BIODIESEL PRODUCTION VIA ACID CATALYSIS

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ABSTRACT. *Vegetable oils and animal fats can be transesterified to biodiesel for use as an alternative diesel fuel. Conversion of low cost feedstocks such as used frying oils is complicated if the oils contain large amounts of free fatty acids that will form soaps with alkaline catalysts. The soaps can prevent separation of the biodiesel from the glycerin fraction. Alternative processes are available that use an acid catalyst. The objective of this study was to investigate the effect of process variables on acid-catalyzed transesterification. The molar ratio of alcohol, reaction temperature, catalyst amount, reaction time, water content, and free fatty acids were investigated to determine the best strategy for producing biodiesel. Food grade soybean oil was used to prepare esters using excess methanol and sulfuric acid as a catalyst. To compare the effect of different alcohol types on ester formation, methanol, ethanol, 2-propanol, and n-butanol were compared. The American Oil Chemists' Society Method Ca 14-56 was used to measure the biodiesel's total glycerin amount as an indicator of the completeness of the reaction. It was found that acid catalysis can provide high conversion rates but much longer times are required than for alkaline catalysts. The acid catalyst also requires the concentration of water to be less than 0.5%, which is about the same as is required for alkaline catalysts. Water formed by the esterification of free fatty acids limited their presence in the oil to 5%.*
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Many researchers have concluded that vegetable oils hold promise as alternative fuels for diesel engines (Scholl and Sorenson, 1993; Wagner et al., 1984). However, using raw vegetable oils for diesel engines can cause numerous engine-related problems (Korus et al., 1982). The increased viscosity, low volatility, and poor cold flow properties of vegetable oils lead to severe engine deposits, injector coking, and piston ring sticking (Perkins et al., 1991; Pestes and Stanislao, 1984). These effects can be reduced or eliminated through transesterification of the vegetable oil to form a methyl ester (Perkins et al., 1991; Zhang et al., 1988). The process of transesterification removes glycerol from the triglycerides and replaces it with radicals from the alcohol used for the conversion process (Kusy, 1982). This process decreases the fuel's viscosity but maintains the cetane number and heating value. The monoesters produced by transesterifying vegetable oils with alcohol are known as *biodiesel*.

Most of the biodiesel that is currently made uses high quality soybean oil and an alkaline catalyst. However, there are large amounts of waste oils (restaurant waste, frying oils, trap grease, etc.) that could be converted to biodiesel. The problem with processing waste oils is that they often contain significant amounts of free fatty acids that cannot be converted to biodiesel using an alkaline catalyst. These free fatty acids react with an alkaline catalyst to produce soaps that inhibit the separation of the ester, glycerin, and wash water. An alternative way of processing these waste oils, that may have a greater tolerance for free fatty acids, is to use an acid catalyst. Nye et al. (1983) have shown that it is possible to produce methyl esters from used frying oil using an acid catalyst. The objective of this study was to investigate the effect of the molar ratio of alcohol, the reaction temperature, the catalyst amount, the reaction time, and the presence of water and free fatty acids on the completeness of acid-catalyzed transesterification.

Transesterification is the chemical process of converting one ester, in this case the glycerol esters that constitute vegetable oils, into another ester such as the alkyl monoesters that compose biodiesel. This process occurs by reacting the vegetable oil with alcohol in the presence of an alkaline or acidic catalyst. Alcohols such as ethanol, methanol, or butanol can be used in the transesterification and these alcohol esters are named methyl esters, ethyl esters, or butyl esters, respectively (Formo, 1954). The most commonly preferred catalysts are sulfuric, sulphonic, and hydrochloric acids as acid catalysts and sodium hydroxide, sodium methoxide, and potassium hydroxide as alkaline catalysts (Sridharan and Mathai, 1974).

There are several comprehensive studies of base catalyzed transesterification. Freedman and Pryde (1982) and Kusy (1982) report greater than 95% fatty ester yields in 1 h using alkaline catalysts such as sodium hydroxide, sodium methoxide, and sodium ethoxide with alcohols such as methanol, ethanol, and 1-butanol. Freedman et al. (1984) found that to maximize ester yield, the acid value should be less than 1 mg KOH/g, and the alcohol and vegetable oil should be moisture free. Nye and Southwell (1984) have reported the effects of several catalysts on

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methyl and ethyl esters of rapeseed oil at room temperature conditions. They concluded that only alkoxides and hydroxides were effective at room temperature. Boocock et al. (1996) developed a new method which formed methyl ester much faster. They found that when they added tetrahydrofuran to the solution of catalyst and methyl alcohol, the reaction occurred much more rapidly. In that research, by using 1.3% sodium hydroxide as catalyst, they produced 95% methyl ester from soybean oil in 15 min.

Schwab et al. (1987) and Liu (1994) compared acid and base catalysts. They noted that acid catalyzed transesterification requires heating to accelerate the reaction and that the reaction time may vary from a few minutes to several hours. Nye et al. (1983) compared base and acid catalysts in the process needed to produce methyl esters from used frying oil. The highest ester productions at 25°C were found to be 1-butyl ester with an acid catalyst which gave 81% conversion, methyl ester with a base catalyst which gave 74% conversion, and ethyl ester with an acid catalyst which gave 72% conversion. They noted that the methyl ester (in base catalysis) and 1-butyl ester (in acid catalysis) reacted more completely than the other five esters. In the literature (Freedman et al., 1984; Liu, 1994), it has been stressed that the acid catalyst will be much more effective than the alkaline catalyst if the vegetable oil has more than 1% free fatty acid. This research (Schwab et al., 1987; Liu, 1994; Freedman et al., 1986) has also stressed that the transesterification reaction by acid catalysis was much slower than by base catalysis.

The residue of either catalyst in the ester can cause engine problems. Base catalysts can produce higher levels of incombustible ash and acid catalysts attack the engine metallic parts. Therefore, the catalyst must always be eliminated from the fuel when the reaction is complete (Romano, 1982).

EXPERIMENTAL METHODS

TEST SYSTEM SETUP

To understand the effects of molar ratio, reaction temperature, reaction time, catalyst amount, water, and free fatty acids on the yield of acid-catalyzed methyl ester, a laboratory-scale reactor consisting of a 1000 cc glass flask was used. The flask was heated and stirred by a combination hot plate and magnetic stirrer. A water-cooled condenser was mounted on the top of the flask to capture and return any vaporized alcohol. Alcohol loss during the reaction was monitored and always found to be less than 5% of the alcohol added. Unless otherwise indicated, the reaction temperature was 60°C, the alcohol used was methanol, and the reaction time was 48 h. The mixture was stirred at the same rate for all runs.

PREPARATION OF METHYL ESTER WITH
SULFURIC ACID CATALYST

To prepare acid-catalyzed biodiesel fuel, soybean oil was purchased from a local food store and the methyl esters were prepared under laboratory conditions. The general procedure for preparing acid-catalyzed biodiesel was as follows. Solutions of sulfuric acid in methyl alcohol were prepared at room temperature. The sulfuric acid percentage was based on the weight of soybean oil to be reacted. The solution was added to the soybean oil at the

required temperature (60°C, 45°C, and 25°C) while stirring. The mixture was gently stirred for 48 (or 96) h at atmospheric pressure and the required temperature. A reflux condenser returned vaporized alcohol to the reaction vessel. After the reaction period was complete, the ester and glycerol layers were separated using a separatory funnel and the methyl esters were washed four times with distilled water to remove soap and residual alcohol. Previous studies (Van Gerpen et al., 1997) have shown that after being washed four times, there is no further benefit. As a final step, a small amount of pure glycerin was added to the ester and stirred for 15 min. This glycerin was removed by gravity separation. We have found that this step helps to remove mono- and diglycerides. The completeness of the reaction was measured by the American Oil Chemists' Society (A.O.C.S.) Official Method Ca 14-56 known as the Total, Free, and Combined Glycerol (Iodometric-Periodic Acid) Method. This method determines the total glycerol by saponifying the unreacted oil and then reacting the glycerol produced with periodic acid. The resulting compound is titrated with normalized sodium thiosulfate solution using a starch indicator solution. The extent of the reaction was calculated by computing the amount of glycerin in the original oil and subtracting the total glycerin measured in the washed ester using method Ca 14-56. This difference was stated as a percentage of the original total glycerin amount.

Because of the large number of variables that affect the transesterification reaction, it was not possible to conduct an exhaustive series of tests based on all the possible combinations of the variables. Rather, a standard condition was established and the effect of each variable was investigated as a deviation from that condition. The standard condition consisted of 60°C reaction temperature, 3% sulfuric acid catalyst, 6:1 molar ratio of methanol to oil, and a reaction time of 48 h. Each experiment was conducted twice and the results were averaged. In all cases, the two results were within 5%.

RESULTS AND DISCUSSION

The effect of the reaction variables on the completeness of the transesterification reaction was determined by direct measurements under laboratory conditions. The effect of each variable is described below.

THE EFFECT OF MOLAR RATIO

The molar ratio of alcohol to vegetable oil is one of the most important factors that influences the conversion of vegetable oil to its ester. For the stoichiometric reaction, 3 moles of alcohol (96 g of methanol) are required per mole of oil (885 g of soybean oil). In reality, the molar ratio should be higher than the theoretical ratio to drive the reaction to completion. Formo (1954) advocates the use of a large excess of methanol (15 to 35 moles per mole of fatty acid) when using sulfuric acid as a catalyst. He noted that after refluxing for several hours ester yields of 95% or higher can be obtained. Freedman and Pryde (1982) found that a 30:1 molar ratio of methanol to soybean oil with 1% sulfuric acid gave good conversion after 44 h of heating at 60°C. The actual conversion percentage was not stated. They emphasized that if vegetable oil has more than 1% free fatty acid, the acid catalyst will be much more
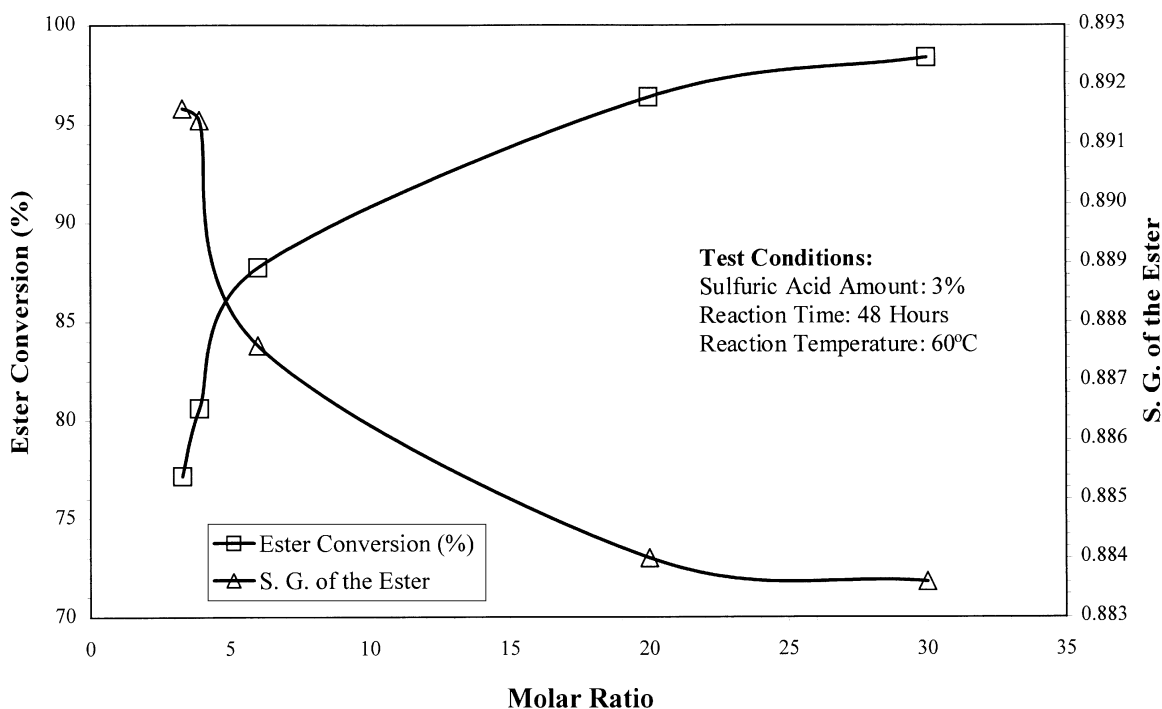
Figure 1–Effect of molar ratio on ester conversion and specific gravity.

effective than the alkali catalyst. Freedman et al. (1984) compared molar ratios of 6:1, 20:1, and 30:1 for acid catalysis with methanol. The molar ratios of 6:1 and 20:1 gave unsatisfactory ester conversion after 18 h of reaction time. The highest conversion with acid catalyst was measured at the 30:1 molar ratio.

In this study, to investigate the effect of molar ratio, five different molar ratios, 3.3:1, 3.9:1, 6:1, 20:1, and 30:1, were selected. Each reaction was run for 48 h with 3% sulfuric acid catalyst at 60°C. The changes in conversion and the specific gravity of the methyl ester are shown in figure 1. Ester conversions reached 98.4% at the 30:1 molar ratio. The specific gravity of the ester decreased with increasing molar ratio. This was probably due to a decrease in residual triglycerides.

THE EFFECT OF REACTION TEMPERATURE

Another important factor that affects the conversion of vegetable oil to its ester is the reaction temperature. Liu (1994) noted that heating is required for faster reaction and the reaction time may vary from a few minutes to several hours for acid catalysts.

For this experiment, three different reaction temperatures, 25°C, 45°C, and 60°C, were selected. The highest reaction temperature, 60°C, was selected because it is near the boiling point of methanol (65°C). Each reaction was run for 48 h with 3% sulfuric acid catalyst and a 6:1 molar ratio of methanol to oil. The changes in ester formation and the specific gravity of the methyl ester are shown in figure 2. As can be seen, the ester conversion increased almost linearly with increasing reaction temperature. Ester conversions were 8.3%, 57.2%, and 87.8% at 25°C, 45°C, and 60°C, respectively. The specific gravity of the ester decreased with increasing reaction temperature. The maximum specific gravity of the methyl

ester was 0.917, which is close to the specific gravity of soybean oil.

THE EFFECT OF CATALYST AMOUNT

The catalyst amount also affects the conversion of vegetable oil to its ester, at least for the molar ratios of alcohol to oil used in this study. Other researchers have varied the catalyst amount from 0.05 to 1% with alkaline catalyzed transesterification reactions (Schwab et al., 1987; Muniyappa et al., 1996). Muniyappa et al. (1996) found little effect of catalyst amount for alkali catalyzed transesterification at high (30:1) molar ratios of methanol to oil. In acid-catalyzed transesterification, the amount of sulfuric acid is typically varied from 1% to 3% (Formo, 1954; Freedman et al., 1984).

Three different catalyst amounts, 1%, 3%, and 5% of sulfuric acid, were selected for our study. These percentages were weight fractions of the oil supplied for the reaction. For each case, the reaction continued for 48 h at 60°C and with a 6:1 molar ratio of methanol to oil. The effect of the catalyst amount on the ester conversion and the specific gravity of the methyl ester are shown in figure 3. Ester formation increased from 72.7% to 95.0% as the catalyst was increased from 1% to 5%.

EFFECT OF REACTION TIME

In the study of Freedman et al. (1984), the transesterification was catalyzed with sulfuric acid at 6:1, 20:1, and 30:1 molar ratios of methanol to oil. The molar ratios of 6:1 and 20:1 gave unsatisfactory ester conversion even after 18 h of reaction time. Although conversion percentages are not stated they found that 69 h were required for satisfactory conversions of methyl alcohol at a 30:1 molar ratio.
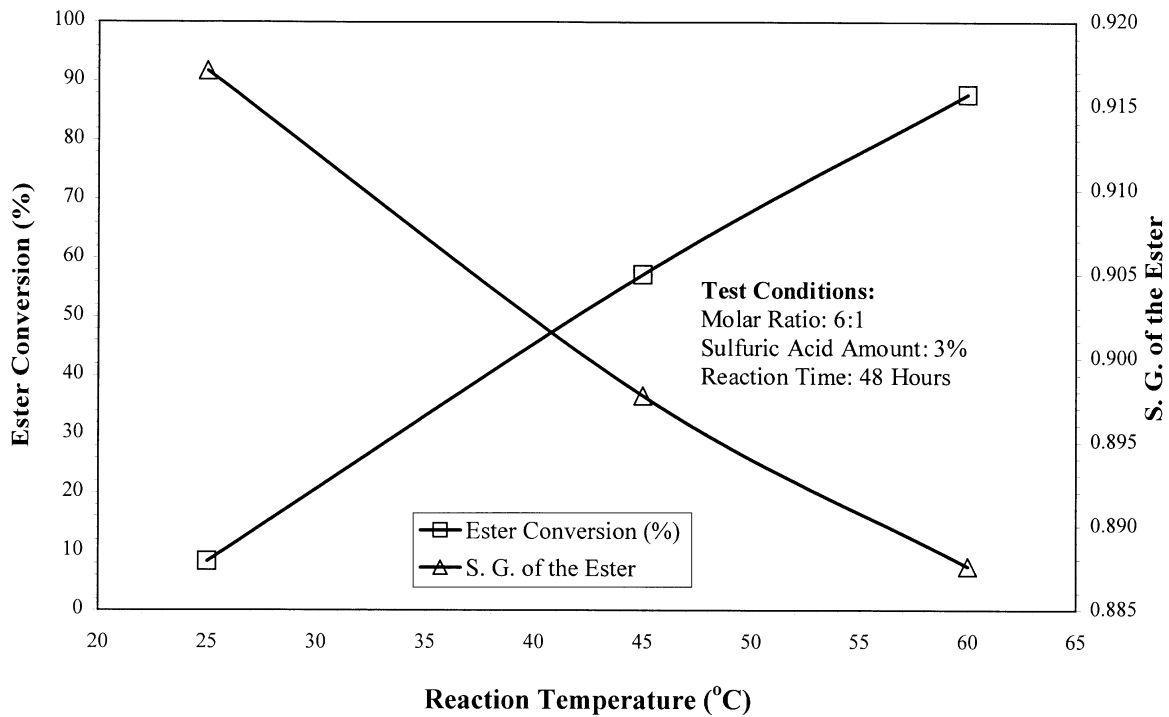
Figure 2–Effect of reaction temperature on ester conversion and specific gravity.
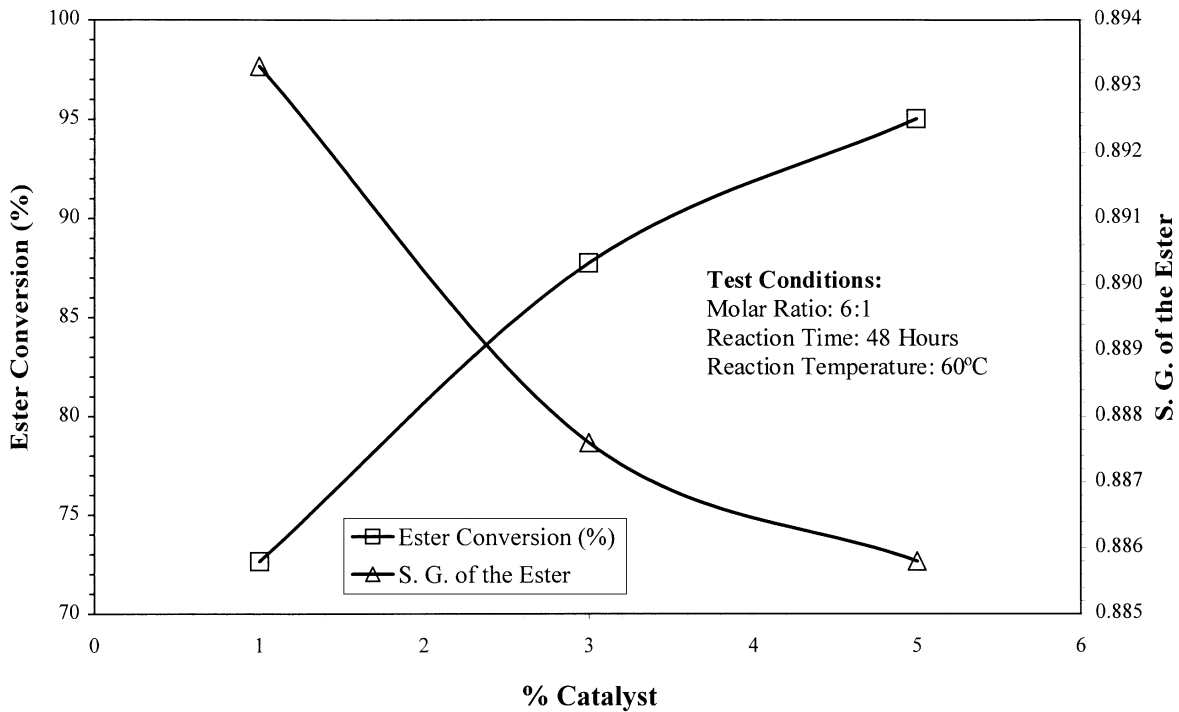

Figure 3–Effect of catalyst amount on ester conversion and specific gravity.

Two different reaction times, 48 and 96 h, were selected for our experiment. For each case, the reaction was conducted with 3% sulfuric acid catalyst, 6:1 molar ratio, and at 60°C. The effect of reaction time on the ester conversion is shown in table 1. Ester conversion increased from 87.8% to 95.1% when the reaction time was increased from 48 h to 96 h. It is clear that long times are required for high conversion rates using an acid catalyst. Although 48 h

was used as the standard test duration for this study, longer times would have given higher conversions.

EFFECT OF ALCOHOL TYPE

Freedman and Pryde (1982) investigated the effects of alcohol type with acid catalysts. They mentioned that a 30:1 molar ratio of methanol to soybean oil with 1% sulfuric acid gave good conversion after 44 h of heating at

**Table 1. Effect of reaction time on ester conversion
and specific gravity of ester**

Run Time (h)	Ester Conversion (%)	Ester S.G.
48	87.8	0.8876
96	95.1	0.8838

Test conditions:
   Molar ratio: 6:1; Sulfuric acid amount: 3%; reaction temperature: 60°C.

**Table 2. Effect of alcohol type on ester conversion
and specific gravity of ester**

Alcohol Type	Boiling Temperature (°C)	Reaction Temperature (°C)	Ester Conversion (%)	Ester S.G.
Methanol	65	60	87.8	0.8876
2-Propanol	82.4	75	92.9	0.8786
1-Butanol	117	110	92.1	0.8782
Ethanol	78.5	75	95.8	0.8814

Test conditions:
   Molar ratio: 6:1; Sulfuric acid amount: 3%; Reaction time: 48 h.

60°C. Butyl alcohol with 1% sulfuric acid converted the oil to 98% ester after only 4 h of heating at 114°C. The higher boiling temperatures of the longer chain alcohols allow higher reaction temperatures to be used. In a separate study, Freedman et al. (1984) studied three different alcohols at 30:1 molar ratios of alcohol to soybean oil with 1% acid catalyst at temperatures just slightly below the boiling points of the alcohols. They noted that they achieved maximum ester conversions after 3, 22, and 69 h for butyl, ethyl, and methyl alcohol, respectively.

Gauglitz and Lehman (1963) measured the rate of transesterification for a number of simple alcohols up to n-hexanol. While they used sodium methoxide as a catalyst, their results may be relevant to acid catalyzed reactions. They found that longer chain alcohols have longer reaction times when the same reaction temperature is used. The addition of one methylene group (CH_2) to the alcohol doubles the reaction time and the addition of a branched chain is equivalent to an increase of two methylene groups.

To investigate the effect of different alcohol types on transesterification, we tested 2-propanol, 1-butanol, ethanol, and methanol for a 48-h test period, with 3% sulfuric acid catalyst, and a 6:1 molar ratio of alcohol to oil. The reaction temperatures were selected to be a few degrees below the boiling points of the alcohols and are shown in table 2. The relationship between alcohol type and ester conversion is also shown in table 2. The highest ester conversion occurred with ethanol. The conversion to ethyl ester was 95.8% compared with 92.9%, 92.1%, and 87.8% for 2-propyl ester, 1-butyl ester, and methyl ester, respectively. The higher conversion rates found for the longer chain alcohols compared with methyl ester are probably due to the higher reaction temperatures allowed by their higher boiling points. This effect apparently dominates any decrease in reaction rate associated with the longer chain alcohols.

EFFECT OF WATER

In the transesterification of vegetable oil with alkaline catalysts, other researchers (Schwab et al., 1987; Romano, 1982) have emphasized that the vegetable oil and alcohol should be water free. Romano (1982) investigated the

effect of water in soybean oil on transesterification with ethanol and 2% sodium hydroxide catalyst. When the water amount was changed from 0.15% to 0.66% in the reaction, glycerine yields decreased from 95.4% to 73.2%.

To investigate the effect of water on the transesterification, different amounts of distilled water were added to the vegetable oil. The test duration was 96 h with 3% acid catalyst, 60°C, and 6:1 molar ratio. The effect of water percentage in the oil on the ester conversion and specific gravity of the methyl ester is shown in figure 4. As little as 0.1% water addition (based on the weight of the vegetable oil) reduced the ester yield. When more water was added to the vegetable oil, the amount of methyl esters formed was significantly reduced. The addition of 5% water reduced the ester conversion to only 5.6% while the ester conversion without water was 95.1% at the same reaction conditions. Our results indicate that more than 0.5% water in the oil will decrease the ester conversion to below 90%.

EFFECT OF FREE FATTY ACIDS

To investigate the effect of free fatty acid level on ester conversion, different amounts of palmitic acid (Aldrich Chemical Co., 99%) were added to soybean oil to obtain 5%, 10%, 15%, 20%, and 33% free fatty acid levels. Palmitic acid was chosen as a representative for the free fatty acids because it is easily obtained and relatively inexpensive. It also is one of the major fatty acids found in the processed grease from dead stock rendering plants, one of the feedstocks under consideration in the current study. The test conditions included a 96-h test period, 3% sulfuric acid catalyst, 60°C, and 6:1 molar ratio. The relationship between the palmitic acid in the oil and the ester conversion and specific gravity of methyl ester is shown in figure 5. The conversion rate of soybean oil to methyl ester drops below 90% for free fatty acid levels above 5%. An attempt was made to compare the tolerance of an alkaline catalyst (potassium hydroxide) to free fatty acids, but the addition of 5% palmitic acid produced a solid soap mixture that prevented separation of the glycerin from the methyl ester.

When the palmitic acid is added to the oil, water is produced by the reaction of palmitic acid and methanol. As was seen in the previous section, water strongly inhibits the ester-formation reaction. For each case of palmitic acid addition, the water amount produced by palmitic acid was calculated, and compared with the water addition test. The results are shown in figure 6. The test results show that palmitic acid addition and water addition tests have similar effect on esterification. For comparison, the effect of water on an alkaline (KOH) catalyst is also shown. The results show that the esterification with alkaline catalyst is more tolerant of water.

CONCLUSIONS

The objective of this project was to investigate the acid-catalyzed methyl ester production process. The variables affecting ester formation including molar ratio, reaction temperature, catalyst amount, and reaction time were investigated to determine the best strategy for producing biodiesel. The American Oil Chemists' Society (AOCS) Official Method Ca 14-56 test procedure was used to
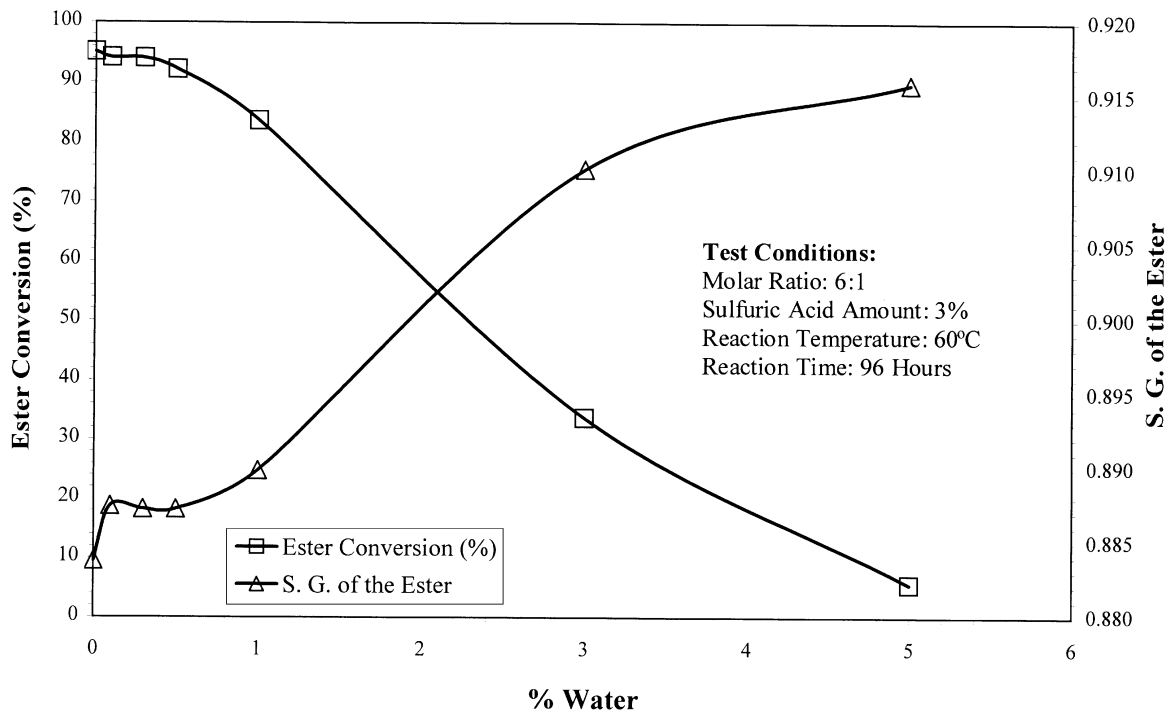
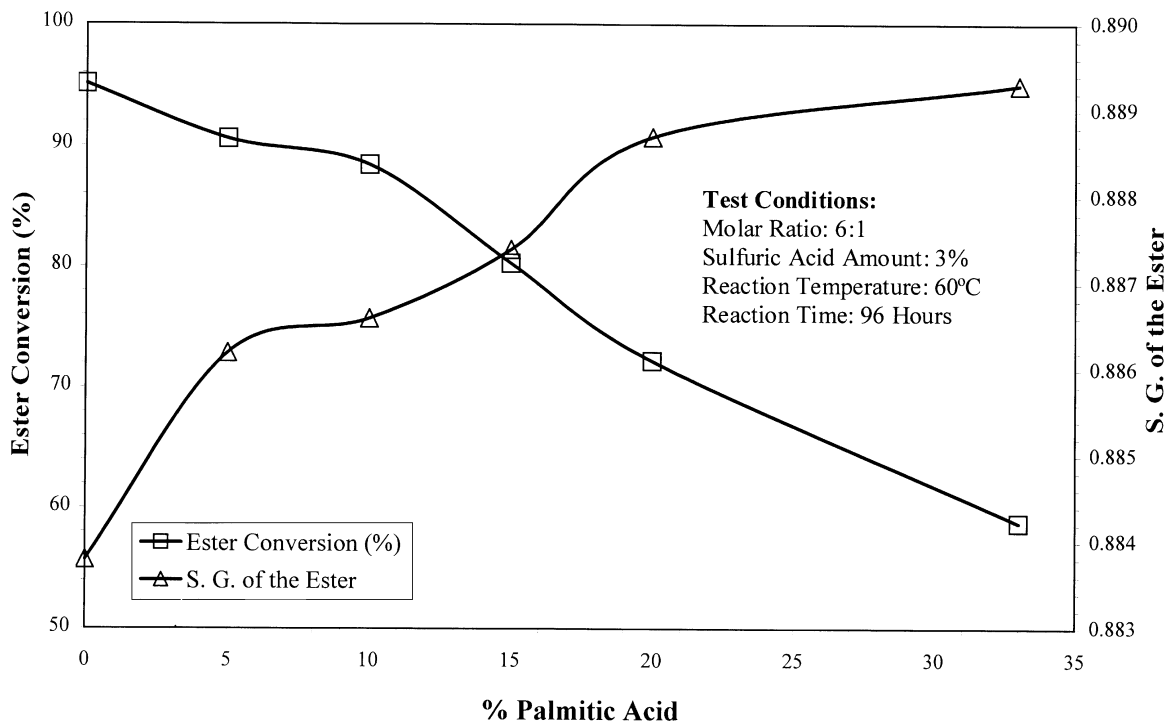Figure 4–Effect of water on ester conversion and specific gravity.

Figure 5–Effect of palmitic acid on ester conversion and specific gravity.

measure the methyl ester's total glycerin amount as an indicator of the completeness of the reaction. The specific gravity of the methyl ester at 60°F was also measured and recorded after each test. The following conclusions can be drawn from the acid-catalyzed biodiesel production study.

1. Acid-catalyzed transesterification is much slower than alkali-catalyzed.

2. The ester conversion efficiency is strongly affected by the molar ratio of alcohol to oil. In acid-catalyzed esterification, a higher molar ratio is required than that of alkali-catalyzed.
3. If the acid catalyzed reaction occurs at room temperature, the reaction is very slow and poor ester conversion is obtained.
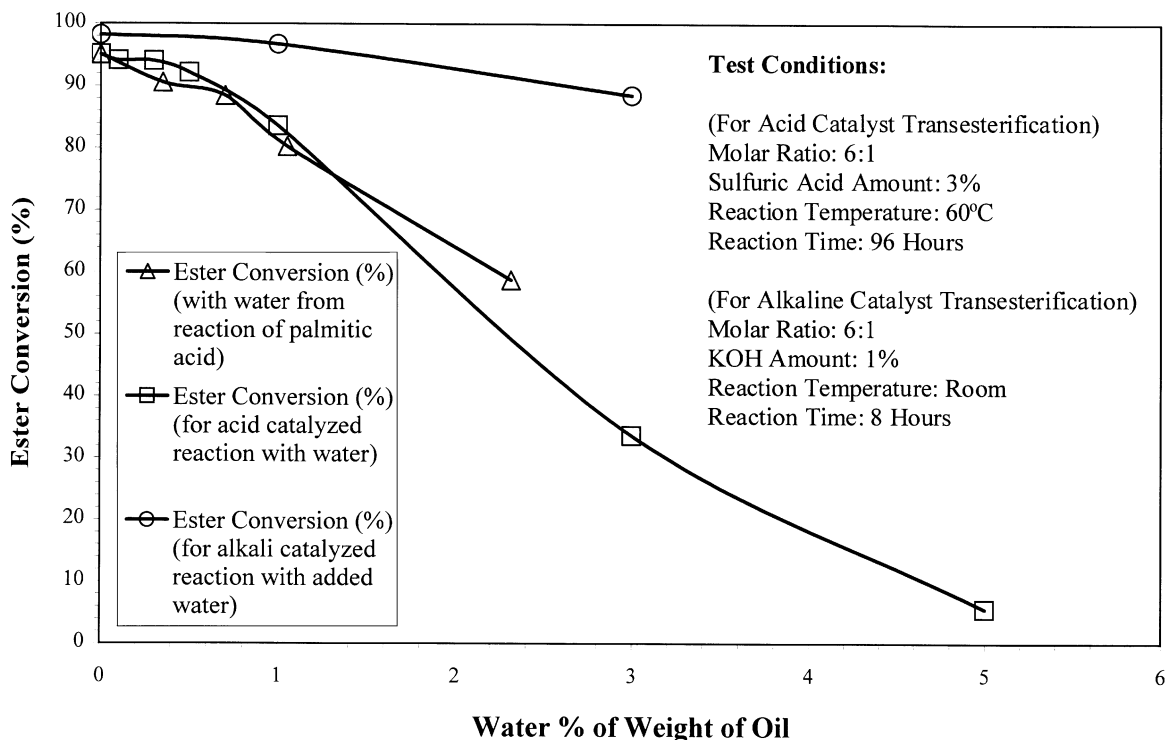
Figure 6–Comparison of the free fatty acid and water addition tests.

4. The completeness of ester formation increases with increasing acid catalyst amount.
5. The ester conversion is strongly inhibited by the presence of water in the oil. If the water concentration is greater than 0.5%, the ester conversion rate may drop below 90%.
6. Alcohols with high boiling temperature increase ester conversion. The higher reaction temperatures allowed by longer chain alcohols apparently dominate any tendency toward reduced reaction rates for these alcohols.
7. The amount of free fatty acid in vegetable oil can have a significant effect on the transesterification reaction. The water formed by the esterification inhibits further reaction. Free fatty acid levels above 5% can lower the ester conversion rate below 90%.

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