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Determination of fatty acids by head space single drop microextraction gas chromatography (HS-SDME-GC)**Aysel Berkkan and Aslihan Ekici**
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Statement of the Problem: There are three major aspects in analytical measurements; first, to develop microanalytical methods. Second to perform rapid measurements on small sample volumes. Third, green analytical methodologies. Single Drop Microextraction (SDME) have special value in sample cleanup, rapid extraction and large enrichment factor. So, head space-single drop microextraction (HS-SDME) gas chromatography coupled to flame ionization detector (HS-SDME-GC-FID) was used to determine the fatty acids.

Methodology & Theoretical Orientation: Fatty acids in oil samples were derivatized to improve volatility with esterification reaction. Methylation was carried out by trans-esterification with methanolic potassium hydroxide in order to convert the polar carboxylated groups to less polar methyl ester derivatives. Then, microextraction step was applied in 10 mL head space vial by using the GC microsyringe with a bevel tip that holds the drop. Extraction solvent (sodium dodecyl sulphate: 1-butanol, 1:3), concentration of ionic strength adjustment solution (NaCl), temperature (45°C), and extraction time (35 min) that affect the yield were optimized. Agilent Technologies, Inc. HP-5 column (30mx 0.320mm ID, 0.25 µm film thickness) was used in GC. Inlet and detector temperature were 270°C. Oven temperature program was as follow; 80°C (2.0 min), 4°C/min ramp to 210°C (5 min), 15°C/min ramp to 300°C (5.0 min), helium was used as a carrier gas (1 mL/min flow rate). Injection sample volume was 1 µL with 100:1 split ratio.

Findings: Chromatogram of fatty acids' methyl esters in olive oil was shown in Figure.

Conclusion & Significance: The identification of linoleic acid, and linolenic acid's methyl esters in olive oil were performed by comparing their retention times with the standarts by HS-SDME-GC-FID. The amount of acids was expressed as a percentage (w/w) of all fatty acids' esters detected, linoleic acid (3%), and linolenic acid's (16%).

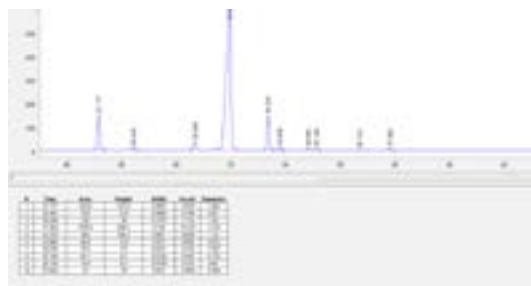


Fig. HS-SDME-GC-FID chromatogram of fatty acids' methyl esters of olive oil

Recent Publications

1. Jain A, Verma KK, (2011) Recent advances in applications of single-drop microextraction: A review. *Analytica Chimica Acta* 706:37– 65
2. Rezaee M, Assadi Y, Milani Hosseini MR, Aghaee E, Ahmadi F, Berijani S (2006) Determination of organic compounds in water using dispersive liquid-liquid microextraction. *J. Chromatogr. A* 1116:1–9.
3. Yao C, Anderson JL (2009) Dispersive liquid-liquid microextraction using an *in situ* metathesis reaction to form an ionic liquid extraction phase for the preconcentration of aromatic compounds from water. *Anal. Bioanal. Chem.* 395:1491–1502.

4. IUPAC Method 2.301. International Olive Oil Council. COI/T.20/Doc.no. 24 2001
5. European Pharmacopoeia - 8th Edition (2013), Druckerei CH Beck, Nördlingen, Germany 136-138.

Biography

Aysel Berkkan has been an Associated Professor at Gazi University, Faculty of Pharmacy, Division of Analytical Chemistry in Turkey since 2014. She received her PhD. degree in 2004 at Gazi University, Faculty of Pharmacy, Division of Analytical Chemistry about Hydride Generation Atomic Absorption Spectrometry with Professor Nusret Ertas. She has been an Assistant Director in the Institute of Medical Sciences at Gazi University in 2016. Dr Berkkan's reseachs involve Hydride Generation Atomic Absorption Spectroscopy, Inductively Couple Plasma- Mass Spectrometry, and Head Space Gas Chromatography.

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