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Gas Chromatographic analytical method for the analysis of oxygenated polar fatty acids

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Introduction

Lipid oxidation is one of the major reactions that affect food quality and safety. Some of the compounds produced like polar fatty acids have health hazards to humans. In a food, lipid oxidation is measured by monitoring a number of biomarkers. Polar fatty acids (epoxy, oxo and hydroxy) are some of the biomarkers that can be used to monitor lipid oxidation. However, their analysis is challenging due to their close similarity in behaviour which leads to co-elution during chromatographic analysis thus giving complex chromatograms. A method based SPE fractionation followed by GC- FID/MS has been developed and can be used to analyse these compounds.

Materials and methods

Fatty acids were methylated using sodium methoxide in TBME followed by SPE fractionation on silica gel (10 % moisture content) using a hexane: diethyl ether mixture with increasing polarity (98:2, 90:10 and 70:30 % v/v) procedure as described by Mubiru et al., (2013). Methyl nonadecanoate was used as the internal standard.

The resultant FAMEs were identified by GC-MS and quantified on GC-FID coupled with a polar FAME CP-Sil 88 capillary column. Quantification was based 1.04 as a response factor.

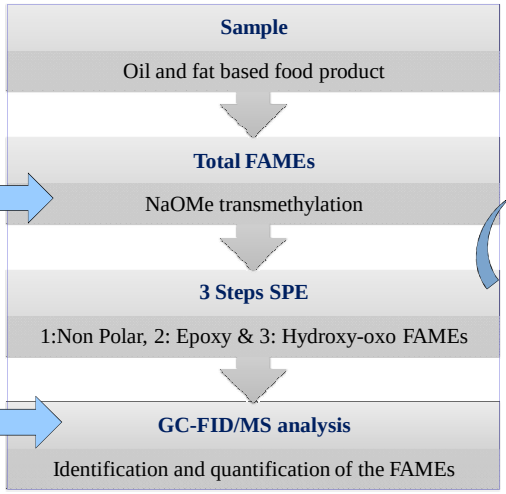

Fig.1. Flow diagram of the analytical procedure for the analysis of polar fatty acids.

Fig. 2. SPE set up for fractionation of polar fatty acids.

Results and discussion

Results after SPE fractionation on silica and injection on GC-FID showed that less complex chromatograms could be obtained for the fraction which had more of epoxy compounds **(Fig.3A).** Fraction three which had more of hydroxy fatty acids could only be analysed after silylation with BSTFA and 10 % TMCS at room temperature **(Fig.3B).**

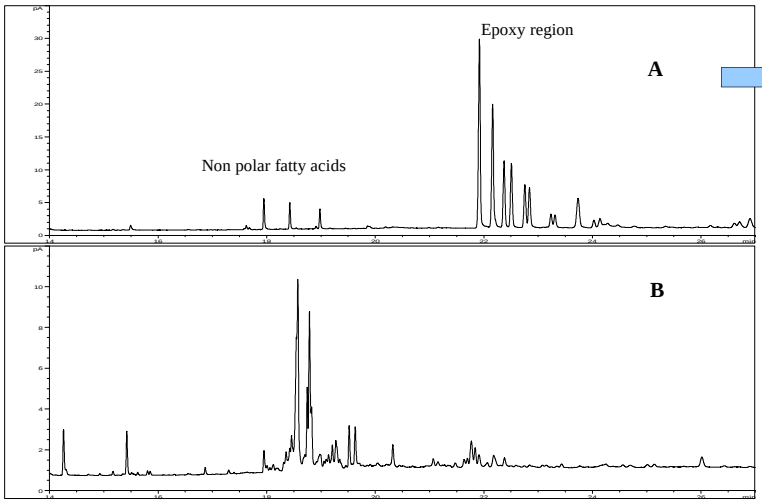

Table 1. Total epoxy fatty acids obtained from fresh vegetable oils purchased from Belgian supermarkets

Type of oil	Number of samples (N)	Total epoxy fatty acid composition (µg g^{-1} of oil)			PV range (mequiv. O_2 kg^{-1})
		Minimum	Mean	Maximum	
Arachid	2	4.6	747.8	685	1.5 – 2.4
Colza	5	0.4	74	67	2.3 – 4.4
Corn	5	3.8	293.5	161.9	0.0 – 3.1
Olive	7	0	214.4	221.5	5.2 – 12.6
Mixture	4	2	169.3	109.6	1.7 – 4.5
Sunflower	5	4.2	1448.2	1434	1.8 – 4.8
Frying	9	1.6	449.1	576.1	2.2 – 5.3

The method can be an effective tool for trace analysis and may be useful in exposure assessment studies.

The elution profile of the hydroxy-oxo fatty acids **(Fig.3B)** can further be simplified by hydrogenation which reduces the number of compounds allowing better chromatographic interpretation.

Conclusions

A simple method was developed which allows to quantify epoxy fatty acids in oils and foods. Preliminary screening of oils revealed quite high concentrations of these potentially hazardous substances in fresh oils obtained from Belgian supermarkets.

Reference: Mubiru, E.; Shrestha, K.; Papastergiadis, A.; De Meulenaer, B., Improved gas chromatography-flame ionization detector analytical method for the analysis of epoxy fatty acids. J Chromatogr A 2013, 1318, (0), 217-225

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