Comparative Study of Transesterification of Refined Oil Using a Batch Reactor versus a High Shear Mixer Pilot Scale Reactor

Biodiesel was synthesized with refined soy bean, corn, and canola oil using methanol in the presence of sodium methoxide catalyst. The transesterification reaction was first studied using a 3 liter batch reactor in the laboratory. Following the reactor study a pilot scale process using a 1 gallon per minute high shear mixer reactor was designed and built to mimic transesterification conditions conducted in the laboratory.
In each reactor setting (i.e. 3 liter reactor and high shear mixer reactor), synthesized biodiesel was made in two successive steps with glycerine separation in between. A typical biodiesel synthesis process utilizes an acid to neutralize the sodium methoxide followed by water washing to clean the biodiesel before vacuum drying. Here the biodiesel was cleaned and neutralized using commercially available ion exchange resins (Amberlite BD 10 dry, Dowex DR-G8, and Purolite PD-206). For the first study (3 liter reactor), these resins were mixed in with the biodiesel for 20 minutes. The resins were separated before vacuum drying. In the case of the high shear mixer reactor, the resins were packed into a fixed bed column.

Analysis of both the crude and refined biodiesel in this study included soap content analysis, free fatty acid analysis, as well as free and total glycerine analysis. The total glycerine analysis gave an indication of how well the starting refined oil was converted into biodiesel.

Preliminary experiments were conducted using an IKA labor pilot shear mixer. The configuration of the mixer included a single mixing head generator (stator/rotor). Refined soy bean oil was subjected to a transesterification reaction of triglyceride which was conducted in the presence of methanol solvent and sodium methoxide catalyst. The reaction was divided into two stages. The theoretical conversion for stage 1 was calculated at 90 wt%, whereas the remaining unreacted triglyceride was reacted in stage 2. At the end of the second stage the resulting crude methyl esters were polished using a resin Amberlite BD 10 dry. During the reaction, samples were taken at 15 minute intervals to monitor how the reaction proceeded. Shown below (Figure 1-4) are results obtained from the analysis of the soap content during the reaction and total glycerine.

Based on results in Figure 1-2, high levels of soaps are present throughout the reaction. The presence of these soaps did not seem to impact the conversion of triglyceride. Supplemental results suggested that diglycerides and monoglycerides were formed. Results in Figure 3-4 further emphasize that the reaction was not affected tremendously by the presence of soaps. In addition, Amberlite BD 10 dry removed any impurities present in the crude biodiesel. The levels of free glycerine were minimal. The high soap levels present seemed to favor the glycerine phase during the separation at the end of each stage.

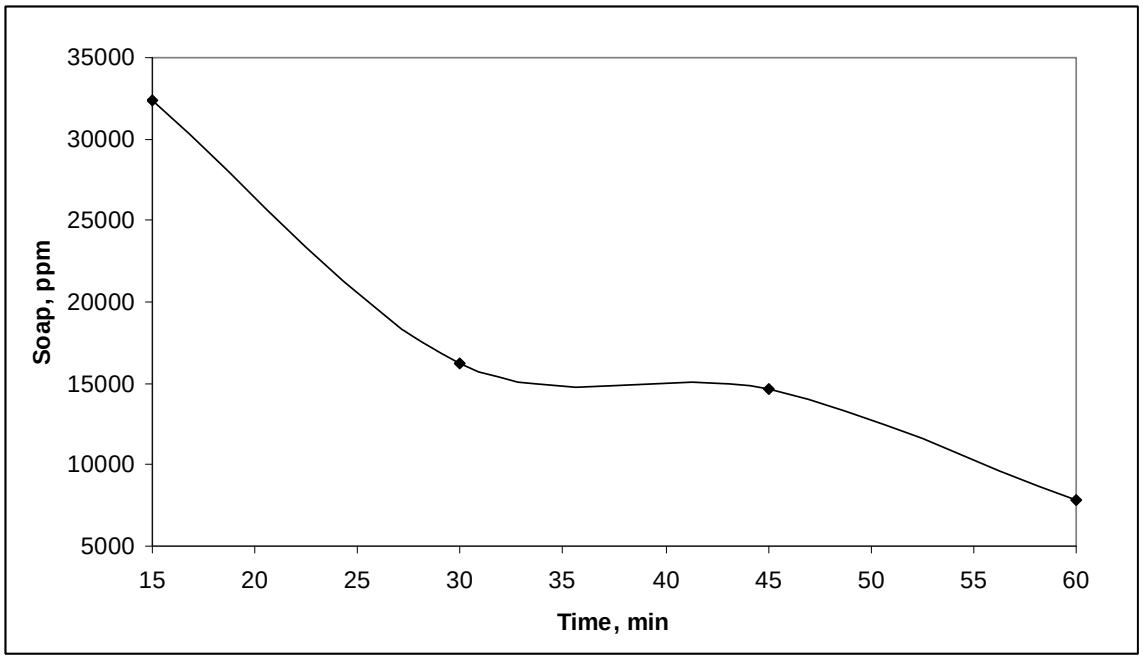

Figure 1. Soap content measurement as sodium oleate during a transesterification reaction Stage 1

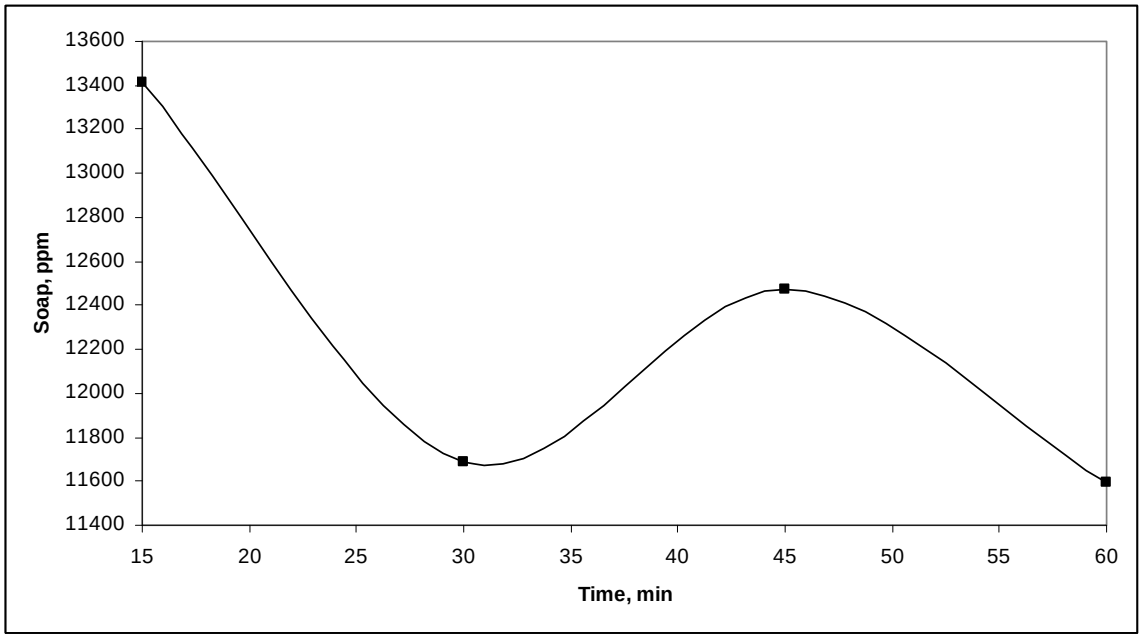

Figure 2. Soap content measurement as sodium oleate during a transesterification reaction Stage 2

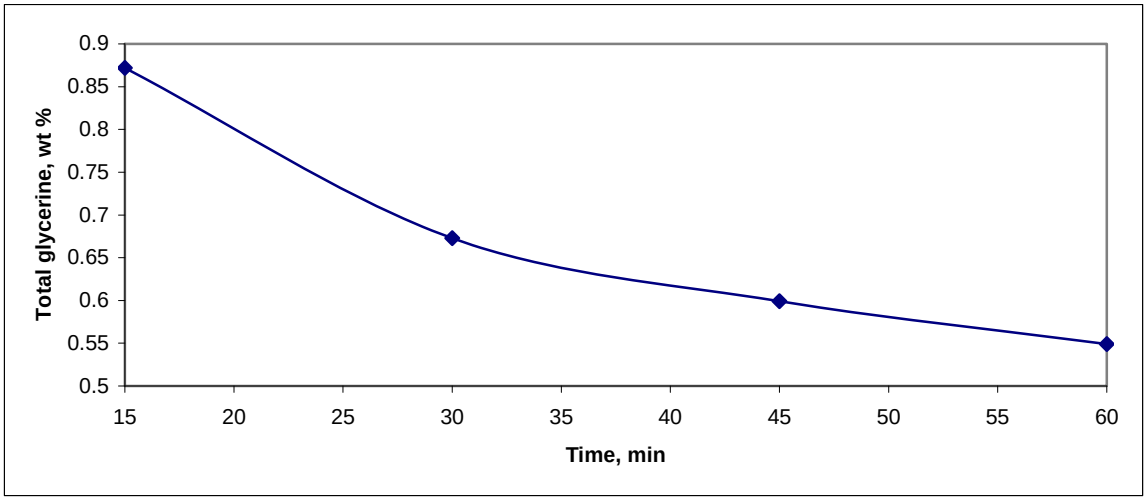

Figure 3. Change in the total glycerine during a stage 1 transesterification reaction

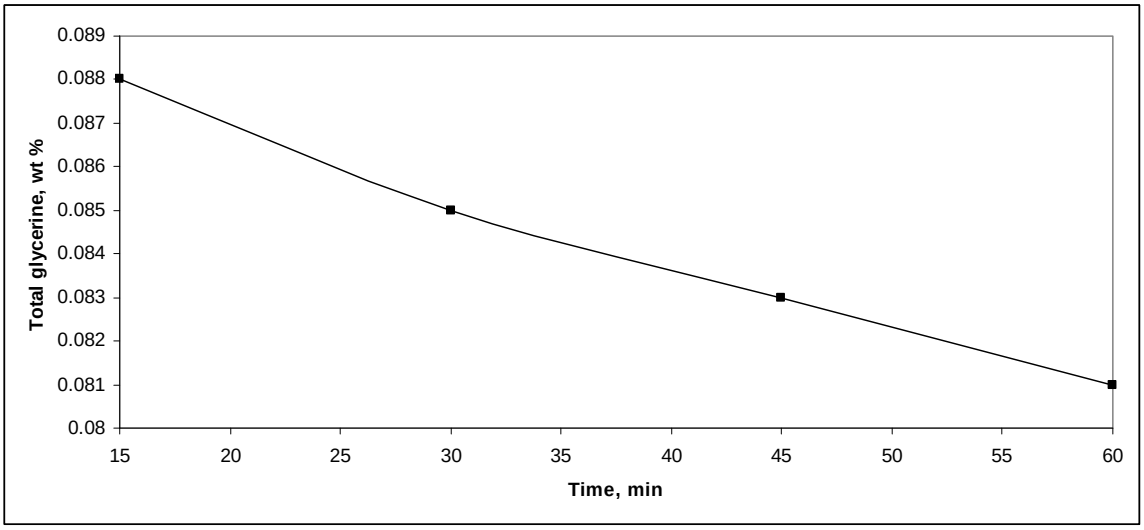

Figure 4. Change in the total glycerine content during a stage 2 transesterification reaction