Fast Separation of FFA, FAME, and Glycerol for Biodiesel Analysis by Supercritical Fluid Chromatography

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The alternative fuel is increasing in popularity owing to various technical and economic factors, especially the upsurge in petroleum prices and the implementation of financial incentives for its use. Biodiesel holds a prominent position among the alternative fuels. Depending on the production process, biodiesel product typically contains not only the desired alkyl esters, but also some residual starting material, catalyst, by-product and intermediates. Rapid, reliable and precise determination of the contaminants in biodiesel is of great importance for both process monitoring and product quality control.

Gas chromatography (GC) and high performance liquid chromatography (HPLC) are common analytical techniques for biodiesel analysis. However, both techniques suffer from the relatively long analysis time. We present herein our preliminary study on using SFC for fast biodiesel analysis, taking advantage of the higher separation speed and efficiency of SFC, rendered by the intrinsic low viscosity and high diffusivity of supercritical CO_2.

The utility of SFC methodology for biodiesel analysis is illustrated in Figure 1 and 2 with an overall analysis time of 3.5 minutes. It is interesting to note that the three main classes of compounds in biodiesel product, namely, glycerol, FFA, and FAME, are separated in group, with glycerol being the first to elute off the column and FAMEs being the last. In biodiesel production, information on the relative amount of FAME, FFA, and glycerol is sufficient for critical process decision-making. Unlike the group separation by SFC in ASTM D5186 for fossil fuel analysis, the group separation of aforementioned compounds by SFC does not involve any valve-switching scheme and is achieved with a C18 column in 3.5 minutes.

We have shown a group separation of glycerol, FFAs, and FAMEs, the main target compounds for biodiesel analysis, by SFC in 3.5 minutes, representing a 3- and 5- fold increase in throughput compared to UPLC and HPLC, respectively. SFC holds great potential to become the choice of chromatographic technique for biodiesel analysis.

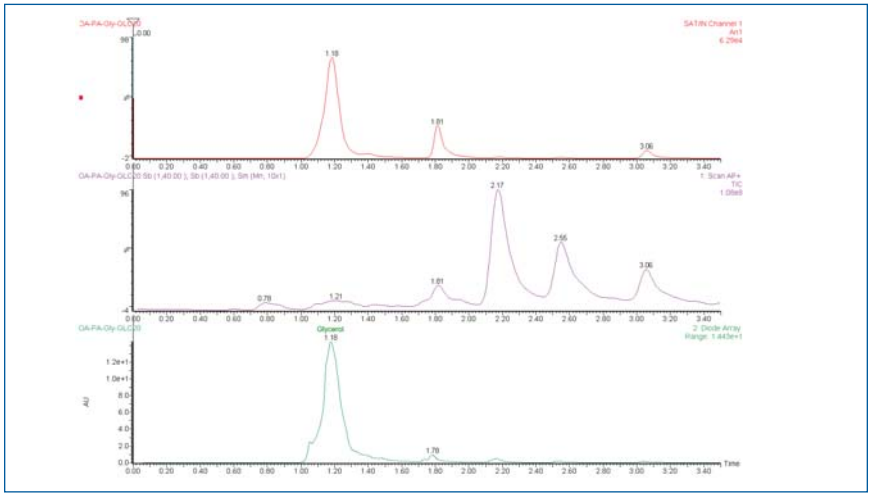

Figure 1. *Chromatogram of FAMEs, FFA and Glycerol mixture. From top to bottom are ELSD, MS (TIC) and UV trace, respectively.*

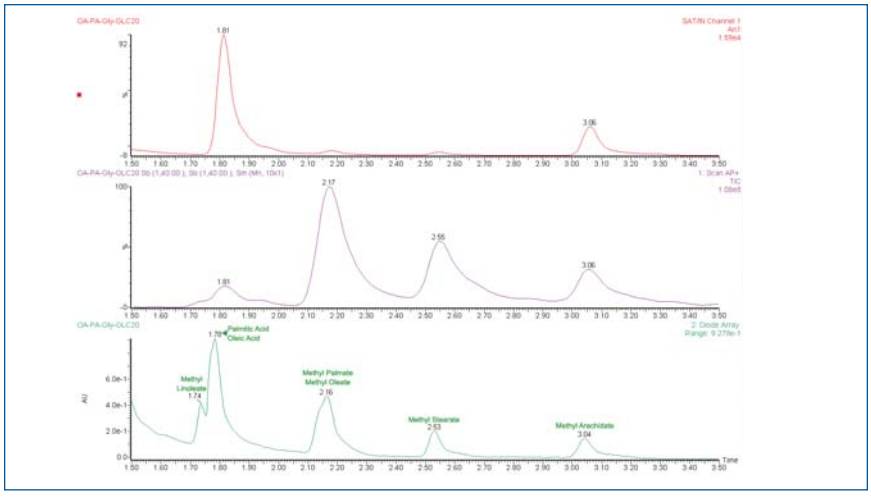

Figure 2. *Expanded chromatogram of FAMEs, FFA and Glycerol mixture (1.5-3.5 min). From top to bottom are ELSD, MS (TIC) and UV trace, respectively.*