PRODUCTION PROCESS OF BIO-DIESEL

A PROJECT OF ALTERNATIVE ENERGY DEVELOPMENT BOARD (AEDB)
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SYNOPSIS	:	This document gives the basic process of producing bio-diesel from any resource and at any scale and establishes guidelines to set-up production units of bio-diesel.
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LIST OF ABBREVIATIONS

CSTR	Continuous Stirred Tank Reactor
PFR	Plug Flow Reactor
SVO	Straight Vegetable Oil
WVO	Waste Vegetable Oil

SECTION 1

EXECUTIVE SUMMARY

Production of Bio-Diesel is a two phased process. It involves extraction of oil by pressing seeds in an oil expeller. The oil is then passed through a chemical reaction (Called Transesterification) to control its properties up to desired standards.

The oil may or may not be used straight after extraction from seeds. It depends upon the desired application like type of engine running on Bio-Diesel and the blending ratio. Moreover, the chemical reaction can be customized to achieve the required chemical properties.

The process of making bio-diesel remains same from laboratory setups to mega scale plants. The plant layout is designed based upon the production capacity. The economies of scale for production depends upon supply chain mechanism available for the plant.

This document explains the process of producing Biodiesel (Both oil extraction & Transesterification). Oil extraction is always batch type production process. However, transesterification can be continuous flow line type or otherwise batch type. Both types of processes are covered in this document.

SECTION-2

OIL EXTRACTION

Oil extraction is the process of getting neat oil from the seeds. The oil may or may not be further processed depending upon the requirement. Because of a wide range in physical characteristics of oil yielding seeds, various techniques have been developed to extract the oils and process them to finished products (Fangrui and Milford, 1999).

The common objective of all these processes is to

- Maximize the yield of fat or oil from the oil bearing material.
- Minimize the damage to the fat or oil and solid fraction.
- Produce components as free from undesirable impurities as much as possible.
- Produce a residual oil cake of the greatest possible value.

2.1 SEED PREPARATION

The process of oil extraction for Bio-Diesel production requires following steps of seed preparation.

2.1.1 DE-SHELLING

The requirement of de-shelling varies on case to case basis. The seeds of *Pongamia pinnata* specifically requires removal of seed coat. There are certain seeds that do not require de-shelling and can be processed as such. Seed Coat or shell reduces the yield of oil from seed during pressing. Because it absorbs some quantity of oil during the process.

2.1.2 SEED CLEANING

The second step in the commercial processing of oilseeds is cleaning. Its purpose is to remove foreign materials, such as sticks, stems, leaves, other seeds, sand and dirt. High capacity dry screeners, such as a Burnaby cleaner, are used to remove all materials that are over or under size, by utilizing a combination of screens and aspiration (Antolin and Tinaut, 2002). Permanent or electromagnets are also used for the removal of tramp iron objects. The cleaning is done carefully, so that the resultant oil is not contaminated with foreign materials.

Seed cleaning is an important step in the preparation of oil bearing materials for extraction. Proper seed cleaning increases oil yields and reduces machine maintenance. However, seed cleaning needs not to be a mechanized operation. Filtering of foreign elements manually (by labor) can do the needful to keep the process simple on a small scale.

2.1.3 SEED DRYING

If the oil bearing material is dry, it must be stored so that it remains dry, for optimum extraction and quality of oil. If the oil bearing material is wet plant tissue, it should be dried in sun. Oil in the presence of water deteriorate rapidly, forming free fatty acids and rancid off flavors.

2.1.4 GRINDING, ROLLING OR FLAKING

This is a recommended but not compulsory step considering that oil extraction is much more efficient if the starting material is in small particles.

However, it can be typically practiced for very large size seeds. Experiments have revealed that most of the bio-diesel producing seeds in Pakistan do not require this process.

Grinding the oilseed is one effective way to reduce the particle size. For example, a hand operated mortar, millstone grinder or even a kitchen mortar can be used to convert the seeds to a coarse meal. Small hammer mills, motor or hand powered, can also be used for grinding.

Another way to reduce particle size is to roll the oilseeds to produce flakes for extraction. Most commercial extraction plants find this to be most effective approach. With large oilseeds it may be necessary to rough grind the seed first, and then put the pieces through flaking rollers.

Either process makes the actual pressing more efficient. The final particle size that leads to most efficient extraction can best be determined by experiment, as the size will vary depending on the type of seed and the method of pressing operation. Generally, smaller sized pieces are better for oil removal, but if the pieces are too small, they may contaminate the oil and be difficult to remove during the subsequent filtration step.

2.1.5 HEATING

The final step in raw materials preparation prior to extraction is heating the ground or flaked oilseed to increase oil yields. The impact of this process remains very low specially after drying the seed in sunlight. The process is again not enforced on emerging technology of Bio-Diesel. It can become a part of continual process improvement.

Heating combats enzymes in the plant tissues which would have a detrimental effect on the oil quality (Saka and Kusdiana, 2001). If the oilseeds cake, i.e. the residue remaining after oil extration is to be used for feed or food, controlled heating may be useful in increasing protein availability in the resultant meal fraction.

Heating oil bearing materials prior to expelling tends to:

- Thermally rupture cells which have integrally survived the flaking process.
- Reduce oil viscosity and promote coalescing of minute droplets of oil.
- Increase the diffusion rate.
- Denature hydrolytic enzymes.
- Permit moisture control of the expeller feedstock to an optimum 4 to 6%. Inadequate moisture levels during pressing results in a granular discharge from the press or expeller, while excessive moisture results in a sloppy product. Either type of expeller cake will cause problems in the solvent extraction process.

Drum and stack type heaters can both be used satisfactorily for the heating of oilseeds. The drum heater may offer higher heat transfer rates, but at the expense of the integrity of the fragile flakes. Typical stack heaters consist of a series of four to eight vertical, closed, cylindrical steel kettles, with each kettle usually 30 to 50 cm in diameter and 50 to 70 cm high. Oilseeds are usually moistened before heating, or during the early stages of heating, unless they are initially high in moisture content. An initial moisture content of 9 to14% is commonly used in the top kettle of a heater. During heating, careful moisture control is important, because moisture content affects the affinity between the seed and the oil.

Proper heating results in:

- The complete breakdown of oil cells.
- Coagulation of the proteins to facilitate the oil and meal separation.
- In-solubility of the Phospholipids.
- Increased fluidity of the oil at higher temperature.
- Destruction of molds and bacteria.
- Inactivation of enzymes.

- Drying to suitable moisture content.

2.2 SEED CRUSHING

2.2.1 CRUSHING YIELDS

Oilseeds with relatively high oil contents are usually processed first by mechanical presses. Table - I represents oil crops processed by mechanical pressing and their oil contents.

SEED	OIL CONTENT %
Pongame (Sukh Chane)	25
Castor	50
Cotton Seed	30
Rape seed/Canola	40
Sunflower seed	35

Table- I: Oil Content in Seeds

In addition to the oil content of the source material, the fatty acid composition is also important for establishing processing conditions. Vegetable oil remains liquid at room temperature, because of the degree of un-saturation and the number of carbons in the fatty acid radicals of the triglyceride.

2.2.2 CRUSHING EQUIPMENT

(a) PRESSES

In many parts of the world, commercial hydraulic presses are the most practical and economical way to extract oil form seeds. They may be powered either by hand or by electricity. Ground seed material or wet plant tissue is placed in the press in layers, with each layer separated from the next by a press cloth. Pressure is applied, slowly at first and then increased, as the oil content in the tissue decreases. Maximum total pressure is 13970 KPa for one inch layers. Total time to load, apply pressure and remove cake is approximately one hour. Drainage of the oil while under pressure may require 30-45 minutes.

The advantages of the commercial size batch press are:

- They can be driven by hand or electricity
- They are simple and economical to operate and maintain
- They require minimum operator training and provide an excellent oil recovery.

Disadvantages include:

- The substantial cost of the machinery
- Long delivery times
- Difficult to obtain spare parts in remote areas
- Necessity of electric power to operate larger models.

(b) EXPELLERS

This is a more recommended option. Continuous screw presses, or expellers, are used in higher technology areas throughout the world for the expulsion of oil where there is sufficient seed supply to justify a continuous operation. Sufficient pressure is achieved by means of an auger that turns inside a barrel (Vicente and Coteron, 1998). The barrel is closed, except of small openings, through which the oil drains and a choked outlet for departing pressed cake.

Expellers exert much greater pressure on seed cake than that produced by a hydraulic batch press. This increased pressure results in a greater recovery of oil content in the feedstock. Expellers can vary in size from units that process

40 kg of conditioned seed per hour, to machines that process 200 tones of seed per hour. Expellers are an essential part of most modern oilseed extraction plants that employ a prepress step in the oil extraction process.

Expellers have a higher plant capacity and a higher rate of oil recovery with a lower labor requirement than the press system.

FIGURE-I: Screw Press

Disadvantages of the expeller system include the requirement of electric power, continuous operation and availability of skilled mechanics. Equipment and maintenance costs are also high. The oil produced contains more impurities and must be heated and filtered for edible use.

2.2.3 CRUSHING TECHNIQUE

The crushing objective is to remove from each microscopic oil cell within each tiny seed, the maximum quantity of oil present without chemically altering the oil or protein quality. During extraction, the various processing steps can never improve the quality of oil, but only minimize the damage done to the oil. The following steps are essential for the extraction operation.

- Rupture the cell wall.
- Obtain diffusion and agglomeration of the oil.
- Make a final and complete separation of liquid and solids.

In recent years, increased mechanization and higher labor costs have made hydraulic pressing of oilseeds uneconomical for modern commercial operation. Continuous expellers or screw presses are now used for the mechanical extraction.

There is general agreement throughout the industry that pre-pressing followed by solvent extraction in medium to large scale plants give better overall economy when high oil content seeds are processed. This holds true, despite the higher power requirements and mechanical maintenance costs required of the prepress operation. Cooked flakes, of crops such as rapeseed, canola containing 4% moisture and 44% oil, are fed to a series of screw presses or expellers, to receive a mild pressing operation (Kumar et al., 2003). This removes most of the oil while avoiding excessive pressure, power consumption and temperature. These expellers will reduce oil content of well flaked and cooked rapeseed from 42% to 15%, with a power consumption of 125 to 300 H.P., at an output of up to 250 tones per day, per unit.

On a continuous basis, each screw press gradually increases the pressure on the incoming material as it progresses through the interior of a closed barrel. There are provisions for the oil to drain out through small gaps between carefully positioned, hardened steel bars in the barrel cage. A column or plug of compressed meal is formed by a choke device at the discharge end of the barrel. This acts as a continuous hydraulic press head with new cake being formed at the choke, as the old cake is continuously discharged from the expeller, past the choke device. Good quality cake is considered to be spongy and permeable and resists disintegration during conveying to the solvent extractor.

Pressing also consolidates the tiny flakes into larger units of cake fragments. Cake fragments substantially larger than the original flakes are essential in the subsequent extraction operation to obtain satisfactory rates of solvent gravity percolation through the cake bed. Some compromise must be accepted between percolation rate, which is a function of size and thickness of the cake fragment reaching the extractor, and the mean diffusion dimension, cake thickness. Fortunately, cake thickness is the one and only dimension that can be directly controlled at the press and it also remains the most durable physical parameter throughout the conveying and extraction operations.

The coordination of the flaking, cooking and press operations is still considered to be more of an art rather than an exact science. **Good cake is characterized as having a dry texture, with a preferred moisture level of 4 to 5 % (6% maximum) and an oil content of 15 to 18 %. The cake should be between 1/8 to 3/16 of an inch thick with good physical integrity and durability.**

2.3 OIL SETTLING & FILTERING

All expelled press oils contain some entrained solid matter and are allowed to settle in a screening tank through gravity. Settled oil is continuously drawn off from the screenings tank and the remaining suspended fines in the oil can be removed by either filtration or centrifugation.

The filtration can be done by simple means to remove elements of large size. For a very high quality, a totally enclosed multiple screen type filter can be deployed but it increases the production cost. The enclosed filtering plates consist of double sided stainless steel screens, pre-coated with the fines themselves. Many units are powered to open and close for cleaning and some are fully automated.

The simplicity of filtration also depends upon the transesterification, which also purifies the product.

FIGURE-II: Oil Filtration Unit

2.4 SOLVENT EXTRACTION

When dealing with relatively low oil content materials, extraction with a solvent, normally hexane is an efficient technique for oil recovery. Oil produced by this method is of high quality, because very little heat treatment is required. In addition, the resultant meal fraction contains protein which has encountered a minimum amount of deterioration from heat damage.

There are however, several disadvantages related to the solvent extraction process, due to which, it is not recommended as a common practice. The disadvantages include:

- More expensive equipment compared to other extraction systems.

- The danger of fire and explosion unless non-flammable solvents can be used.
- The direct extraction of raw unheated flakes may contain material that is toxic to no ruminants. Therefore, products of direct solvent extraction such as cotton flakes require additional treatment to ensure adequate inactivation of such contaminants.

2.5 HIGH PRESSURE EXTRACTION

A new experimental method for extracting fats and oils from oilseed crops utilizes carbon dioxide, heated and compressed above its critical temperature and pressure to alter its properties. Such supercritical carbon dioxide is an ideal solvent because it is nontoxic, non explosive, cheap, readily available and easily removed from the extracted products (Antolin and Tinaut, 2002). It is as efficient as hexane at removing triglycerides while yielding a high quality, gum free, light colored crude oil with low iron content.

This methodology is still under experiments and additional development work is required to overcome the high pressure engineering problems related to an economical high volume continuous process.

SECTION-3

TRANSESTERIFICATION

3.1 TRANSESTERIFICATION

Transesterification is the process of reacting a triglyceride molecule (oil extracted from seed) with an excess of alcohol in the presence of a catalyst (KOH, NaOH, NaOCH3, etc.) to produce glycerol and fatty esters. It reduces the high viscosity of triglyceride oils. In this process, the long fatty acid chains are removed from the glyceride molecule by reacting with alcohol and a catalyst. Common catalysts are potassium hydroxide, sodium hydroxide, and sodium methoxide (Antolin and Tinaut, 2002). The reaction produces fatty monoesters and free glycerin. Any remaining unreacted monoglycerides, diglycerides, or triglycerides make up the bonded portion of the remaining glycerol in the fuel. Together, the free and bonded glycerol make up the total glycerol percentage remaining. This total glycerol percentage is used to determine the completion of the reaction.

- **TRI-GLYCERIDES**

  Molecules having three fatty acid chains are referred to as triglycerides.

- **DI-GLYCERIDES**

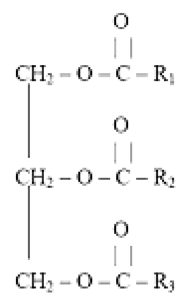
  Those molecules which have two fatty acid chains are called di-glycerides.

- **MONO-GLYCERIDES**

  Molecules with one fatty acid chain are called mono-glycerides.

- **METHYL ESTERS**

The monoesters commonly known as Bio-Diesel are usually produced through the transesterification of vegetable oils or animal fats. Both oils and fats are triglycerides or fatty esters of glycerin. Fat usually refers to the triglycerides which are solid at room temperature while oils are liquid at room temperature. The triglyceride molecule has the chemical structure shown in figure given below, where R1, R2, and R3 represent long chain fatty acids.



Triglyceride Chemical Structure.

The final product after reaction with methanol is methyl esters. Other alcohols may be used, such as ethanol or butanol, resulting in ethyl esters and butyl esters, accordingly.

3.2 CHEMICAL REACTION

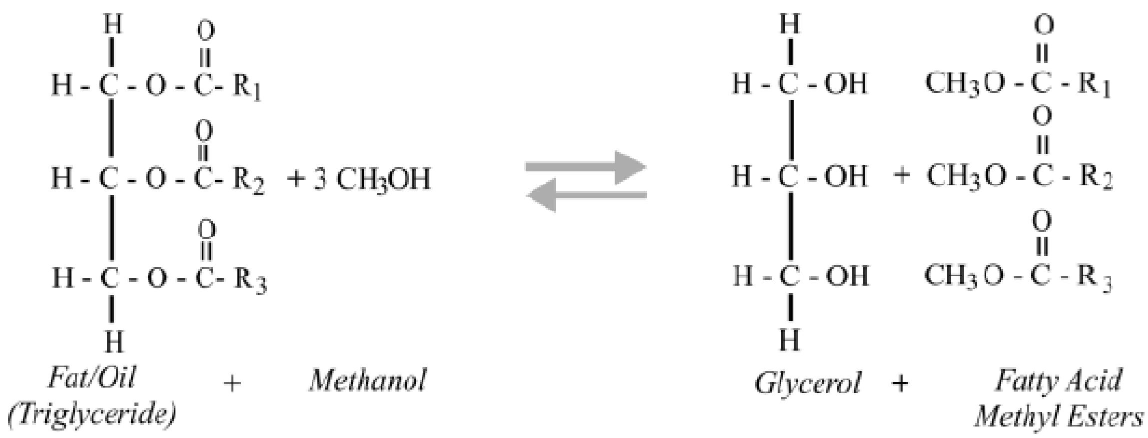


FIGURE-III: Chemical Reaction of Bio-Diesel Production

3.3 PRODUCTION PROCESS

As mentioned above Bio-Diesel can be produced from straight vegetable oil (SVO), animal oil / fats, tallow and waste vegetable oil (WVO). For this purpose base catalyzed transesterification of the oil is performed.

3.3.1 INGREDIENTS REQUIRED

(a) For Mixture

1- vegetable oil -- used cooking oil, fryer grease, animal fats, lard, plants oil

2- Methanol (CH_3OH) -- 99% + pure

3- Sodium hydroxide (NaOH -- caustic soda, lye) -- must be dry

(b) For Titration

1- Isopropyl alcohol (rubbing alcohol) -- 99%+ pure

2- Distilled water

3- Phenolphthalein solution (not more than a year old, kept protected from strong light) - "Phenol" or "Phenol Red" from swimming pool or hot tub supply stores may not be the same as phenolphthalein; it can be used but the directions for use may be different.

(c) For Washing

1- Vinegar
2- Water

3.3.2 FILTRATION AND PRE HEATING

Filtration of oil is carried out through oil filter or by using cotton cloth placed on a funnel and oil is poured through this cloth into a beaker. Filtration is carried out to remove unnecessary particles or contaminations present in the oil. Heat the oil after filtration up to 120 °C to remove water contents present in the oil. After heating, the oil is allowed to cool down to 60 °C.

3.3.3 MIXING OF ALCOHOL AND CATALYST

The catalyst is typically sodium hydroxide (caustic soda) or potassium hydroxide (potash). It is dissolved in alcohol (Methanol) using a standard agitator or mixer producing Sodium Methoxide (Kumar et al., 2003). The agitation time is almost one hour. Generally the amount of methanol needed is 20% of the oil by mass. The densities of these two liquids are fairly close so measuring 20% of methanol by volume should be about right.

CAUTION:

Do not inhale any vapors. If any sodium methoxide gets splashed on your skin, it cause burning with out feeling it (killing the nerves) - wash immediately with lots of water. Always have a hose running when working with sodium methoxide. Sodium methoxide is also very corrosive to paints.

3.3.4 REACTION

The alcohol / catalyst mixture is charged into a closed reaction vessel and the oil or fat is added. The system from this point onwards is preferably closed to the atmosphere to prevent the loss of alcohol. The reaction mix is kept just above the boiling point of the alcohol (around 160 °F) to speed up the reaction. Recommended reaction time is about 1 hour. Excess alcohol is normally used to ensure total conversion of the fat or oil to its esters. Care must be taken to monitor the amount of water and free fatty acids in the incoming oil or fat. If the free fatty acid level or water level is too high it may cause problems with soap formation and the separation of the glycerin by-product downstream.

3.3.5 SETTLING & SEPARATION

The solution is allowed to sit and cool for at least eight hours. The methyl esters Bio-Diesel floats on top while the denser glycerin congeals on the bottom of the container forming a hard gelatinous mass.

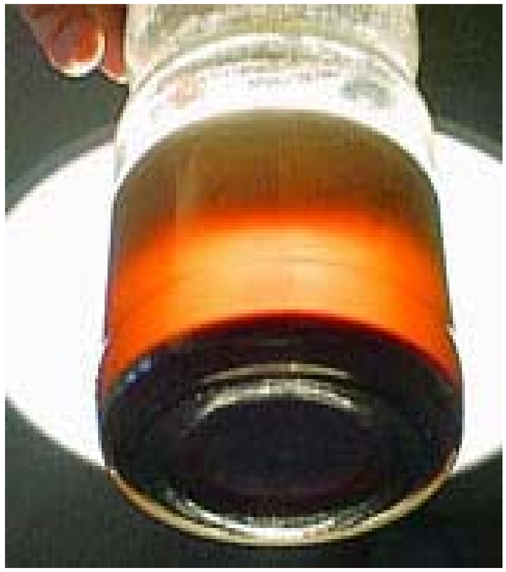


FIGURE-IV: Glycerin Settled Down

An alternative method is to allow the reactants to sit for at least an hour after mixing while keeping the brew above 100 deg F (38 deg C), which keeps the glycerin semi-liquid (it solidifies below 100 deg F). Then carefully decant the bio-diesel.

This can be done by draining the reactants out of the bottom of the container through a transparent hose. The semi-liquid glycerin has a dark brown color; the Bio-Diesel is honey-colored. There has to be a close watch on what flows through the sight tube. When the lighter-colored bio-diesel appears deliver it to a separate container. If any bio-diesel stays with the glycerin it is easy to retrieve it later once the glycerin has solidified.

In case, mixture is left in the tank until the glycerin gelled, reheating is required to liquefy the glycerin again. It is then decanted out as above without stirring.

Once the reaction is complete, two major products exist: glycerin and bio-diesel. Each has a substantial amount of the excess methanol that was used in the reaction. The reacted mixture is sometimes neutralized at this step if needed. The glycerin phase is much denser than bio-diesel phase and the two can be gravity separated with glycerin simply drawn off the bottom of the settling vessel. In some cases, a centrifuge can be used to separate the two materials faster.

3.3.6 ALCOHOL REMOVAL

Once the glycerin and bio-diesel phases have been separated, the excess alcohol in each phase is removed with a flash evaporation process or by distillation. In others systems, the alcohol is removed and the mixture neutralized before the glycerin and esters have been separated. In either case, the alcohol is recovered using distillation equipment and is re-used. Care must be taken to ensure no water accumulates in the recovered alcohol stream.

3.3.7 GLYCERIN NEUTRALIZATION

The glycerin by-product contains unused catalyst and soaps that are neutralized with an acid and sent to storage as crude glycerin. In some cases the salt formed during this phase is recovered for use as fertilizer. In most cases the salt is left in the glycerin. Water and alcohol are removed to produce 80-88% pure glycerin that is ready to be sold as crude glycerin. In more sophisticated operations, the glycerin is distilled to 99% or higher purity and sold into the cosmetic and pharmaceutical markets.

3.3.8 WASHING & DRYING

Once separated from the glycerine, the Bio-Diesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps, dried, and sent to storage. This is normally the end of the production process resulting in a clear amber-yellow liquid with a viscosity similar to petro-diesel. In some systems the bio-diesel is distilled in an additional step to remove small amounts of color bodies to produce a colorless bio-diesel. Washing is typically repeated thrice for best quality. Each time, water of equal volume as of bio-diesel and vinegar in minute quantity is added and left for 2-3 hours. Water due to its higher density settles down at the bottom and recovered. Thus Bio-Diesel is separated after washing as shown in the figure given below.

FIGURE-V: Pure Bio-diesel after Washing

3.4 PRODUCT QUALITY

Prior to use as a commercial fuel, the finished bio-diesel must be analyzed using sophisticated analytical equipment to ensure it meets any required specifications. The most important aspects of bio-diesel production to ensure trouble free operation in diesel engines are:

- Complete Reaction
- Removal of Glycerin
- Removal of Catalyst
- Removal of Alcohol
- Absence of Free Fatty Acids

3.5 TITRATION

To determine the correct amount of lye (NaOH) required, a titration is advised on the oil being transesterified.

3.5.1 PROCEDURE

Make up a solution of one gram of lye to one liter of distilled water. 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. Mix 10 milliliters of isopropyl alcohol in a small container with a 1 milliliter sample of oil make sure it's exactly 1 milliliter (Vicente and Coteron, 1998). Take the oil titration sample from the reaction vessel after it's been warmed up and stirred. Add to this solution 2 drops of phenolphthalein, an acid-base indicator that's colorless in acid and red in base.

Using a graduated eye dropper (with increments marked in tenths of milliliters) or some other calibrated instrument (from medical supply outlets), while carefully keeping track of the amounts, drop measured amounts of the lye/water solution a couple of tenths of milliliters at a time into the oil/isopropyl/phenolphthalein solution.

Follow each drop with vigorous stirring of the solution. In cold weather the oil might congeal and not work so it might need to do the titration in a heated room. If conditions are right eventually the solution turns pink (magenta), and stays pink for 10 seconds. This is the indicator color for a pH range of 8-9, "Color of titrated liquid sample when at the correct pH"). It's important to find the exact amount, to just reach this pH without dropping in too much. It is a good idea to do this entire process more than once to ensure that your number is correct. It was found that depending on the type of oil, how hot it got in the fryer, what was cooked in it and how long it was used, the amount of lye/water solution needed to titrate it is usually 1.5 to 3 milliliters. Litmus paper or a digital pH tester can also be used instead of the phenolphthalein. Try it with fresh cooking oil in kitchen too; it should need much less lye to reach pH 8-9.

NOTES

1- The lye (NaOH or KOH) must be dry, keep it away from water, store it in an airtight container.

2- Phenolphthalein has a shelf life of about a year; it is very sensitive to degradation by light so after a while it will start giving erroneous readings.

3.5.2 THE CALCULATION

The next step is to determine the amount of lye needed for the reaction. Take the number of milliliters derived from the titration and multiply by the number of liters of vegetable oil to be transesterified. There is one more thing to be included in the calculation. Every liter of neat vegetable oil (fresh never been burned) needs 3.5 grams of lye for the reaction. So for every liter of vegetable oil to be transesterified add an additional 3.5 grams of lye. If the titration result was 1.8 milliliters to reach pH 8-9, the final amount of lye needed for the reaction would be 795 grams. The number of grams of lye needed per liter of vegetable oil has generally been between 6 and 7.

3.6 BY PRODUCTS OF BIO-DIESEL PRODUCTION PROCESS

By products obtained from the Bio-Diesel production process are explained below:

3.6.1 GLYCERIN

The glycerin from used vegetable oil is brown and usually turns to a solid below about 100 deg F (38 deg C). Glycerin from fresh oil often stays as liquid at lower temperatures.

Reclaimed glycerine can be composted after being vented for three weeks to allow residual methanol to evaporate off or after heating it to 150 deg F (66 deg C) to boil off any methanol content (the boiling point of methanol is 148.5 deg F, 64.7 deg C). The excess methanol can be recovered for re-use when boiled off if you run the vapors through a condenser.

Another way of disposing of the glycerine, though a great bit more complicated, would be to separate its components, mostly methanol, pure glycerine (a valuable product for medicines, tinctures, hand lotions, dried plant arrangements and many other uses) and wax. This is often accomplished by distilling it, but glycerin has a high boiling point even under high vacuum so this method is difficult.

The glycerine by-product makes an excellent industrial-type degreaser / soap. One way to purify it is heat it to 150 deg F (65.5 deg C) to boil off excess methanol, making it safe for skin contact (take precautions with fumes). Once the glycerin is back to a liquid the impurities sink to the bottom and the color will become a more uniform dark brown (Antolin and Tinaut, 2002). This can be cut with water leaving it a tan color, less concentrated and softer and easier to handle when washing hands. Produced this way the degreaser could be sold in squeeze or pump dispensers.

Other ideas for disposing off the glycerine are breaking it down to usable methane gas, with a methane digester or, for a much wilder idea; it could be broken down with pyrolisis. (Pyrolisis was used extensively to run cars on firewood in oil-scarce Europe and elsewhere during World War 2). The processor has a heat source that heats the fuel (wood or glycerine) in an airtight box without oxygen. This allows the fuel to release its methane while not allowing it to burn. The methane is trapped in an inflatable storage container or compressed into a tank. This is an area of biodiesel development that warrants further work.

3.6.2 SOAP RESIDUE

Suspended in the biodiesel will also be some soapy residues. These are the result of Na+ ions from the sodium hydroxide (NaOH) reacting with water created when the methanol bonds with the ester chains along with any other water that was suspended in the oil.

If the reaction produces more than the usual amount of soap, this happens when lye comes into contact with water before it has a chance to react with the oil. In this case, the excess water should have been boiled off first..

The part of the process where it's vital to keep all water out of the reaction is when making the sodium 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 on a warm dry summer day than on a damp winter day.

SECTION-4

PROCESS LAYOUTS

4.1 INTRODUCTION

There are multiple operating options available for making biodiesel. Many of these technologies can be combined under various conditions and feedstocks in an infinite number of ways. The technology choice is a function of desired capacity, feedstock type and quality, alcohol recovery, and catalyst recovery. The dominant factor in biodiesel production is the feedstock cost, with capital cost contributing only about 7 % of the final product cost. However, some reaction systems are capable of handling a variety of feedstocks and qualities, while others are not. Also, the different approaches to the esterification process result in quite different operating requirements, different water use requirements, and different operating modes. In general, smaller capacity plants and variable feedstock quality suggest use of batch systems.

Continuous systems generally lead the operation on a 24/7 basis, requiring larger capacities to justify larger staffing requirements and require a more uniform feedstock quality.

4.2 BATCH PRODUCTION SYSTEM

The simplest method for producing alcohol esters (Bio-Diesel) is to use a batch, stirred tank reactor system. Alcohol to triglyceride ratios from 4:1 to 20:1 (mole: mole) is used, with a 6:1 ratio most common. The reactor may be sealed or equipped with a reflux condenser. The operating temperature is usually about 65°C, although temperatures from 25°C to 85°C can also be used. The most commonly used catalyst is sodium hydroxide, with potassium hydroxide can also be used. Typical catalyst loadings range from 0.3 % to about 1.5%.

Thorough mixing is done at the beginning of the reaction to bring the oil, catalyst and alcohol into intimate contact. Towards the end of the reaction, less mixing can help increase the extent of reaction by allowing the inhibitory product, glycerol, to phase separate from the ester – oil phase. Completions of 85% to 94 % are resulted. There is another two-step reaction, with glycerol removal between steps, to increase the final reaction extent to 95 percent. Higher temperatures and higher alcohol: oil ratios also can enhance the percent completion. Typical reaction times range from 20 minutes to more than one hour.

4.2.1 PROCEDURE

The oil is first charged to the system, followed by the catalyst and methanol. The system is agitated during the reaction time. Then agitation is stopped. In some processes, the reaction mixture is allowed to settle in the reactor to give an initial separation of the esters and glycerol. In other processes the reaction mixture is pumped into a settling vessel, or is separated using a centrifuge. Figure - VI shows a process flow diagram for a typical batch system.

The alcohol is removed from both the glycerol and ester stream using an evaporator or a flash unit. The esters are neutralized, washed gently using warm, slightly acid water to remove residual methanol and salts, and then dried. The finished biodiesel is then transferred to storage (Fangrui and Milford, 1999). The glycerol stream is neutralized and washed with soft water. The glycerol is than sent to the glycerol refining section.

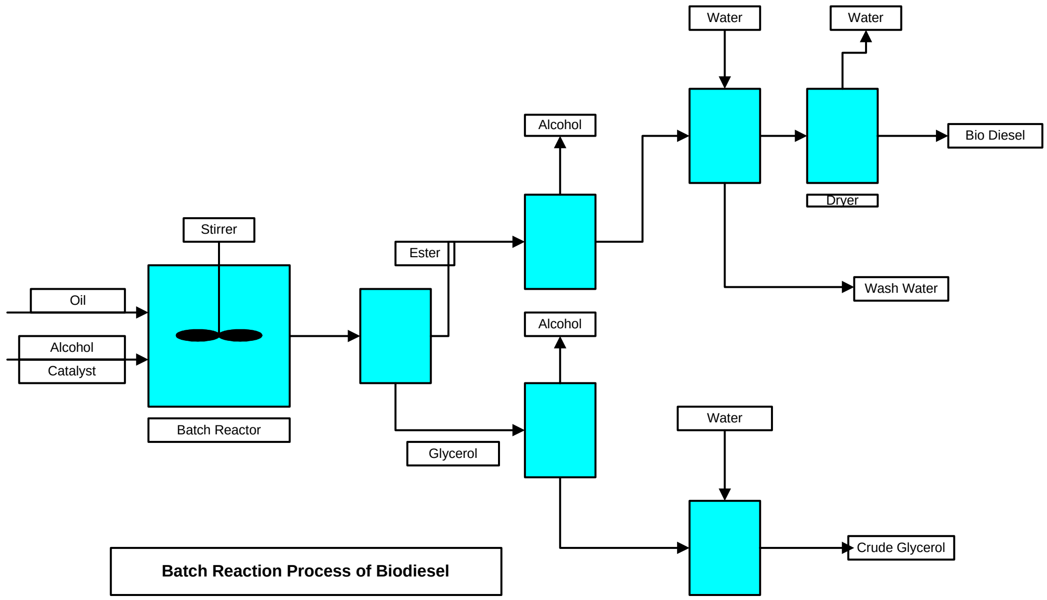


FIGURE-VI: Batch Flow Line of Biodiesel Production

FIGURE-VII: Batch Production Kit

4.3 CONTINUOUS PRODUCTION SYSTEM

A popular variation of the batch process is the use of continuous stirred tank reactors (CSTRs) in series (Fangrui and Milford, 1999). Such type of system is called continuous production system. The CSTRs can be varied in volume to allow for a longer residence time in CSTR 1 to achieve a greater extent of reaction. After the initial product glycerol is decanted, the reaction in CSTR 2 is rather rapid, with 98+ completions not uncommon. An essential element in the design of a CSTR is sufficient mixing input to ensure that the composition throughout the reactor is essentially constant. This has the effect of increasing the dispersion of the glycerol product in the ester phase. The result is that the time required for phase separation is extended. There are several processes that use intense mixing, either from pumps or motionless mixers, to initiate the esterification reaction. Instead of allowing time for the reaction in an agitated tank, the reactor is tubular. The reaction mixture moves through this type of reactor in a continuous plug, with little mixing in the axial direction. This type of reactor, called a plug-flow reactor (PFR), behaves as if it were a series of small CSTRs chained together. The result is a continuous system that requires rather short residence times, as low as 6 to 10 minutes, for near completion of the reaction. The PFRs can be staged, as shown, to allow decanting of glycerol. Often this type of reactor is operated at an elevated temperature and pressure to increase reaction rate. A PFR system is shown in Figure - VIII.

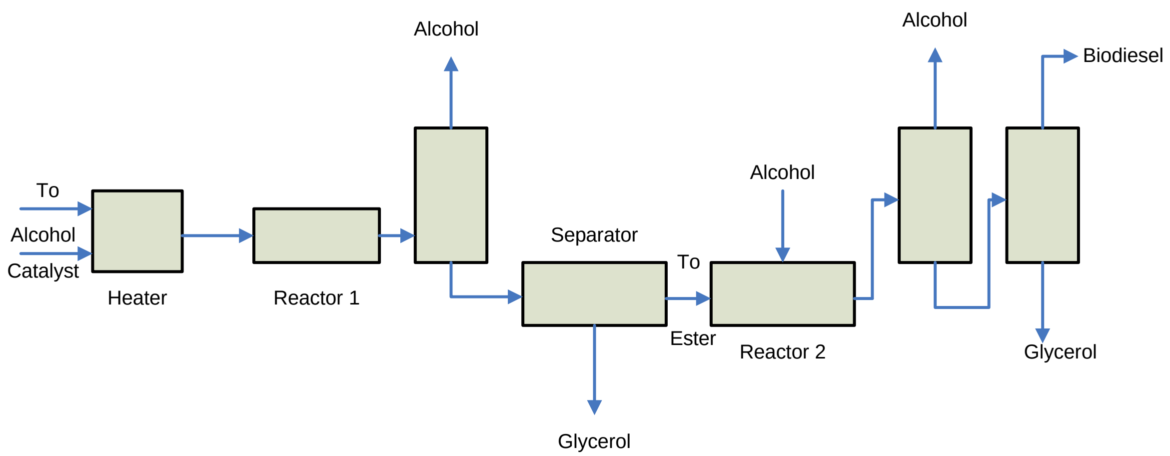


Figure. Continuous Plug Flow System of Biodiesel

FIGURE-VIII: Continuous Flow Line for Bio-diesel Production

FIGURE-IX: Continuous Flow Production Kit

4.4 COMPARISON BETWEEN BATCH PRODUCTION AND CONTINUOUS SYSTEM

- Batch type production system of bio-diesel is better suited to smaller plants (<1 million gallons/yr).
- Batch does not require 24/7 operation.
- Batch provides greater flexibility to tune process to feedstock variations.
- Continuous system allows use of high-volume separation systems (centrifuges) which greatly increase throughput.
- Hybrid systems are possible.

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