores and the retention abilities of a squirrel. It piles up and does NOT go away even after a weekend of partying. If you want a fast, simple and super -

cheap way to quickly end exam stress for good – safe and natural without pills or doctors - then read this …...These MusikTracks use an exciting and powerful stress busting sound technology, called binaural music. When it is played alongside ordinary soothing music and listened to with stereo headphones, this creates remarkable positive changes in listeners. Thousands of people are using it and I've been hearing an avalanche of positive feedback about the benefits they've received.

23. **MAKING MATH MORE FUN**: "Making Math More Fun Printable Math Games" gives you 4 books jam packed full of kid math games and fun math activities to make math exciting and easy to learn. These are electronic books that are downloaded to your computer in a flash. Which means you can be reading them and playing all these games in as little as 5 minutes from now. Finding quality, kid math games isn't easy. But now you can have all of the kids' math games that you need.

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30. **CHEEKIE EARLY LEARNING SERIES**: A comprehensive resource you can be confident that you are not missing out on anything important in your child's early learning and development experience. The 5 Jumboworkbooks are fun, stimulating and educational for children aged 3- 7 years old.

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