BIODIESEL PRODUCTION
AT THE AGRICULTURAL ENGINEERING DEPARTMENT

University of Idaho

ETHYL ESTER PROCESS

Ethyl ester production from winter rapeseed oil described in this paper is derived from the techniques developed by Cruz (1992). An extensive lab or bench scale studies were conducted to identify the optimum conditions and concentrations for a successful transesterification at room temperature. The total procedure is illustrated in Fig. 1 of the last page.

Disclaimer: The procedure described in this paper is not to be construed as a recommendation for the use of alternative fuel mentioned. It is the responsibility of the engine operator to make the decisions concerning the use of alternative fuels. Biodiesel production involves certain hazardous and flammable materials. The personnel that may involve in the production must follow standard safety handling procedures, and the use of suitable available equipment must be considered before attempting to do some biodiesel production.

1. **Clean** and **dry** the inside surfaces of the biodiesel reactor and the alcohol-catalyst mixing container. Make sure **no water** is present in such surfaces during the addition of oil and alcohol into the containers.

2. **Pump** the needed amount of rapeseed oil into the reactor. Refer to the recipe example below. Make sure the pump is **clean** and **devoid of water.** If the pump was previously used for other purposes other than oil, ester and alcohol, use anhydrous liquid to flush the pump before pumping the oil into the reactor.

3. **Pump** the required amount of ethanol[1] into the mixer, (70% stoichiometric amount excess, or a molar ratio of 5.2:1 EtOH to oil). Refer to the recipe example described in **step 4** of this procedure. Make sure the pump is **clean** and **free of water.** If the pump was previously used for liquids other than ethanol, flush the pump with anhydrous ethanol or acetone.

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[1] - Every new ethanol must be tested first for its purity. A lab-scale transesterification may be done prior to any big batch process. This can be done by processing a 100 g oil, 25 g ethanol and 1.3 g KOH. Melt the KOH in the ethanol and add them into the flask or beaker which contains the oil. Agitate the mixture vigorously using magnetic stirrers. A reaction can be observed within 35 seconds, exhibiting color change and clearin up. Stir the mixture for 30 minutes. Let settle for about two hours. If separation can be observed, then the ethanol 200 proof.

4. **Weigh accurately** the required amount of catalyst (1.3% KOH based on the amount of input oil, by weight). See the recipe below:

MATERIALS	QUANTITY	DISTANCE FROM CONTAINER TOP TO LIQUID SURFACE
Rapeseed oil	500 liters	61.5 cm
Ethanol, 200 proof	137 liters	28.7 cm
Potassium hydroxide, pellets (85% pure)	9.91 kg	

For any quantities other than above, use the following equation to obtain the exact quantities of ethanol and catalyst:

$$ETOH = 0.2738 \times RO$$

$$KOH = 0.0118 \times RO$$

where;

ETOH = amount of ethanol needed, in liters
RO = the desired amount of oil to be processed, in liters
KOH = amount of KOH needed, in kg

For **batches greater than 530 liters (140 gals)** of input rape oil, the **system pump of the reactor must be used, or the mixer motor must be upgraded** to higher power. The existing mixer motor (1/3 hp) cannot deliver enough vigorous agitation above 530 liters of input oil.

If the pumps have inaccurate metering system, **use the reactor and the catalyst-alcohol plastic barrel as the measuring device.** Both containers have been calibrated and the distance from container-top-to-liquid-surface equation below should be used:

Using the alcohol-catalyst mixer;

$$H_m = 79.5246 - 0.3713 \times ETOH$$

Using the biodiesel reactor;

$$H_r = 110.7987 - 0.0987 \times RO$$

where;

H_m = height from container top to surface of ethanol, cm
H_r = height from reactor top to surface of oil level, cm
RO = quantity of rapeseed oil desired
ETOH = quantity of ethanol needed

5. Move the catalyst-alcohol mixing container beside the reactor to allow for the reactor mixer to be used in stirring the ethanol and catalyst. Use the reactor motor mixer to accomplish the stirring. The mixing motor and shaft are movable. **Pour** the catalyst into the alcohol. **Follow safety measures** in preparing the catalyst and ethanol. **Melt** the catalyst by **vigorously stirring** the mixture for at least **15 minutes.**

6. **Transfer** the catalyst-alcohol mixture into the oil in the reactor using the same pump utilized in pumping the ethanol.

7. Relocate the mixing motor and shaft to their original position. **Stir vigorously** the whole mixture for **30 minutes.** Make sure **splashing** of the mixture or turbulent surface can be ubserved. During the **first minute of vigorous agitation**, the mixture **changes color and becomes clear.** Changing color and clearing up are indications of a successful process.

8. Let the mixture **settle** for at least **20 hours.** After settling, two liquid phases are produced. The upper liquid constitutes the **ethyl ester.** This liquid is **clear** and **golden yellow or light yellow** in color. The bottom liquid constitutes the **crude glycerol.** The liquid is **dark brown or yellow brown, translucent and thicker.** In most cases, the liquid interphase can be seen by looking from above the ester phase.

9. **Separate** the two liquid phases very carefully. The best way is to **drain or pump** out the crude glycerol first. **Draining** can be accomplished by elevating the reactor with a forklift for about 3 ft. Open the drain valve of the reactor, and funnel the crude glycerol into a barrel or any suitable container. **Observe the color and consistency** of the exiting fluid. When the liquid **change from dark to light,** with the presence of whitish soap material, and the liquid flows faster, **funnel** the liquid in a **separate container** (a 5-gal bucket is enough). This liquid constitutes the interphase and some ester can be saved from this interphase after further settling. When the **exiting liquid becomes very clear** and the color is light yellow or golden yellow, funnel the liquid into the ethyl ester storage tank. Another way to determine how much crude glycerol to be drained, watch out the volume of the drained liquid. Normally the **volume of crude glycerol is 90% that of the input ethanol.** Or the **volume of the ethyl ester is equivalent to the volume of input rapeseed oil.** Other ways to separate the two liquids is to pump each phases carefully. Place the ethyl ester in a clean storage container. **Any impurities will contaminate the fuel,** and make the fuel inferior in quality. The **presence of any visible glycerol** in the ester will **encourage the continued development of glycerol.** Thus, **glycerol must be removed immediately.**

10. The separated ethyl ester now becomes a **BIODIESEL** and is ready to be used in engines.

Precaution:

The fuel, if not directly used may continue to have internal reaction or reversal reaction that may lead to glycerol formation. In tests, glycerol formed about 0.5% per month based on the amount of the ester being stored. The glycerol is thought to cause some gumming in the engine's combustion chamber. If an old fuel is to be used, make sure that it is not contaminated with the glycerol that may form over the time the fuel was stored. The best thing to do is drain the glycerol first, and pump the top layer of the fuel into the fuel tank.

Solution:

Follow one of the optional steps below to stop the continuous glycerol formation:

OPTIONS:

The following options are strongly felt unnecessary. It is the operator's decision to follow any of these options. The total fuel **production time will be longer, at least four times longer than the preceding procedure and add more cost.** The washing option adds **at least $1.40 per gallon** of ester washed, unless a cheap acidifying agent is purchased. The additional cost is based on the current price of the added chemicals only. Any of the options entails at least 5 working days to produce one batch. Without the options, a batch can be done per day.

11a. **Neutralization**

11a-1. The ethyl ester can be neutralized with an addition of an acid solution. Add an amount of 0.08% HCl (36-40% pure) acid solution based on the amount of the ester. Stir the acid in the ester for 5-10 minutes. The mixture becomes murky. Neutralization accomplishes two tasks. First, it will bring back the pH of the ester (alkali, about 9.5 pH) to the original oil pH, which is about 7.0 (neutral). Secondly, the acid will inhibit any reversal or further reaction within the ester phase.

11a-2. Let the liquid settles for several hours until the ester becomes very clear again. Settling usually takes 3-4 days. If a centrifuge is available, settling is accomplished in less than 30 minutes. Remove the sediments by draining or pumping, or transfer the ethyl ester into a storage tank. The **BIODIESEL** is now ready for engine use. **Make sure no impurities or foreign materials are found** in the ester. If so, **remove the contaminants immediately.**

11b. **Washing**

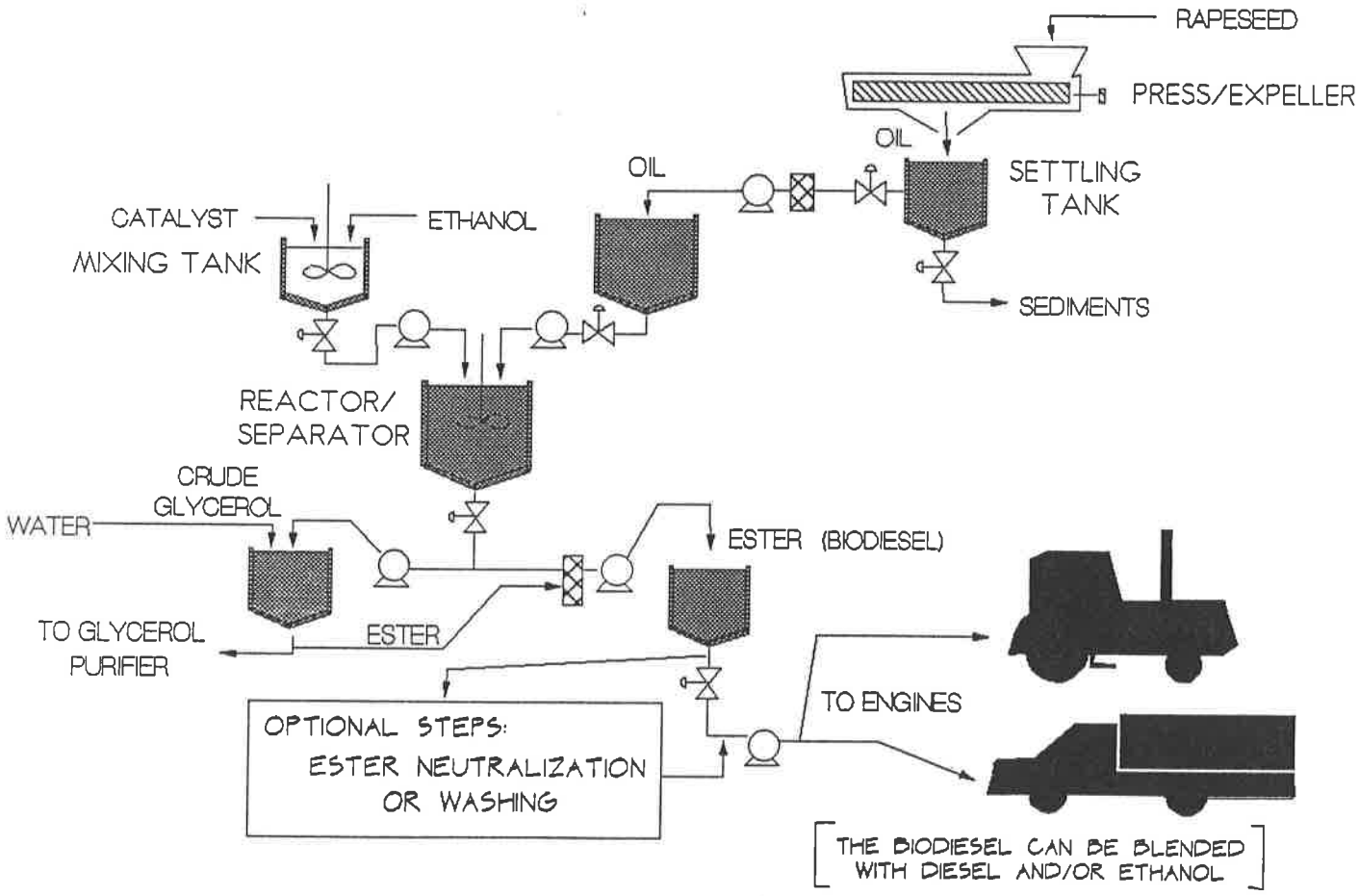
11b-1. The ethyl ester can be washed with tap water. However, washing should be done very gently. To minimize emulsion formation, acidify the water until it has a pH of 2.0. Acidification of the water can be done by melting an amount of citric aid in the water. About 17 grams citric acid (99+ % pure) per liter of water is needed to bring the pH to 2.0. When using sprinkle or trickle washing, make sure that the terminal velocity of the water is gentle and slow as it reaches the ester. Otherwise, a considerable amount of emulsion is produced when water is in contact with the ester. Up to 20% of the ester may be lost due to emulsion formation.

11b-2. Let the liquid settle for several hours until the ester becomes very clear. Settling usually takes 3-5 days. If a centrifuge is available, separation time can be accomplished in less than 30 minutes. Separate carefully the wash water and liquid interphases by draining or pumping them first. Transfer the **BIODIESEL** into an appropriate tank. **Make sure no impurities or foreign materials are found** in the ester. If so, **remove the contaminants immediately.**

NOTE:

KOH can be purchased from UI Chemstores. Advance notice of 2 weeks is often necessary. It is cheaper to purchase KOH in bulk of 50 kg (110 lbs).

Ethanol (industrial grade, 200 proof, denatured) can be purchased directly from Thiessen Oil Company in Lewiston. A phone call is enough to notify them for any purchase.



A FARM SCALE RAPESEED OIL AND BIODIESEL PLANT