OP-08

GAS CHROMATOGRAPHIC DETERMINATION OF METHANOL IN BIODIESEL SAMPLES BY HEAD-SPACE SOLID PHASE MICRO EXTRACTION TECHNIQUE


*E-mail: levent.pelit@ege.edu.tr

Ege University Science Faculty, Dep. of Chemistry, Izmir, Turkey

Biodiesel is a promising and alternative fuel to conventional diesel since it is environmentally friendly and renewable fuel source. It is produced from vegetable oils or animal fats via transesterification using methanol to yield Fatty Acid Methyl Esters (FAME) and glycerine. The yield, pure FAME is called B-100. In order to ensure the quality of B-100 biodiesel, determination of its methanol content is essential [1]. Gas chromatographic (GC) methods with flame ionization detector (FID) are typically employed for methanol determination and sample preparation usually determines the sensitivity of the method.

In the last two decades, simple and greener extraction techniques are being searched. Solid phase micro extraction (SPME) is a solventless and environmental friendly technique along with other advantages, such as high sensitivity, simplicity and rapidity. A polymeric film coated on the SPME fiber provides an adsorbent surface for organic molecules and it can also be combined with headspace technique (HS) for the determination of volatile organic compounds eliminating the interferences from complex matrices.

Present study describes the use of polythiophene coated SPME fiber doped with clay for the preconcentration of methanol in biodiesel for subsequent determination by GC-FID system. For this purpose a stainless steel wire was cleaned and used as a working electrode in a voltammetric cell containing 0.3 M thiophene monomer and 0.88 mg of montmorillonite (MMt) clay prepared in 0.1 M NaClO₄ solution in acetonitrile. The electropolymerization procedure was initiated by cycling the potential in a range of -0.2 +2.2 V with a scan rate of 50 mV s⁻¹. Close examination of the surface by Scanning Electron Microscopy (SEM) has revealed a porous structure of the fiber. The fiber was then inserted into the vial in HS position where it is exposed to the methanol content of the biodiesel sample.

A variety of parameters such as adsorption time and temperature, salt effect, desorption temperature were studied for optimization of the method. Under optimized conditions, the linear range was determined as 0.004-0.1% (m/m) and limit of detection (LOD) was 0.0012% (m/m). The method developed was successfully applied to the biodiesel sample and the results were confirmed with those obtained in an accredited petroleum analysis lab (Egepal).

KEYWORDS: Methanol determination, biodiesel, Head space, SPME, Gas chromatography

REFERENCES: