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A Continuous Process for the Conversion of Vegetable Oils into Biodiesel

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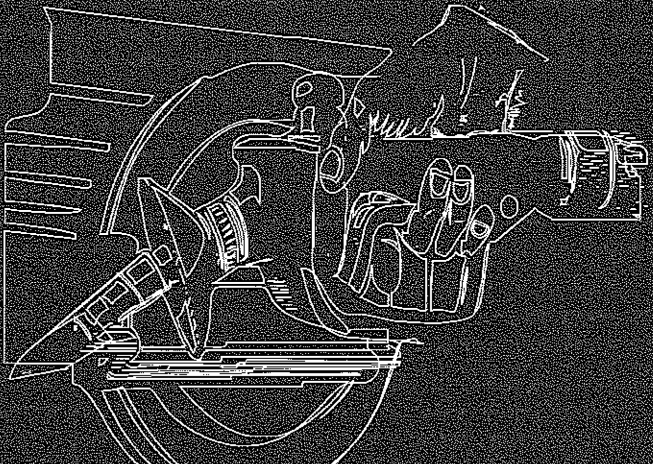

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September 15-17, 1996
Nashville, Tennessee

A Continuous Process for the Conversion of Vegetable Oils into Biodiesel

H. Noureddini, D. Harkey and V. Medikonduru

ABSTRACT

Over the last few years fatty acid methyl esters have assumed importance as research has intensified on the utilization of vegetable oils and animal fats derivatives from liquid fuels (better known as biodiesel). Traditional batch processing has been and still is the most widely used process for this conversion. New and more efficient processing schemes, for large-scale production of biodiesel for fuel purposes, is a key for commercialization of biodiesel. In this study a continuous process for the transesterification of triglycerides to methyl esters was investigated. This process is based on a combined high shear and motionless reactor system. The experimental studies explored the variations in the mixing intensity, stoichiometry and catalyst concentration on the overall conversion.

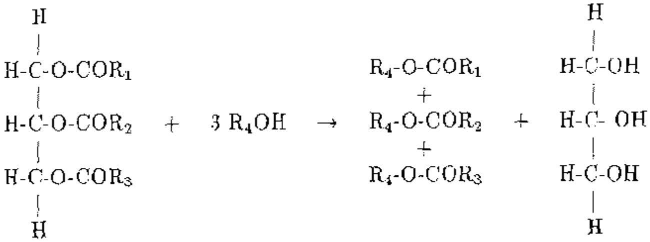
Keywords. Biodiesel, Vegetable oils, Pilot plant, Transesterification

INTRODUCTION

The idea of using vegetable oil as a substitute for diesel fuel is an old one going back to the invention of the compression ignition engines. In fact, Rudolph Diesel, the inventor of the Diesel Engine, operated an engine with peanut oil in a demonstration in 1900. Vegetable-oil fuels have been examined as a possible substitute for diesel fuel by many researchers (e.g., Peterson et al., 1983; Mazed et al., 1985; Samson et al.,1985). Although the concept of vegetable-oil fuels has been periodically introduced, it has never been seriously considered. This is mostly due to the fact that even though short term tests are satisfactory (e.g., Mazed et al., 1985; Samson et al., 1985), long term use of neat vegetable oils leads to severe engine problems (Pryde, 1983). The durability problems have been blamed on the fuel degradation due to its high viscosity, chemical structure of triglyceride molecules and incomplete combustion (Ryan III et al.,1984).

Esters of fatty acids have shown the greatest potential as alternative diesel fuels as a result of improved viscosity relative to the triglycerides. The viscosity of vegetable oils is reduced from about 10-15 times that of No. 2 diesel fuel to about twice that of No. 2 diesel for methyl esters which is low enough to be used as diesel fuel. Other physical properties of fatty acid methyl esters such as cloud point and pour point are considerably higher than No. 2 diesel fuel which limits its use as an alternative to diesel fuel. However, up to 30 vol% of the methyl esters in diesel fuel does not change the cold-flow properties of the fuel significantly (Ali et al., 1995; Dunn and Bagby, 1995). Studies have also shown that engines running on methyl esters emit slightly lower HC and CO and significantly lower particulates (Mittelbach and Tritthart, 1988). The NO_x emissions, however, are higher for methyl esters (Clark et al., 1984; Mittelbach and Tritthart, 1988).

Transesterification of bio-oils and fats is the most effective process for transformation of the larger triglyceride molecules of bio-oils and fats into smaller, straight-chain molecules of fatty acid esters. Transesterification involves the reaction between an alcohol and a triglyceride molecules in the presence of an alkaline catalyst.



$$\begin{array}{c}\text{H}\\|\\\text{H-C-O-COR}_1\\|\\\text{H-C-O-COR}_2\\|\\\text{H-C-O-COR}_3\\|\\\text{H}\end{array} + 3\,\text{R}_4\text{OH} \rightarrow \begin{array}{c}\text{R}_4\text{-O-COR}_1\\+\\\text{R}_4\text{-O-COR}_2\\+\\\text{R}_4\text{-O-COR}_3\end{array} + \begin{array}{c}\text{H}\\|\\\text{H-C-OH}\\|\\\text{H-C-OH}\\|\\\text{H-C-OH}\\|\\\text{H}\end{array}$$

The transesterification of vegetable oils (also known as alcoholysis) has been intensively investigated (e.g., Tanaka et al., 1981; Sankaran, 1990; Freedmen et al., 1984; Kusy, 1982) as widening industrial uses were found for esters (Meffert, 1984). Variables affecting the alcoholysis of vegetable oils such as: reaction time and temperature, catalyst type and concentration and the molar ratios of the reactants have been studied. Most of the research to date has focused on batch transesterification of vegetable oils with methanol or ethanol at or near the boiling point of the alcohol with sodium or potassium hydroxide and 50-300% excess alcohol for 1-2 h of reaction time. Nye and Southwell, (1983) studied the synthesis of methyl and ethyl esters of rapeseed oil in a bench-scale process and reported better than 92% conversion at room temperature. The effect of other reaction parameters such as catalyst concentration and mixing intensity on the yield of esters were also reported. Peterson and co-workers (1989) have also reported high yield of methyl esters at room temperature for the alcoholysis of rapeseed oil. This work was a pilot-scale and was to develop the reaction parameters and process variables. A low-waste process for the production of methyl esters of rapeseed oil has been recently reported by Ahn and co-workers (1995). Based on this technology, a 15,000 tons per year plant is in operation in Austria and another 30,000 tons per year plant is under construction in Czech Republic.

A factor of particular importance in the transesterification process is the degree of mixing between the alcohol and triglyceride phases. The triglyceride and alcohol phases are not miscible and form two liquid layers upon their initial introduction into the reactor. Therefore, mixing is expected to play an important role in the transesterification reaction. Although some type of mixing, mostly in form of mechanical stirrers, has been applied in most of the research studies, the effect of mixing intensity has not been fully addressed. Better understanding about the mixing effects on the transesterification process will be a valuable tool in the process scale-up and design.

The objective of this article was to develop a continuous process for the production of methyl esters of soybean oil as an alternative to diesel fuel. The effect of mixing intensity on the transesterification reaction was the primary variable which was investigated in this study. The process was equipped with two mixers/reactors, first a motionless mixer which was followed by a high shear mixer. The combined effect of the two reactors as well as the individual performance were examined. Other reaction parameters such as: reaction time, ratio of the reactants and catalyst concentration were also examined.

EXPERIMENTAL PROCEDURE

Materials

Refined and bleached grade soybean oil was provided by Archer Daniels Midland Company. The free fatty acid concentration of the oil was measured according to the AOCS #Ca Sa-40 and was determined to be 0.09%. Anhydrous methyl alcohol was obtained from

Mallinckrodt Baker, Inc. *Sodium hydroxide* (98.4%) was obtained from Fisher Scientific Company. The standards for the fatty acid methyl esters and glycerides were obtained from Sigma Chemical Company. HPLC grade solvents, acetone and acetonitrile were both obtained from Mallinckrodt Chemical, Inc.

A solution of 25 grams of sodium hydroxide per one liter of methanol was prepared. *Using this concentrated solution, the molar ratios and catalyst concentrations* were adjusted by changing the flow rates of the reactants.

Equipment

The major sections of the pilot plant include the pump station, preheating/premixing, mixing, the residence tube, cooling, and settling. A flow diagram for the pilot plant is presented in Fig. 1.

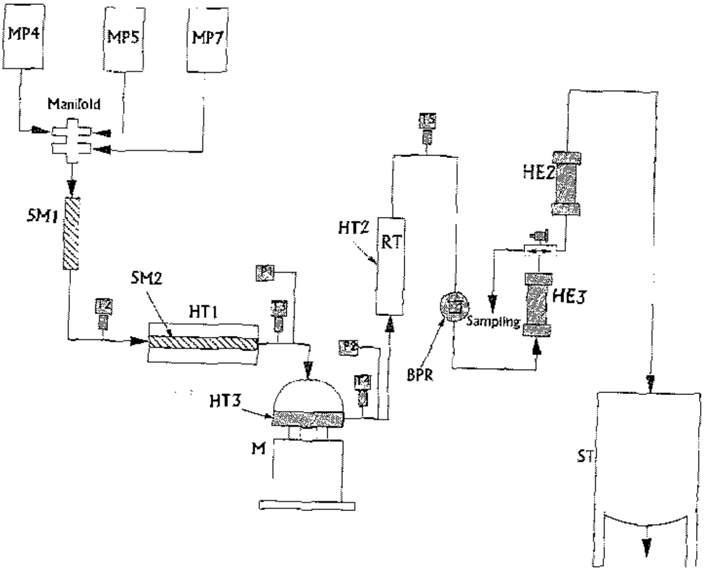


Figure 1: Flow diagram for the continuous. (MPn), metering pumps; (SMn), motionless mixers; (HTn), heaters; (M), high shear mixer; (Pn), pressure transducers; (Tn), thermocouples; (RT), residence tube; (BPR), back pressure regulator; (HEn), heat exchangers; (ST), separatory tank. transesterification process.

Pump Station

The pump station consisting of three E Series Pulsatron Pulsafeeder metering pumps. The in-line pumps were connected to a manifold which converged the flow into the pre-mixer. One of these pumps was used for soybean oil, one for methanol, and one for sodium hydroxide solution in methanol. The wetted parts of the pumps used for sodium hydroxide transfer were constructed with 316 stainless steel and teflon materials and the wetted parts of the other pump were made with PVC and teflon materials.

The flow rates of the pumps were varied linearly by changing the percent stroke rate and the percent stroke length. For example, a pump with a maximum flow rate of 0.1 L/min could be adjusted to 10^{-3} L/min by changing the percent stroke rate and stroke length to 10%. The flow accuracy decreased at very low stroke rates and lengths and they were not operated below 10% stroke rate and 10% stroke length.

The percent stroke rates of these pumps were controlled by the computer via a 4-20 mA signal while the percent stroke length was controlled manually. The percent stroke lengths were adjusted only for flow rates below 10% of the maximum flow rate. The pump station was capable of providing flows from 0.38 x 10^{-3} L/min to 0.8 L/min against a maximum pressure of 344.74 kPa (50 psi). These pumps were all calibrated and the flow rates were found to vary linearly with the percent stroke rate and stroke length.

Preheating/premixing

Once the reactants are converged into the pump manifold, they are first mixed in the in-line static mixers and then sent into the heater. The first static mixer was made of polypropylene and was located directly after the manifold. A second static mixer was installed inside the heater. The heater consists of four 0.3 kW cartridge heaters encased in an aluminum block with a flow tube in the middle. These heaters (heater 1) were controlled via a 4-20 mA signal which set the power output from 0% to 100%. The static mixer inside the heater was made out of stainless steel which further mixed the reactants as well as increased the heat transfer from the heater to the reactants. The static mixers were purchased from Koflo Inc. and the heater was purchased from Watlow.

Mixing

A continuous high shear mixer, was used to further mix the reactants. This high shear mixer was based on a rotor-stator design and was excellent for converting input power into mechanical and hydrodynamic shear energy. This was particularly useful for mixing immiscible phases such as methanol and soybean oil. The mixer had a capacity of 600 cm^3 and could operate at pressures and temperatures as high as 10.34 x 10^2 kPa (150 psi) and 204.44 C (400 F).

The mixer was equipped with a Watlow band heater and was used to heat the mixer during start-up. This heater (heater 3) was 3.81 cm (1.5 in.) wide with a 30.48 cm (12 in.) diameter and was rated at 1000 W.

The high shear mixer was connected to a variable frequency drive (VFD) manufactured by Utech Systems Associates Inc., which was connected to the control box. The VFD controlled the power going into the mixer from 0% to 100% and a linear relationship between the percent power input and RPM's was recorded. 100% power represented 3570 RPM. By this method, the mixing speed was controlled from the control box.

Residence Tube

The residence tube was used to increase the residence time of the transesterification reaction. The residence tube was simply a 38.1 cm (15in.) long hollow stainless steel 5.08 cm (2 in.) diameter pipe with a capacity of about one liter. This tube was heated on the outside with a 7.62 cm (3 in.) wide, 275 W Watlow flexible silicon rubber tape heater. The tape heater was controlled by varying the power output from 0% to 100% from the control box. The reactants leaving the residence tube were found to be homogeneous.

System Cooling

Cooling of the products coming out of the process was done by two miniature shell and tube heat exchangers. The shells of the heat exchangers were 30.48 cm (12 in.) long and 5.08 cm (2 in.) in diameter and contained twelve 0.95 cm (3/8 in.) tubes. The heat exchangers each had 847.28 cm^2 (0.912 ft^2) of heat transfer surface. The coolant could be run either through the tubes or the shell.

The coolant was supplied by a constant temperature circulating bath manufactured by VWR Scientific. A 50/50 mixture of ethylene glycol and water was used as the coolant. The cooling system was closed loop as the coolant was constantly recycled back into the constant temperature bath. A three-way valve was installed between the two heat exchangers for sampling.

Settling

After cooling, the products were collected in a 56.78 L (15 gal) separatory tank. The separatory tank was equipped with a drainage valve on the bottom. The products were allowed to sit for one day to allow the glycerol and methyl esters to separate. After settling, the glycerol and methyl ester phases were separated.

System Pressure

A Tescom 26-1700 Series self contained spring loaded back pressure regulator was used to pressurize the system. The back pressure regulator was rated for a maximum temperature and pressure of 220 C and 34.47 x 10^2 kPa (500 psi) respectively. The regulator was a precision relief device that allowed the working fluid to escape at such a rate to maintain the desired system pressure. The back pressure regulator was installed between the residence tube and the heat exchangers.

To monitor the pressure of the system, the pilot plant was equipped with two Rosemount (model 1151GP) gage pressure transducers. Due to the sensitivity of these pressure transducers to high temperatures, they were placed in a remote position to the flow and did not come in contact with the working fluid. The transducers transmitted a 4-20 mA signal to the control box which in turn transmitted real-time data to the computer. The pressures were measured in the line before and after the high shear mixer.

Control System

A control box was purchased from Landis and Gyr and was controlled by Procomm Plus for Windows on a personal computer. The control box had the ability to control the pumps, heaters and mixer and could also read various temperatures and pressures from the thermocouples and pressure transducers. The box was equipped with eleven separate power and controller modules.

PILOT PLANT OPERATION

Preparation

The control box had its own control language called the Power's Process Control Language (PPCL) developed by Landis and Gyr Powers Incorporated. Various control options such as setting up a PID controller on the heaters, using calibration constants to calculate flow rates and installing safety shutoffs were programmed. Meta keys were used in Procomm to make quick system changes for each run. The meta keys were macros which perform a specific operation(s) when activated. The meta keys were used to avoid the individual input of various process variables into the control box.

Using the Powers Process Control Language (PPCL), a program was written specifically for pilot plant operation. For each run, the desired flow rates and set point temperatures were programmed into meta keys in Procomm Plus to allow for quick system changes during the run. The PID controller's gain values were optimized to obtain temperature control within ± 5 C of the set point temperatures.

Start-Up

The High shear mixer was initially heated for five minutes by the hand heater. Then the mixer, preheater, and residence tube heater were heated for an additional five minutes with 100% power for the preheater and 50% power for the residence tube heater. Using the meta keys, the soybean oil flow and the temperature controllers for the high shear mixer heater and residence tube heater were activated simultaneously. Once the temperatures before and after the high shear mixer (T3 and T4) exceeded 70 C, and the temperature after the residence tube (T5) exceeded 65 C, the methanol and sodium hydroxide flows were started. The system temperature and pressure were set to 80 C and 172.37 kPa (25 psi) respectively. The heating process took about 20 to 30 minutes. The coolant for the heat exchangers was set to 15 C.

Steady State Operation

The system was operated for a period of time which would allow six liters of material to pass through the system before a sample was taken. This was approximately two times the system volume. On a series of consecutive runs, this would allow the system to come to near steady-state conditions as well as to completely flush out the material from the previous run before sampling. The samples were collected in a conical flask. After each sample was taken, the meta keys were used to change the desired system parameters including flow rates and mixing speeds.

Each sample was covered and was allowed to stand for one day before separating the methyl esters from the glycerol. After one day, the methyl esters were removed, neutralized and then placed in a vial for further analysis.

ANALYSIS

A high pressure liquid chromatography system consisting of a pump (model no. 2350) manufactured by ISCO and a refractive index detector (Refracto Monitor IV) made by Thermo Separation Products was used for analyzing the samples. A spherisorb ODS 2 column 250 x 4.6mm with 80 micron pore size and 5 micrometer particle size was used for the separation. Temperature of the column was maintained at a constant value of 35 C by a Brinkman RC 6 temperature bath. Data collection and analysis were performed with a Hewlett-Packard's Chemstation. The mobile phase for the system was a 50/50 volumetric ratio of acetone and acetonitrile. The mobile phase was degassed for about 20 min before use in the system.

The flow rate of the mobile phase was set to 0.7 mL/min. The temperature of the column was maintained at 35 C. About 6 mL of degassed HPLC mobile phase was used to dissolve about 2-3 drops of the methyl esters sample. Once the above conditions were set, the system was allowed to stabilize.

The components measured by the HPLC included methyl esters, monoglycerides, diglycerides, and triglycerides. The percent conversion was taken as the conversion of triglycerides to methyl esters. The relative HPLC areas and component masses were calibrated using standard samples of known compositions.

RESULTS AND DISCUSSION

Experiments were performed to study the effects of catalyst concentration, residence time and the mixing intensity on triglyceride conversion. The results are summarized in the following sections.

Effect of Catalyst Concentration

Pilot plant runs were conducted to study the effect of catalyst concentration. The *mixing intensity and the flow rate were held constant at 25% power* and 0.3 L/min respectively. Figure 2 summarizes the experimental results for the effect of variations in the catalyst concentrations from 0.1-1.0 wt% (based on soybean oil) on the reaction conversion for molar ratios of 6:1 and 8:1 (methanol:soybean oil). This figure shows that as the catalyst concentration increases, the triglyceride conversion also increases. However, at higher catalyst concentrations, this effect becomes minimal. The upper limit to the catalyst effectiveness is reached at a much lower catalyst concentration for larger alcohol excess levels.

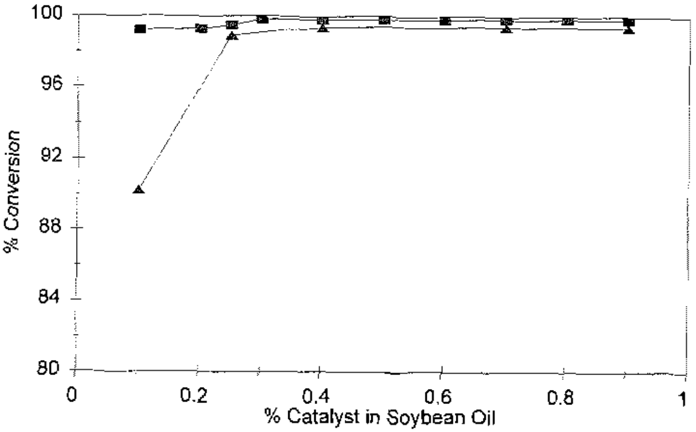


Figure 2: *The effect of variations in the catalyst concentration on the overall conversion* subject to the reactants molar ratios of 6:1 and 8:1. (▲), 6:1 reactants molar ratio; (■), 8:1 reactants molar ratio.

Although higher conversions are achieved at larger catalyst concentrations under otherwise identical flow and stoichiometric conditions, there are drawbacks to the excessive use of alkaline catalyst in the trasesterification reaction. Higher catalyst concentrations increases the solubility of methyl esters in the glycerol phase in the final product. As a result, a significant amount of methyl esters remains in the glycerol phase after the phase separation. Batch experiments were performed to observe this phenomena. The ratio of the weight of the top phase (methyl esters) to bottom phase was monitored as the catalyst concentrations was varied. These experiments were conducted using catalyst in both aqueous (50 wt%) and solid form. The results are summarized in Fig. 3. The results reveal that as the catalyst concentration was varied from 0.1 to 1.0 wt%, the top layer decreased from about 85 wt% to about 75 wt% of the total product. No significant reduction in the amount of methyl esters was observed in the 0.1-0.4 wt% range for solid catalyst. Moreover, usage of aqueous catalyst resulted in a significantly lower methyl ester layer. The formation of soap may be responsible for this behavior.

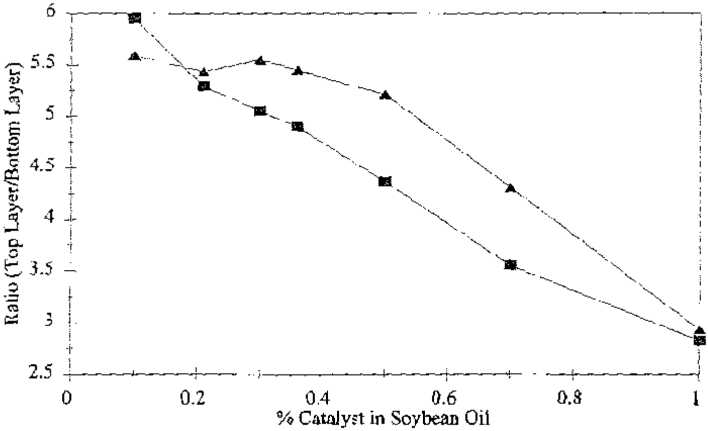


Figure 3: The effect of variations in the catalyst concentration and form on the product distribution. (■), aqueous catalyst; (▲), solid catalyst.

In the transesterification process, separation of products by gravity settling is very desirable. Therefore, solid form of the alkaline catalyst at less than 0.4 wt% was considered for further investigations.

Effect of Molar Ratio

Even though the stoichiometric molar ratio of methanol to triglyceride for transesterification is 3:1, higher molar ratios are used to enhance the solubility and to increase the contact between the triglyceride and alcohol molecules. Experiments were conducted with molar ratios of 8:1 and 6:1 for mixing intensities at 0-100% of the high shear mixer. Figure 4 represents the results of these experiments. The figure shows that for all mixing levels the molar ratio of 8:1 has better conversions than 6:1.

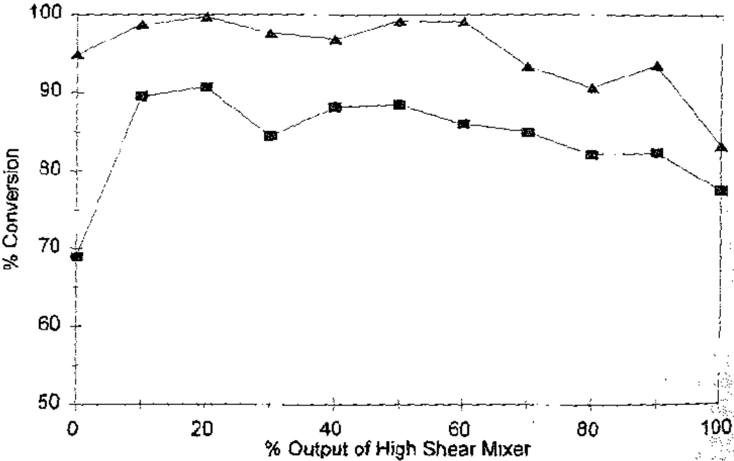


Figure 4: The effect of variations in reactants molar ratios on the overall conversion at various mixing intensities. (■), 6:1 reactants molar ratio; (▲), 8:1 reactants molar ratio.

Effect of Flow Rate

The pilot plant was operated at flow rates ranging from 0.250-0.350 L/min to study its effect on triglyceride conversion. Figure 5 shows that conversion was not significantly affected by the flow rate.

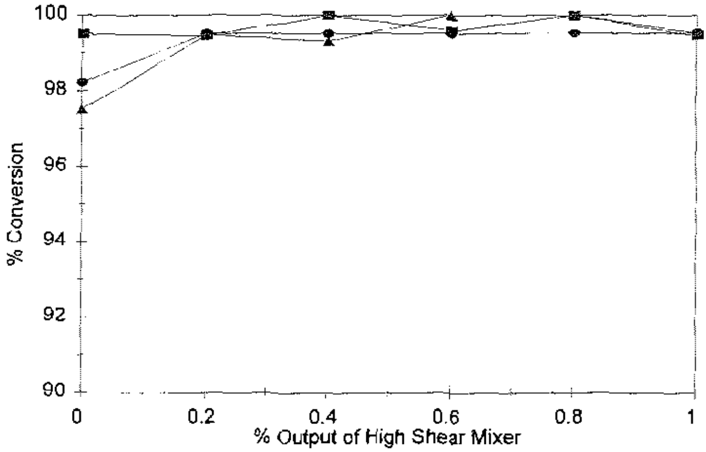


Figure 5: The effect of variations in flow rates on the overall conversion at various mixing intensities. (▲), 0.25 L/min; (■), 0.30 L/min; (●), 0.35 L/min.

Effect of Mixing

The effect of mixing intensity at various catalyst concentrations was examined first. These experiments were conducted with motionless mixers in the process while the high shear mixing intensity was varied. Several experiments at catalyst concentrations of 0.1-0.4 wt% were conducted with varying mixing and molar ratios. These results are presented in Figs. 6 and 7. As these figures indicate higher mixing intensities and larger catalyst concentrations both have favorable effects on the overall conversion. There is an upper limit to both the catalyst concentration and mixing intensity where almost complete conversion of triglycerides to methyl esters is achieved. This limit is reached much faster for larger alcohol excess levels. An upper limit of 0.3 wt% and 0.4 wt% for the catalyst concentration was picked for the molar ratios of 8:1 and 6:1 respectively.

The next set of experiments examined the effect of the static mixers in the process. At 0.4 wt% and 0.3 wt% catalyst concentration for 6:1 and 8:1 molar ratios of the reactants, experiments were performed with and without the presence of motionless mixers in the processing line. The inteusity of the high shear mixer was varied from 0 to 75 % of the maximum ontput of the mixer. The results are summarized in Figs. 8 and 9. The results show that both the static mixer and the high shear mixer are capable of converting soybean oil to methyl esters. Conversions in excess of 99% are possible. Larger excess amounts of alcohol favors higher conversions. Figures also show that significant reaction occurs even without static mixers and the static mixer.

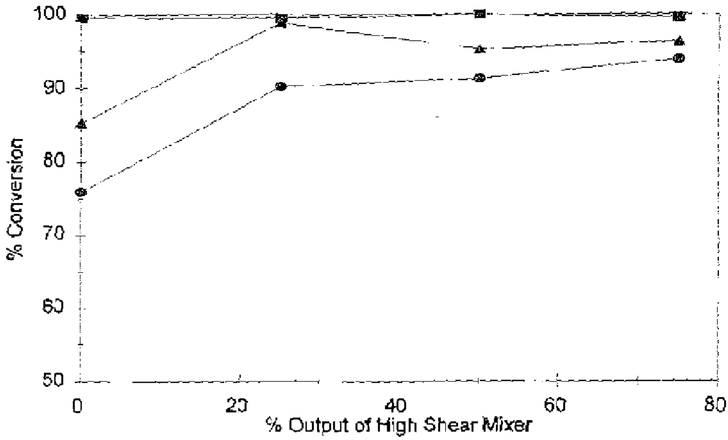


Figure 6: The effect of variations in mixing intensity and catalyst concentration on the overall conversion subject to the reactants molar ratio of 6:1. (●), 0.1% catalyst; (▲), 0.25% catalyst; (■), 0.4% catalyst.

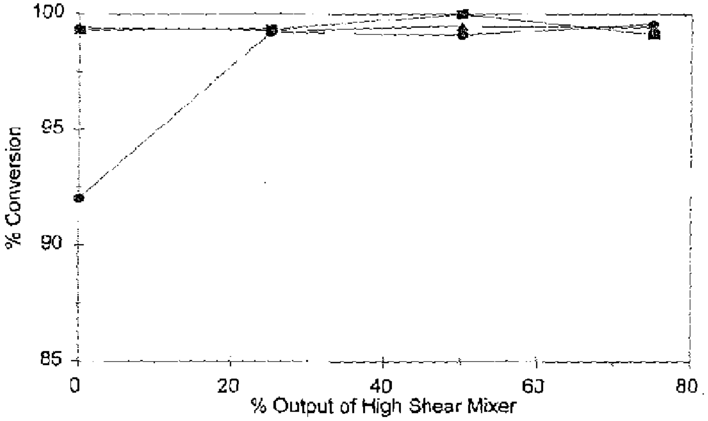


Figure 7: The effect of variations in mixing intensity and catalyst concentration on the overall conversion subject to the reactants molar ratio of 8:1. (●), 0.1% catalyst; (▲), 0.2% catalyst; (■), 0.3% catalyst.

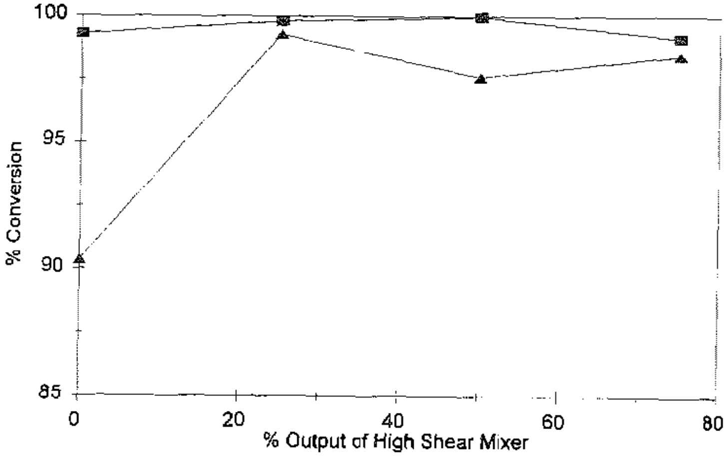


Figure 8: The effect of variations in mixing intensity on the overall conversion subject to the reactants molar ratio of 6:1 with and without the static mixers. (■), with static mixer; (▲), without static mixer.

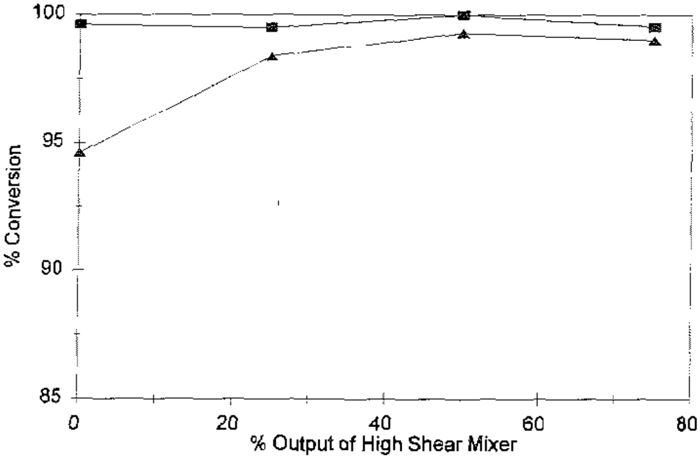


Figure 9: The effect of the motionless mixers on the overall conversion subject to the reactants molar ratio of 8:1. (■), with static mixer; (▲), without static mixer.

ACKNOWLEDGMENTS

The authors express their gratitude to U. S. Department of Agriculture Cooperative State Research Service and Nebraska Soybean Board for their support of this work.

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