

BATCH TYPE TRANSESTERIFICATION PROCESS FOR WINTER RAPE OIL

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ABSTRACT

The methyl ester of winter rape (MEWR) has been found to be a potentially useful substitute for diesel fuel. This article discusses the procedure used to produce MEWR for use as a diesel fuel substitute. Reaction variables, rates, equipment and detailed procedures for making 756 L (200 gal) batches of MEWR are discussed. Systems for methanol and glycerol recovery are included. Zero profit economic data are also presented.

KEYWORDS. Alternative fuels, Diesel fuel, Transesterification, Winter rape oil.

INTRODUCTION

The purpose of this article is to report on the process for transesterification of vegetable oil to produce a more acceptable diesel fuel substitute. The use of vegetable oil as a fuel has been under study at Idaho since 1979. While short term tests are almost always positive; longer term tests with neat vegetable oil lead to severe engine deposits, ring sticking, injector coking, and thickening of the lubricating oil.

Transesterification has shown good potential for reducing engine problems associated with vegetable oil fuels. Transesterification is the process of reacting a triglyceride with an alcohol in the presence of a catalyst to produce glycerol and fatty acid esters. The molecular weight of the ester molecule is roughly one-third that of a neat vegetable oil molecule, and the ester has a viscosity approximately twice that of diesel fuel. In contrast, raw vegetable oil has a viscosity of 10 to 20 times that of diesel fuel. Viscosity of the fuel is of prime concern because of its effect on spray patterns and deposit formation. Zhang et al. (1988) have shown that the methyl ester of high erucic acid rapeseed oil (MEWR) performs similarly to diesel in both

short and long term engine tests. However, cloud points and pour points of vegetable oil esters are known to be much higher than diesel fuel and are much more susceptible to problems when used in cold weather.

Fatty acid esters have surprisingly good emissions characteristics. Mittelbach et al. (1985) found that emissions of two different methyl ester fuels derived from rape seed oil gave significantly lower total particulates and lower polynuclear aromatic hydrocarbons than #2 diesel fuel. However, combustion of methyl ester fuels produced higher levels of NO_x emissions and aldehyde emissions than did #2 diesel fuel. Similar results have been reported by Geyer et al. (1984), Mills and Howard (1983), and Feldman (1988). Additional long term engine tests, cold starting tests, and studies of gaseous emissions are needed before vegetable oil fuels can be commercialized. Both engine modification and fuel modification have potential for enhancing engine heat release rates and further reducing exhaust emissions.

TRANSESTERIFICATION

Transesterification of vegetable oils involves reacting an alcohol with the oil in the presence of an alkaline or acidic catalyst as shown in figure 1. With methanol, a methyl ester and glycerol are produced.

The R groups are the fatty acids which are 16 to 22 carbons in length and in this study have zero, one or two double bonds. Fuel properties of vegetable oil are listed in Table 1. The unsaturated fatty acids can polymerize and cause gum formation in the engine, lubricating oil, and on the injector tips.

Hoffman (1989) gave the following description of the transesterification process, "An ester is the condensation product of an acid and an alcohol. Transesterification may be either an acidolysis, where the acid component of an ester is replaced by another acid, or an alcoholysis, where

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This report contains a summary of research results. This is not to be construed as a recommendation for the use of any alternate fuels. Production of the ester involves the use of certain hazardous materials; the competence of the personnel involved and suitability of available equipment must be considered before attempting to reproduce this work in any form.

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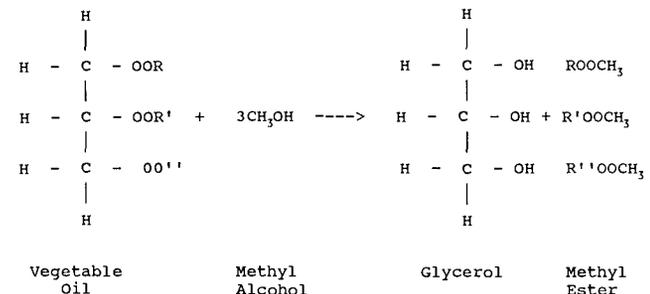


Figure 1—Vegetable oil transesterification.

TABLE 1. Properties of methyl ester compared with #2 diesel and neat rapeseed oil

Test	Rapeseed Methyl Ester	No.2 Diesel	Neat Rapeseed Oil
Cetane rating	54.4	47.8	42.6
Flash point, °C	†	80	274
Cloud point, °C	-2.2	-12.2	-11
Pour point, °C	-9.4	-28.9	—
Viscosity, (cs)			
@ 40° C	6.2	3.2	46.68
@ 100° C	2.4	1.3	—
Sulfur (% wt)	0.031	0.32	0.022
Heat of combustion			
kJ/kg (gross)	40,600	45,300	40,400
Btu/lb (gross)	17,500	19,500	17,370
Specific gravity	0.874	0.852	0.906

* Analysis conducted by Phoenix Chemical Laboratory, Inc., Chicago, IL.

† No flash was observed.

the alcohol component of an ester is replaced with a different alcohol.”

Vegetable oils are esters of fatty acids and glycerol. In this work, the transesterification being studied is an alcoholysis where the glycerine component of a vegetable oil is replaced by an alcohol.

OBJECTIVES

The purpose of this project was to develop a small scale transesterification system which might be used on the farm and in research for the production of MEWR for use as an alternative diesel fuel. Specific objectives were:

- Identification of the process variables which affect the yield and conversion rate of winter rape vegetable oil to ester.
- Identification of the equipment and procedures needed to successfully produce MEWR on a routine basis.

LITERATURE REVIEW

Transesterification of vegetable oils with ethyl alcohol and methyl alcohol to produce ethyl esters and methyl esters has been the topic of several University of Idaho graduate theses including Jo (1984), Madsen (1985), Melville (1987), Mosgrove (1987), and Caringal (1989). In each of these theses, the literature reviews have reported investigations of the transesterification of several oils to produce methyl and ethyl esters; fish oil, soybean, sunflower, cottonseed, peanut, and linseed oils have been transesterified. These transesterification studies have concentrated on optimizing the reaction variables of temperature, agitation time, ratio of alcohol to vegetable oil, and type of reaction rate enhancing agent.

Nye and Southwell (1983) investigated the effects of several important reaction parameters on the methanolysis of rapeseed oil. They reported finding successful conditions at room temperature by systematically optimizing the other operating variables. They identified the main variables as catalyst type, catalyst concentration, oil/alcohol ratio and stirring rate. They found that 1% NaOH or KOH was an effective reaction rate enhancer at room temperature; a 60-min reaction time was allowed. It was determined that a 6:1 molar ratio of methanol to oil gave the best conversions.

They also found that the rate of reaction is satisfactory if the stirring action is vigorous with some splashing.

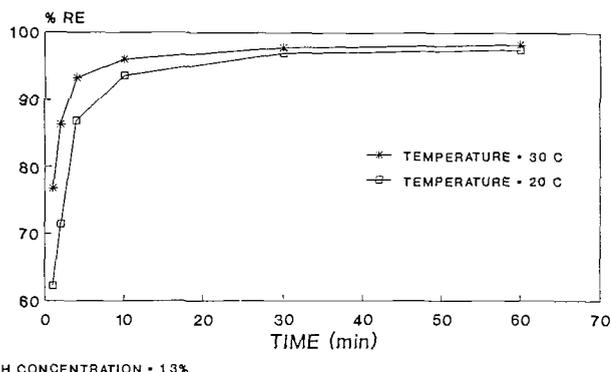
Nye and Southwell (1983) extended their work on transesterification of rapeseed oil to produce the methyl ester in a bench-scale operation. They transesterified 25-L (6.6 gal) batches of oil in an enclosed stainless steel cylindrical drum which was equipped with a 102 mm (4-in.) diameter propeller driven by a 0.25 kW (1/3 hp) electric motor. Rapeseed oil was added to a solution of 1% sodium hydroxide (by weight relative to oil) dissolved in 6 molar equivalents of dry methanol. The solution was stirred with splashing for one h at 24° C. The mixture was allowed to separate into two phases: one phase is rich in ester and unreacted methanol; the other phase is rich in glycerine and unreacted methanol. The ester phase was washed three times with water equal to half the volume of ester to remove methanol and potassium compounds. The ester was then dried over anhydrous calcium chloride and filtered.

University of Idaho workers Jo (1984), Madsen (1985), Melville (1987), and Mosgrove (1987) followed the work of Nye and Southwell (1983). They studied effect of temperature, rate of stirring, KOH concentration, reaction time and methanol-to-oil ratio on reaction rate and degree of conversion. Their results confirmed those of Nye and Southwell (1983). Based on their bench-scale results, a small pilot-plant procedure was developed for production of methyl ester of rapeseed oil. They produced the ester for large-scale laboratory testing and small-scale commercial use. Their system consisted of a 208 L (55 gal) reactor driven by a 0.19 kW (1/4-hp motor) at 510 rpm. The reaction took place at room temperature; a 6:1 molar ratio of methanol to rapeseed oil and 1% KOH by weight of oil were used. KOH pellets [1.46 kg (3.22 lb)] were dissolved in 34 L (9 gal) of anhydrous technical grade methanol by stirring for about 10 min. The reactor was charged with the methanol-KOH solution and 151 L (40 gal) of crude, filtered rapeseed oil. The reaction was allowed to proceed for 4 h. After the stirrer was turned off, the mixture was allowed to settle for 810 h; the mixture separated into an ester-rich layer and a glycerine-rich layer.

Madsen (1985) reported that the percentage conversion from the triglyceride can be determined in several different ways. Gauglitz and Lehman (1963) used thin layer chromatography for checking the conversion of fish oil to ester. More recently Freedman and Pryde (1982) used an Iatroscan Thin Layer Chromatography (TLC) Analyzer with a flame ionization detector which gave more accurate results. Nye and Southwell (1983) used a proton nuclear magnetic resonance (NMR) instrument, using the methyl peak for approximate conversion measurements, and gas chromatography and a flame ionization detector, with an internal standard, for more precise measurements of conversion. Kusy (1982) computed conversion by using the amount of glycerol produced as a measure of conversion. Romano (1982) used viscosity and density differences between the oil and its ester as a conversion measurement. Since the viscosity of the ester is an order of magnitude less than its parent oil a plot of viscosity versus percentage of ester would give a downward sloping curve. Madsen (1985) and Mosgrove (1987) used High Pressure Liquid Chromatography (HPLC) analysis to determine conversion rate. They first determined the retention time of the pure oil

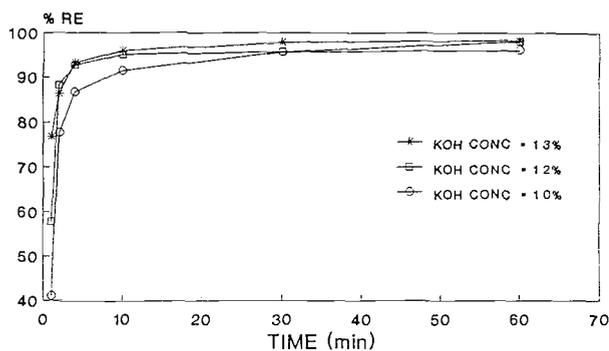
(13.0 min) and then the retention time of pure ester (14.3 min). These were far enough apart to use the separate peak area to calculate the conversion rate.

Madsen (1985) reported reaction rates as shown in figures 2, 3, and 4. Figure 2 is reaction rate for 20° C and 30° C with a concentration of 1.3% KOH. As seen in figure 2, the lower temperatures lowered the initial reaction but by 60 min both were at 98% conversion. Figure 3 is reaction rate for three different KOH levels. Concentrations of 1.0, 1.2, and 1.3% were used. He reported that for the conditions of his study a catalyst concentration of 1.2% is



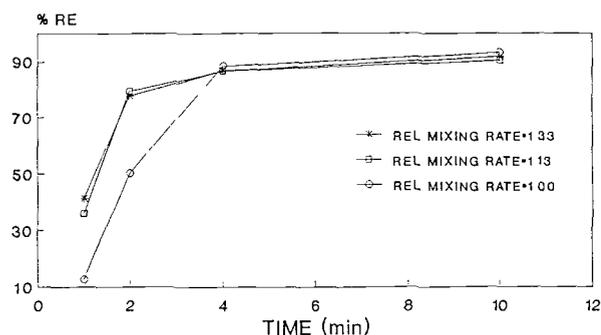
KOH CONCENTRATION • 1.3%

Figure 2—Rate of ester conversion for two temperatures, Madsen (1985).



TEMPERATURE • 30 C

Figure 3—Rate of ester conversion for three catalyst concentrations, Madsen, (1985).



KOH CONCENTRATION • 1.0%
TEMPERATURE • 30 C

Figure 4—Rate of ester conversion for three mixing rates, Madsen (1985).

sufficient and 1.3% excessive. Figure 4 is the reaction versus mixing rate. As the mixing rate is increased the initial rate of reaction increased. However, by 4 min the reaction rates were similar for all mixing rates and approximately the same final yields were obtained.

At this stage of the development, the emphasis was on production of the methyl ester. Consequently, the glycerine-rich phase was discarded; but the ester was purified by a washing process wherein water was allowed to percolate through the ester-rich phase until the methanol and potassium compounds were removed. As explained by Mosgrove (1987), each 151 L (40 gal) batch of MEWR required washing with water at the rate of 238 L/h (63 gal/h) for 8 h. After washing, the MEWR was allowed to stand for 24 h before transferring to a final storage tank.

Caringal (1989) performed considerable analytical work to define the details of the transesterification process. He obtained material balances for each step of the process and accounted for all materials entering and leaving each step of the process. He determined that the potassium leaving the ester phase in the wash water is in the form of a potassium soap. Caringal (1989) also proposed a process for recovering and purifying the by-product glycerine produced during the reaction. A flowsheet showing the transesterification process is shown as figure 5.

MATERIALS AND METHODS

EQUIPMENT

Extracting and Processing. All of the oil used in these studies has been extracted with a small oil extraction plant consisting of a mechanical screwpress manufactured by CeCoCo of Japan. The plant has a capacity of 45 kg/h (100 lb/h) and generates about 19 L (5 gal) of oil per hour. For a complete description of the process see Peterson et al. (1983).

The esterification plant, figures 6 and 7, was designed for a 756 L (200 gal) capacity. Typically, 605 L (160 gal) batches have been produced. The components of the system are: a 1096 L (290 gal), cone bottom, cross-linked polyethylene tank purchased from Pasco Poly-Tank, Inc., Pasco, Washington; an R. S. Corcoran Co., New Lenox, IL,

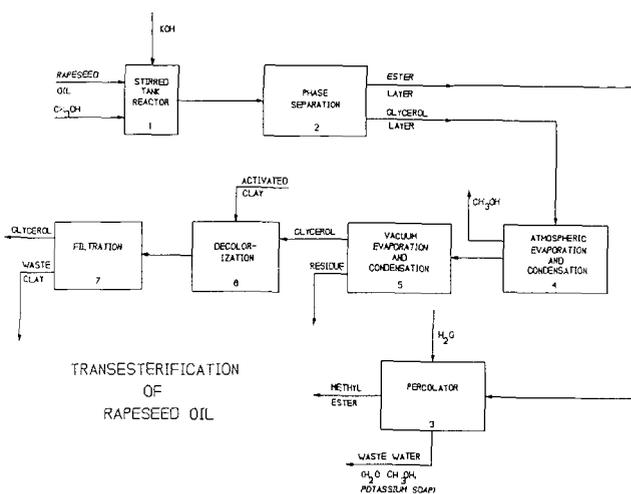


Figure 5—Flow chart for complete transesterification process, Caringal (1989).

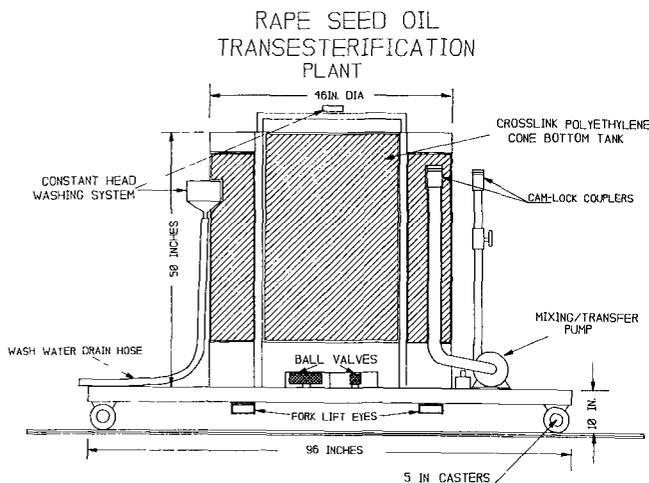


Figure 6—Schematic of the rape oil transesterification plant.

model 2000D Centrifugal pump (75-95 L/min (20-25 gpm) capacity) compatible with methanol, vegetable oil, and KOH solutions, fluid viscosities from 0.65-55.0 centistokes and explosion proof; a 0.2 kW (1/4-hp) lab mixer with 3.4:1 gear reduction (1725 r/min motor speed and 514 r/min rotor speed). The mixer should have an explosion proof motor. A 0.75 kW (1 hp) portable gear pump with totalizing meter is used to transfer the raw vegetable oil and the finished ester to the bulk storage tanks.

The polymer tank and system pump (Corcoran pump) are mounted on a 1.2 m (4-ft) by 2.4 m (8-ft) steel platform with caster wheels and fork lift eyes as shown in figure 6. PVC pipe and fittings are used for all plumbing components. Two hand operated charge pumps are used to prime the centrifugal system pump. A photograph of the system is shown in figure 7. All of the reaction, settling, washing, and separating takes place in this one tank. The methanol and KOH are mixed in a smaller 190 L (50 gal)

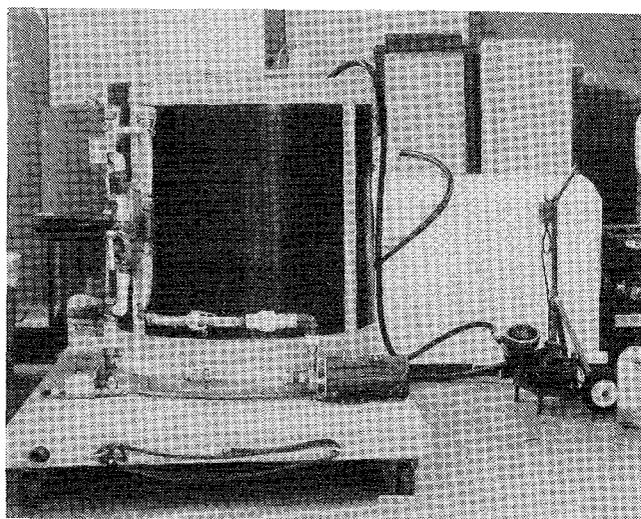


Figure 7—Photograph of the 760 L (200 gal) batch transesterification equipment. The system consists of the cross linked polyethylene tank, portable vegetable oil transfer pump, explosion proof system pump, and storage tanks for the raw vegetable oil and ethyl ester.

tank prior to being transferred to the large tank, as explained below.

INGREDIENTS

151 L (40 gal) batch:

151 L (40 gal) raw, filtered rape oil
34 L (9 gal) methanol
1.46 kg (3.22 lb) KOH

605 L (160 gal) batch:

605 L (160 gal) raw, filtered rape oil
136 L (36 gal) methanol
5.84 kg (12.87 lb) KOH

756 L (200 gal) batch:

756 L (200 gal) raw, filtered rape oil
170 L (45 gal) methanol
7.30 kg (16.10 lb) KOH

PROCEDURE

Phase I – Reaction.

1. Thoroughly clean all parts of the system that will come in contact with the reactants. All parts (tank, pump lines, mixer, etc.) must be clean and dry prior to the start. Before the raw oil is pumped into the tank the entire inside surface of the tank should be wiped down with a clean rag soaked in acetone. Any water in the system will immediately stop the reaction, even though mixing is continued. Dirt will slow the reaction and reduce ester purity.
2. Measurement of component materials must be accurate. The process uses 100% excess methanol (99% purity) to speed the reaction. An error in methanol measurement will affect reaction speed, ester purity and cost. An excess ratio of KOH to rape oil will result in production of an unusable soap, while a deficit ratio of KOH to rape oil will result in an ester of reduced purity. In our plant the mixing tank has been calibrated for measurement of the rape oil while a plastic barrel has been calibrated for measurement of the methanol.
3. Pump the clean raw, filtered rapeseed oil into the tank using the portable pump. If the pump has been used for another purpose, flush it with about a gallon of rape oil before transferring the rape oil to the mixing tank.
4. Pump the methanol into the 190 L (50 gal) tank using the system pump. Again some methanol should be used to flush pump and lines of contaminants and moisture.
5. Add the correct amount of KOH to the methanol, immediately cover and mix using the lab mixer until the KOH is completely dissolved (5 min is usually sufficient.) The barrel cover is designed with a slot for the mixer shaft. Provide good ventilation and use a ground strap to avoid sparks when pumping the methanol and during the first 30 min of mixing the vegetable oil/alcohol mixture.
6. Remove the lab mixer from the methanol and place into the ester mixing tank. Place the center of the shaft into the oil with the prop near the center of the tank.
7. With the mixer running, pump the methanol-KOH solution into the ester mixing tank using the system pump. The reaction will begin immediately and

should be completed within 4-6 h. The system pump is used intermittently during the reaction with the inlet tube near the tank bottom and the outlet near the top to aid the mixing process.

8. The order of the procedure is important. Having the rape oil pre-measured in the ester mixing tank minimizes the amount of time that methanol is evaporating (producing an explosion hazard) and eliminates the possibility of creating an excess ratio of KOH to rape oil during mixing. Likewise, complete dissolution of the KOH in the methanol prior to adding to the rape oil is important to prevent soap formation.
9. The reaction produces two liquids, methyl ester and its by-product, glycerol. After 4 to 6 h the mixer is removed from the tank. The system pump lines are removed. The tank is covered, and the mixture is allowed to settle for 12 h or more to allow the glycerol to settle to the bottom.
10. The glycerol is drained from the bottom. In our tank, PVC hand valves are provided to make the process relatively simple. Approximately 132 L (35 gal) of glycerol will be drained from the tank from a 605-L (160-gal) batch. This is approximately equal to the amount of methanol used, thus the amount of ester produced is about the same as the amount of vegetable oil used in the batch. A distinct color change from dark brown to yellow will occur when all the glycerol has drained off.

Phase II – Washing.

1. Enough cold water is added to raise the level of ester in the tank 200 to 250 mm (8 to 10 in.). This creates a “cushion” of water between the ester and the drain. The outlet constant head drain tube (a tube connected to the outlet of the tank) is placed at the level of the ester. Water is sprinkled into the tank from a common lawn sprinkler at the approximate rate of 375 L/h (100 g/h.) As the water droplets travel through the ester they remove the KOH, methanol, and other impurities. Some impurities will pass through the system with the water and some will remain at the interface of the ester and water cushion. The drain water will start out milky and will gradually become more clear as the process proceeds. The sprinkler is set so that water droplets are distributed evenly over the entire surface area of the ester.
2. Wash for 20 to 30 h, occasionally adjusting water pressure for more complete coverage.
3. Once finished the MEWR will have a milky yellow color. After washing, let the mixture sit for 3 to 4 days. The water will settle to the bottom leaving a crystal clear layer of MEWR. At this time, the interface between the MEWR and water should be visible as a white layer.
4. Pump the clean MEWR off into the storage tank, being careful not to get too close to the water-MEWR interface with the pump inlet tube.
5. Drain the water out the outlet tube. The remaining 5 to 10 gal of MEWR, that could not be pumped off

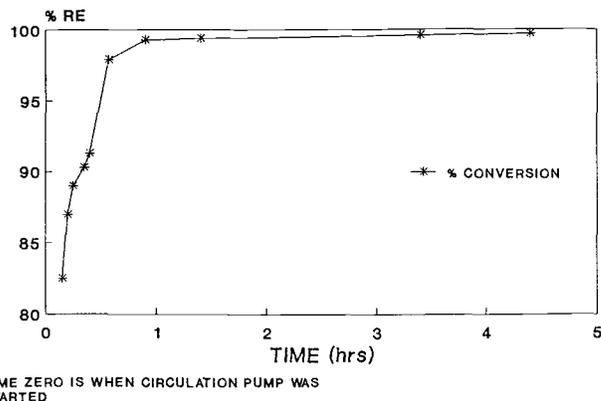


Figure 8—Methyl ester concentration vs. time measured during one of the 605-L (160-gal) batches using the transesterification plant at room temperature.

the top, can be caught in smaller containers, allowed to settle and recovered.

6. Wash the entire unit using a pressure washer with soap and rinse in hot water. Let dry, then cover with a tarp to keep clean.

PRODUCTION

The equipment and procedures described above have been used to make seven, 605-L (160-gal) batches of MEWR for a total of 4100 L (1100 gal). Additional quantities have been produced in smaller containers during the development of the procedures. Monitoring of the reaction rate in several of the batches produced results similar to that shown in figure 8. Nearly complete conversion was reached in approximately one h at room temperature. The ester thus produced has been used in the “Vegetable Oil for Diesel Fuel” project. The methyl ester is used as a replacement for diesel fuel in laboratory studies and in tractors used for field evaluation of problems associated with using the ester under actual field conditions. A 20 kW (27 hp) Satoh tractor has been powered with 100% MEWR since the spring of 1987. A second tractor, a John Deere 3150 of 75 kW (100 hp) size has been powered with a mixture of 50% methyl ester and 50% diesel for 2 years. Both tractors are performing satisfactorily to date.

METHANOL RECOVERY

Caringal (1989) reports that the methanol in the glycerol phase must be removed in order for the glycerol to be of commercial importance. He found that the glycerol has a methanol content of 30% by weight. This corresponded to almost 50% of the excess methanol. The simplest method of recovery is to vaporize the methanol. He found that distillate collected at 68 to 120° C contained 99% by weight methanol.

GLYCEROL

Caringal (1989) proposed two schemes for glycerol recovery. His process B, which was vacuum evaporation, was recommended. It had an 86.29% glycerol recovery and produced 98.2% glycerol. Caringal (1989) reported a price of \$0.336/kg (\$0.74/lb) for glycerol.

ECONOMICS

Two University of Idaho graduate students, Melville (1987) and Caringal (1989) have made zero profit analysis of the cost of MEWR.

Melville (1987) estimated the production cost of the ester as \$0.31/L (\$1.18/gallon) and \$0.425/L (\$1.61/gal) for a Co-op sized unit and Farm-sized unit, respectively. The farm-size unit processed 78.6 t (86.7 tons) of seed per year producing nearly 34,000 L (9000 gal) of ester/year (enough to meet the diesel demand of an average sized farm.) The Co-op sized unit processed 1480 t (1,632 tons) of seed per year producing 655,000 L (173,000 gal) of ester per year (enough to supply approximately 20 average sized farms). Melville (1987) assumed prices of glycerol at \$0.93/kg (\$0.42/lb), methanol at \$0.159/L (\$0.60/gallon), and meal at \$110.23/t (\$100.00/ton.)

Caringal estimated MEWR production costs at \$0.346/L (\$1.31/gallon) by considering raw rape oil at \$0.44/kg (\$0.20/lb), methanol at \$0.158/L (\$0.60/gal), and KOH at \$1.00/kg (\$0.4535/lb). He based his calculations on a plant processing 10 metric tons of rape oil per day.

Alcohol has been made successful as an additive to gasoline by certain tax breaks. In some cases, these breaks amount to approximately \$0.0264/L (\$0.10 for each gallon) of fuel produced containing at least 10% alcohol. Since each 3.78 L (1 gal) of alcohol can be blended to produce 37.8 L (10 gal) of fuel, the subsidy is essentially \$0.264/L (\$1.00/gallon.) A subsidy of this magnitude would make the MEWR an economically attractive fuel. Subsidies for on-farm use could take the form of such things as allowing winter rape, as a total energy crop, to be grown on set aside or CRP land. If the crop were grown solely for energy, there would be no impact on food or feed markets and farmers would be able to meet some energy needs from their own productive capacity.

CONCLUSIONS

The process described in this paper has been used successfully to produce the MEWR oil. Conversion has been 98% and above. All of the problems encountered have been mechanical in nature (leaks, failure to prime the pump, etc.) Handling large quantities of methanol constitutes an explosion and fire hazard and is the biggest major element of concern in implementing the process. The relatively large quantities of water used constitute a disposal problem. Methods to recover the methanol and purification of the glycerol need scale-up and implementation to improve the economics of the system.

Seven, 605 L (160 gal) batches of MEWR oil have been successfully produced at the University of Idaho using the equipment and methods described in this paper. Additional quantities have been produced in smaller containers during the development of the procedures. The ester thus produced has been used in the "Vegetable Oil for Diesel Fuel" project. The methyl ester is used as a replacement for diesel fuel in laboratory studies and in tractors used for field evaluation of problems associated with using the ester under actual field conditions.

No attempt has been made to define the process for any other purpose than for fuel use. Transesterification is used only as a method to lower the viscosity of the vegetable oil to a more suitable level for use as a fuel.

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