

## Process Control for Biodiesel Applications Using the SenGenuity ViSmart™ Viscosity Sensor

### Introduction

In the current economic climate the need for renewable energy sources that are alternatives to traditional petroleum based fuels. Biodiesel is emerging as the fuel of choice for the next generation of ecologically friendly vehicles and much effort is being exhausted in improving its manufacturability. Biodiesel also produces less hydrocarbons, sulfur, and particulate matter than petroleum based fuel.

The process that produces biodiesel involves converting triglycerides to alkyl esters using a catalyzed transesterification reaction. Vegetable oils or animal fat are comprised of esters of free fatty acids bound to the trihydric alcohol, glycerol. During transesterification the triglycerides are mixed with an alcohol (commonly ethanol or methanol) that takes the place of the glycerol with the fatty acid esters forming 3 alkyl esters for every one triglyceride molecule. This reaction usually takes place very slowly or not at all and so is catalyzed with a base such as KOH and the addition of heat, ultrasonic, or microwave energy.

The details of the transesterification process have the biggest impact on the quality and quantity of the biodiesel produced. Contaminates, such as water or an abundance of free fatty acids in the triglyceride feedstock, can greatly reduce the efficiency of the reaction. Monitoring this reaction step with an in-situ measuring device would allow the user accurately judge the condition of the starting fluid and the completeness of the reaction as it takes place. This would compensate for the slight differences in each batch of oil feedstock that lead to differences in process time.

### Viscosity Measurements

It can be shown that the viscosity of the fluid mixture can be used as an indicator of the level of completeness of the process. As triglycerides are broken down the viscosity of the mixture changes from the value measured in the feedstock to a value that is indicative of the percentage of each constituent component's viscosity. The viscosity of feedstock is determined by the mix of oils and/or tallow that goes into it. Figure 1 shows the change in measured shear stress for different oils during the transesterification reaction. From the addition of the catalyst and methanol at the beginning of the measurements the shear stress decreases. When the shear stress plateaus the reaction has fully propagated.

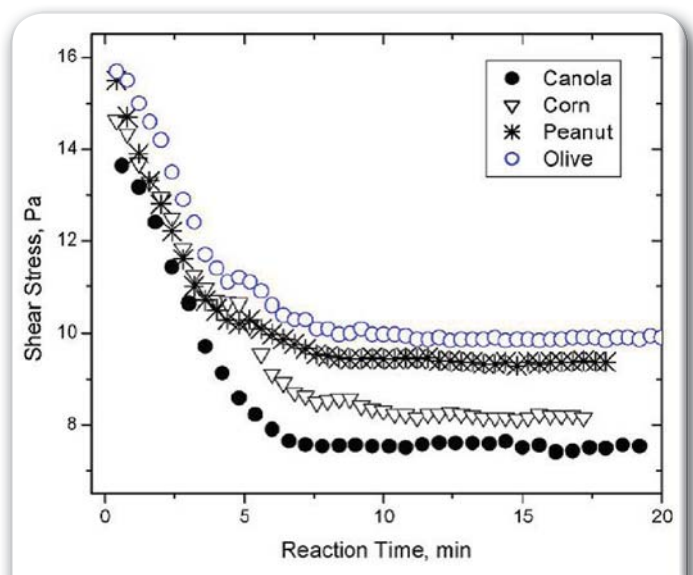


Figure 1: Comparison of change in shear stress of reaction mixture with time for different feedstock oil.

## Customer Application and Data

The transesterification of canola oil and waste vegetable oil was continuously monitored in-situ at 60°C using the SenGenuity ViSmart™ high shear rate viscometer. The typical change in viscosity during this reaction can be seen in Figure 2. Data was logged at one sample per second for one hour. A large drop in viscosity can be seen at the beginning when potassium methoxide (KOCH<sub>3</sub>) was introduced into the canola oil. Once the reaction reached its equilibrium the viscosity remained at a consistent value lower than that of the original oil. Temperature was controlled to 60°C during the test. The initial drop in temperature is due to the addition of the catalyst mix which is kept at room temperature and the slightly endothermic nature of the transesterification reaction. After the initial drop was corrected for there were still some minor fluctuations due to

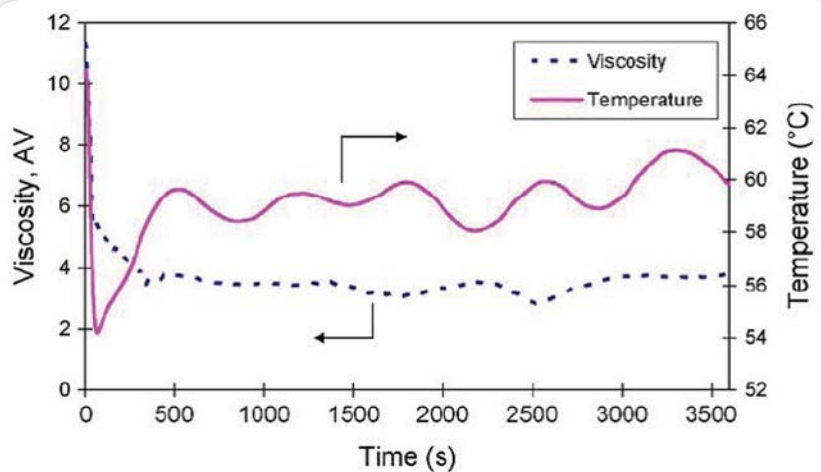


Figure 2: Viscosity and temperature profile during transesterification reaction for canola feed.

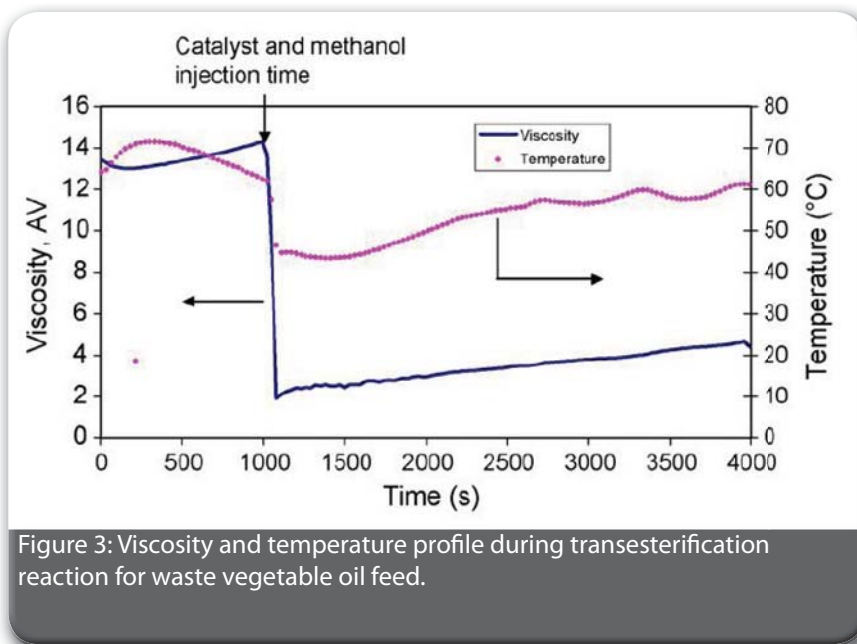


Figure 3: Viscosity and temperature profile during transesterification reaction for waste vegetable oil feed.

the temperature controller. These minor changes did not seem to affect the viscosity measurement, though. Once the mixture was allowed to settle a line between the crude biodiesel and the glycerol was clearly observable.

Waste vegetable oil contains higher concentration of fatty acids, water and impurities than pure canola oil does. These materials make the conversion more difficult due to side reactions. However, for the complete reaction of waste vegetable oil (WVO) to biodiesel, the ViSmart™ sensor was able to monitor the reaction as indicated in Fig. 3.

After studying the characteristics of the ViSmart™ sensor at a bench top scale, the customer investigated the installation in the pilot plant. The sensor was affixed to a rod (all associated materials were resistant to a wide range of pH values and also prevented gas vapors from escaping) and installed from the top of the reactor. The sensor did not interfere with the mixing blade and all acquired measurements were independent of sensor location. Repeated experiments were carried out with 150 L of WVO, and 30 L of methoxide (30 L methanol and 0.6 kg KOH). Viscosity measurements were taken every 10 min for the 150 min duration. Since there was some difficulty in controlling the reactor temperature, a significant drop in temperature was observed after the addition of methanol and catalyst. Despite the continual drop in temperature, the mixture's viscosity decreased until hitting a plateau as shown in Fig. 4.

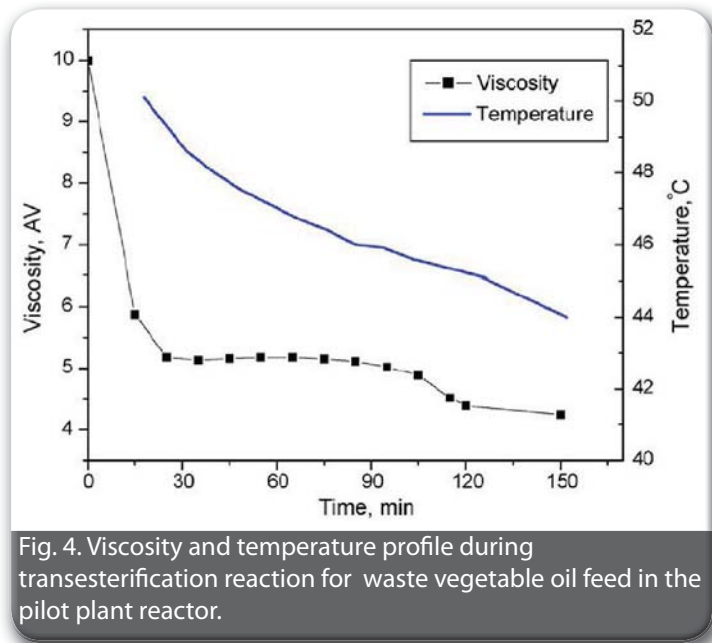


Fig. 4. Viscosity and temperature profile during transesterification reaction for waste vegetable oil feed in the pilot plant reactor.

As seen to the left the transesterification reaction was successfully monitored in a pilot scale reactor using an acoustic wave viscometer. GC analysis of the final product revealed conversion of triglyceride to methyl ester (biodiesel) once the plateau in the viscosity curve was reached.

Hence, by using the SenGenuity ViSmart™ viscosity sensor, which can follow the transesterification reaction of waste vegetable oil into biodiesel and glycerine, a tool for process control allowing more efficient conversion can be utilized.

## Contact Information

If you would like to learn more about our sensors, the markets we serve and customer applications we strive to address, please do not hesitate to contact our Application Engineering group at [support@sengenuity.com](mailto:support@sengenuity.com).

## Reference Material

All figures, application discussion and data are provided greater detail in the paper "Monitoring biodiesel production (transesterification) using in situ viscometer" by Naoko Ellis, Feng Guan, Tim Chen and Conrad Poon of the Department of Chemical and Biological Engineering, University of British Columbia, Vancouver, Canada. The paper is published in Chemical Engineering Journal, Issue 138 (2008), pages 200-206.

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