



Determination of Glycerol in Biodiesel

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Abstract

The one-step extraction of free glycerol from biodiesel was demonstrated to be reliable, fast, and accurate. Using the XBridge Phenyl HPLC column, excellent results were obtained.

Introduction

In recent years, biodiesel fuel has been gaining popularity as a more environmentally friendly alternative to fossil fuels, especially in Europe (which is the leading producer of biodiesel) and Latin America. The United States has tended to place more emphasis on bioethanol as a renewable fuel source but, along with China and Canada, it is beginning to see more attention placed on biodiesel as well. Production of biodiesel is predicted to reach 12 billion liters by 2010. "Biofuel Market Worldwide (2007-2010)", published May 2007, RNCOS.

Biodiesel is produced from renewable sources of oils or fats through a process called transesterification. Within this production process, glycerol arises as a side product. Glycerol is also present in the final product in small amounts which must be kept within regulatory levels (maximum concentration of 0.02 wt-% as set by ASTM specification and EU regulation EN 14214:2003). It is therefore necessary to measure the amount of free glycerol in biodiesel.

Many methods exist for glycerol determination employing gas chromatography, however they require complex extraction procedures. The method detailed in this report is based upon HPLC analysis and utilizes a one-step extraction of free glycerol into water prior to its analysis on an XBridge Phenyl HPLC column.

Experimental

Sample Preparation

A known amount of biodiesel was placed into a glass bulb (50 mL) with a calibrated cylinder. Approximately 4.5 mL of distilled water was added. A capillary was inserted to bubble air through the liquid in order to obtain adequate mixing. After 2 hours of mixing, total separation of the water phase from the biodiesel phase was achieved. The upper biodiesel phase was removed. The water phase, with the extracted glycerol, was evaporated by vacuum.

| HPLC Conditions | |
|-----------------|---------------------------------------|
| Column | XBridge Phenyl, 3 x 150 mm, 5 μ m |
| Part Number | 186003345 |
| Mobile Phase | Demineralized water |
| Flow Rate | 0.5 mL/min |
| Temperature | Ambient |
| Injection | 0.5 mL |
| Detection | Refractometric Detector (RIDK 120) |

Results and Discussion

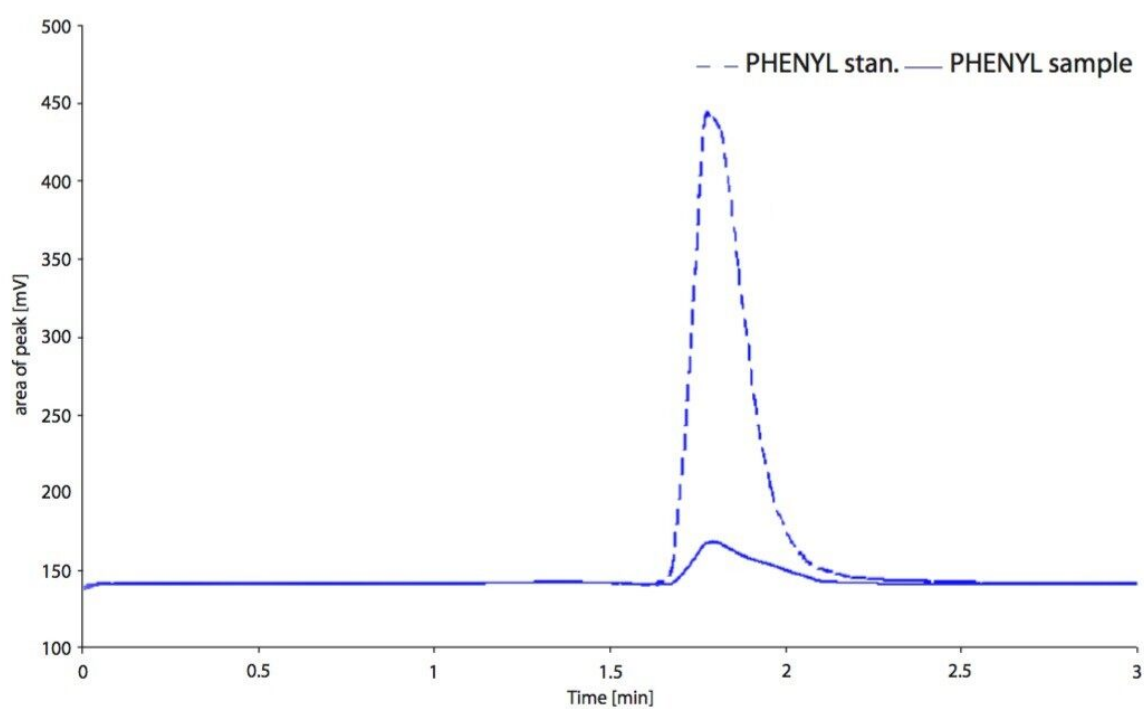


Figure 1: Glycerol standard analyzed using an XBridge Phenyl column, overlaid with a sample which was run following extraction.

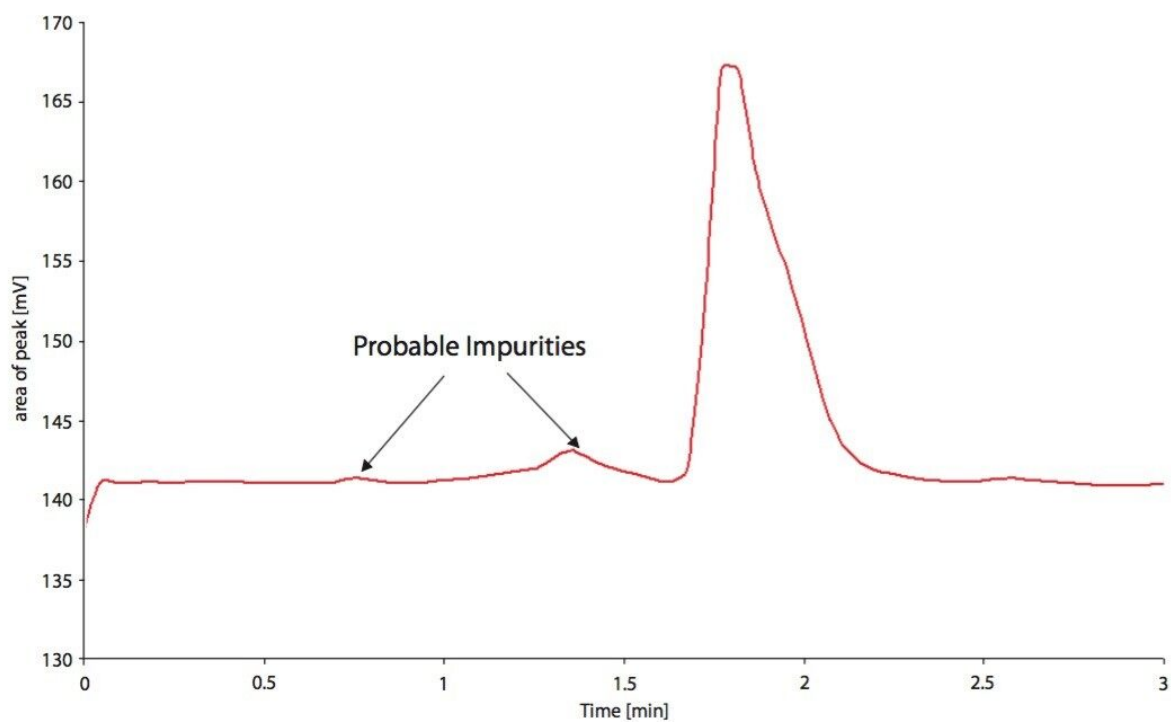


Figure 2: Analysis of glycerol extract using XBridge Phenyl column.

Conclusion

The one-step extraction of free glycerol from biodiesel was demonstrated to be reliable, fast, and accurate. Using the XBridge Phenyl HPLC column, excellent results were obtained.

References

1. Chromatograms and methods for this report provided courtesy of the University of Pardubice, Pardubice, Czech Republic.

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WA60204, April 2008

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