Analysis of fatty acids in a cooking oil using an On-line micro Reaction Sampler

[Background] Reactive pyrolysis using tetramethyl ammonium hydroxide (TMAH) is useful for fatty acids analysis; however, unsaturated fatty acids may isomerize during the reaction with TMAH.[1)] Fatty acids derivatized using boron trifluoride (BF_3) in methanol is commonly used; however, it is a cumbersome procedure and the reagent is known to be toxic to humans. There is a third technique that can be used to derivatize fatty acids. The reaction of fatty acids with methanol at high temperatures and high pressures will results in the formation of fatty acid methyl esters (FAMEs).[2)] The analysis of FAMEs in cooking oil using the On-line micro Reaction Sampler is presented.

[Experimental] The analysis system consisted of a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the split/splitless injector of a GC/MS system. Sample prep.: 5 µL of hexane solution of a commercially available cooking oil (1 µg/µL) was added to a glass capsule and the solvent was allowed to evaporate. Then 5 µL of methanol was added to the capsule and the capsule was flamed sealed. After the reaction (at 250ºC for 10 min) was complete, the capsule was fitted to the Sampler and the Sampler attached to the Pyrolyzer. Pushing the Sampler down loweres the capsule into the pyrolyzer furnace (60ºC). After shattering the capsule, the furnace temperature was rapidly increased to 250ºC to flush the reaction products onto the GC separation column.

[Results] A chromatogram of fatty acids methyl esters in a cooking oil obtained by this method is shown in Fig. 1. Methyl esters of constituent fatty acids such as methyl palmitate (16:0), methyl stearate (18:0), methyl oleate (18:1), methyl linoleate (18:2), and methyl linolenate (18:3) were clearly observed.

The fatty acids composition ratios and the associated precisions (%RSD) using BF3-methanol derivatization and the on-line Reaction Sampler methods are comparable – see Table 1. The composition ratios obtained by both methods are also comparable. The formation of FAMEs using the On-line micro Reaction Sampler which combines the edible oil and methanol at high temperature and high pressure may be a viable alternative to reagent-based derivatization of fatty acids in edible oils.

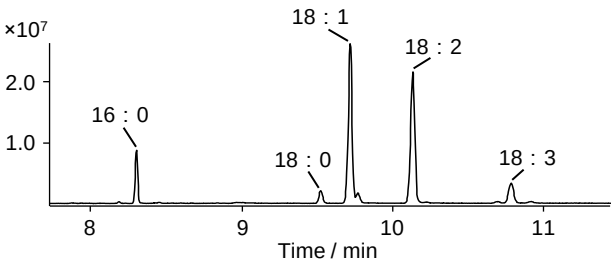

Fig. 1 Chromatogram of fatty acids methyl esters in a cooking oil using the on-line Reaction Samper

Column.: Ultra ALLOY⁺-CW (PEG) L: 30 m, id.: 0.25 mm, df.: 0.25 µm
GC oven temp.: 60 (3 min) - 220ºC (50ºC/min, 7.8 min hold)
GC inj temp.: 220ºC, column flow rate: 1 mL/min He, split ratio: 1/100
Rxn condition, 250ºC (10 min), Reagent: methanol (0.01 wt% NaOH)
capsule inner pressure: ca. 7.1 MPa

Table 1 Compositions of fatty acids (%)

	16:0	18:0	18:1	18:2	18:3	Total
Offline methylation (BF_3)*	9.09	3.00	39.7	40.3	7.88	100%
RSD% (n = 5)	0.70	0.71	0.70	0.44	1.40	
Reaction Sampler	9.61	3.28	41.8	38.2	7.15	100%
RSD% (n = 5)	2.90	0.51	0.32	0.59	1.88	

*1 mL of BF_3 methanol solution (10 wt%) added to 1 mg of a cooking oil, and heated at 75ºC for one hour. 1 µL of the hexane extract was injected into GC for analysis.

1) Y. Ishida et al., J. Anal. Appl. Pyrolysis , 49 (1999) 267-276
2) S. Saka et al., J. of Chem. Eng. Jpn , 34 (2001) No.3 383-387

Keywords : Reactive pyrolysis, TMAH, Closed system, Fatty acids, FAME, compositional analysis

Products used : Multi-functional pyrolyzer, On-line Micro Reaction Sampler, Vent-free GC/MS adapter

Applications : Biomass, Triglyceride, Polymer

Related technical notes : PYA2-029E

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