



Contents lists available at ScienceDirect

Journal of Food Composition and Analysis

journal homepage: www.elsevier.com/locate/jfca

Original research article

Improved analysis of olive oils triacylglycerols by UHPLC-charged aerosol detection

P. Lucci^{a,*}, S. Moret^a, F. Buchini^a, G. Ferlat^b, L. Conte^a^a Department of Agri-Food, Animal and Environmental Sciences, University of Udine, via Sondrio 2/a, 33100 Udine, Italy^b Thermo Fisher Scientific, Strada Rivoltana, 20090 Rodano, Milano, Italy

ARTICLE INFO

Keywords:

Olive oil
Charged aerosol detector
Food analysis
Food composition
Triacylglycerol
Trilinolein
Liquid chromatography
Authenticity

ABSTRACT

In the present study, a novel ultra high-pressure liquid chromatography method using charged aerosol detector (UHPLC-CAD) with an inverse-gradient solvent compensation has been developed for the analysis of triacylglycerols (TAGs) in extra-virgin olive oil. Using a gradient method with a superficially porous particles column, an improved separation of TAGs was obtained, with notably shortening of both solvent consumption and analysis time, in comparison with the IOC official method for ECN 42 analysis. Furthermore, an almost baseline separation of trilinolein was obtained. CAD provides good linearity ($R^2 > 0.999$) in the range of 0.05–10 μg of trilinolein injected on column as well as uniformity of response factor for different TAG molecular species. Finally, the proposed UHPLC-CAD methodology has been successfully applied to the determination of trilinolein in low-linoleic acid extra-virgin olive oil mixed in different proportions with high-oleic sunflower oil, palm olein and a mix of them at different percentages of 2, 4, 6, 8 and 10% (w/w). Good linearity was achieved in all the three blends with correlation coefficients greater than 0.975 in all cases.

1. Introduction

The expansion of the market of vegetable oils, made necessary authentication from both a commercial and health perspectives (Aparicio and Aparicio-Ruiz, 2000). Natural plant oils, including olive oils, are complex mixtures of various non-polar compounds such as triacylglycerols (TAGs) that represents the 90% of the matrix (Lisa et al., 2007) and other minor species such as diacylglycerols (DAGs), free fatty acids (FAs), phospholipids, phytosterols and tocopherols (Boskou, 1998; Buchgraber et al., 2004), among others. Each TAG is chemically characterized by its total number of carbons (CN), the degree of unsaturation of each FA, and the position and configuration of double bonds of each FA (De la Mata-Espinosa et al., 2011a; De la Mata-Espinosa et al., 2011b). The esterification position of FAs in the glycerol skeleton of TAGs is not random (Martínez-Force et al., 2009). Normally unsaturated FAs (no longer than 18 carbon chains) occupy the 2nd position of TAGs derived from vegetable oils (Beccaria et al., 2016). The different TAGs profile of each vegetable oil and the percentage of different FAs in the TAGs mixture are influenced by various factors such as botanical origin, climate growth zone and olives maturation stage.

Due to the importance of extra-virgin olive oil (EVOO) and virgin olive oil (VOO) as ingredients in the Mediterranean diet (Bach-Faig et al., 2011), they can be rather expensive products and, therefore,

fraud is frequent. For instance, EVOO is often diluted with other olive oils of inferior quality or with cheaper vegetable oils (Filmore, 2004). Within this context, the investigation of fraud play a key role in authentication of genuine products, hence, many analytical methods have been introduced by the main international institutions involved in oil standards (European Union – EU – International Olive Council – IOC – and the Codex Alimentarius). The analysis of TAGs, earlier as trilinolein, then as Delta ECN42 was in the European Regulation since its first release in 1991 (European Commission Regulation, 1991) and maintained in every following amendment.

From an analytical point of view, TAGs mixture from plant oils is very complex. Several instrumental techniques have been applied for TAGs analysis including gas-liquid chromatography (GLC) and high-performance liquid chromatography (HPLC), with the latter being the most widely used approach. A number of papers focused on olive oil TAGs have already been published describing both non-aqueous reversed-phase HPLC (NARP-HPLC) and normal-phase HPLC with silver ion embedded columns (Ag-HPLC) (Buchgraber et al., 2004). With regard to reversed-phase HPLC, columns packed with chemically bonded C18 phases having particles with diameters of 3–5 μm are frequently used. Mobile phase affects the degree of separation and the choice of the most suitable detector. One of the most widely employed solvent system is a mixture of acetonitrile with acetone (in an adjustable

* Corresponding author.

E-mail addresses: paololucci2001@yahoo.it, paolo.lucci@uniud.it (P. Lucci).<https://doi.org/10.1016/j.jfca.2017.12.028>Received 31 August 2017; Received in revised form 13 December 2017; Accepted 15 December 2017
0889-1575/ © 2017 Elsevier Inc. All rights reserved.

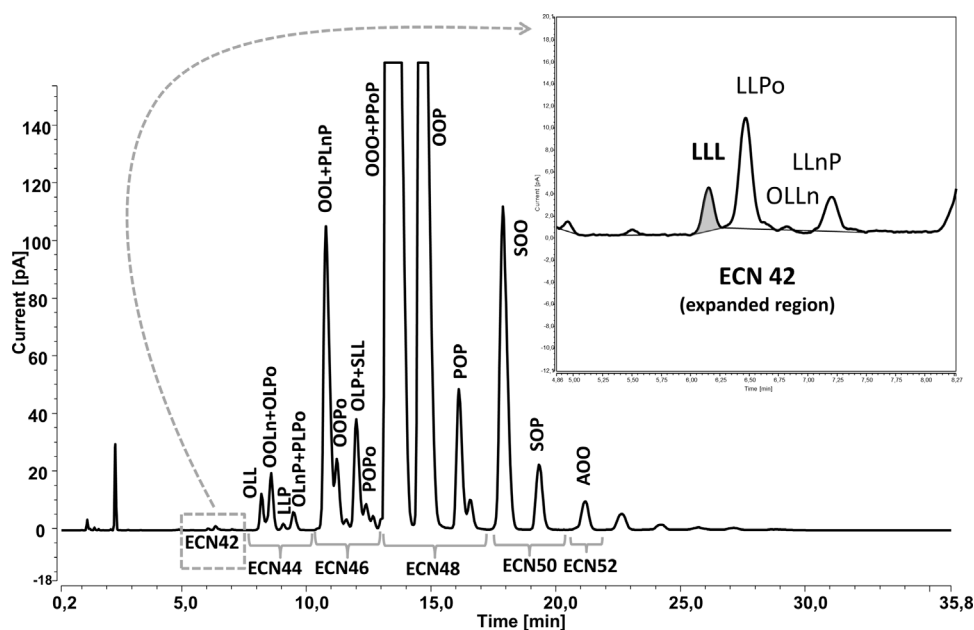


Fig. 1. UHPLC-CAD chromatogram of EVOO with low linoleic content. In the box, it is reported the expansion of the ECN 42 peak group. TAGs were nominated using the initials of FAs trivial names; the lists of acronyms used for FAs were as follow: P = (C16:0) palmitic acid; Po = (C16:1) palmitoleic acid; S = (C18:0) stearic acid; O = (C18:1) oleic acid; L = (C18:2) linoleic acid; Ln = (C18:3) linolenic acid; A = (C20:0) arachidic acid; ECN = equivalent carbon number (International Olive Council, 2001, 2013).

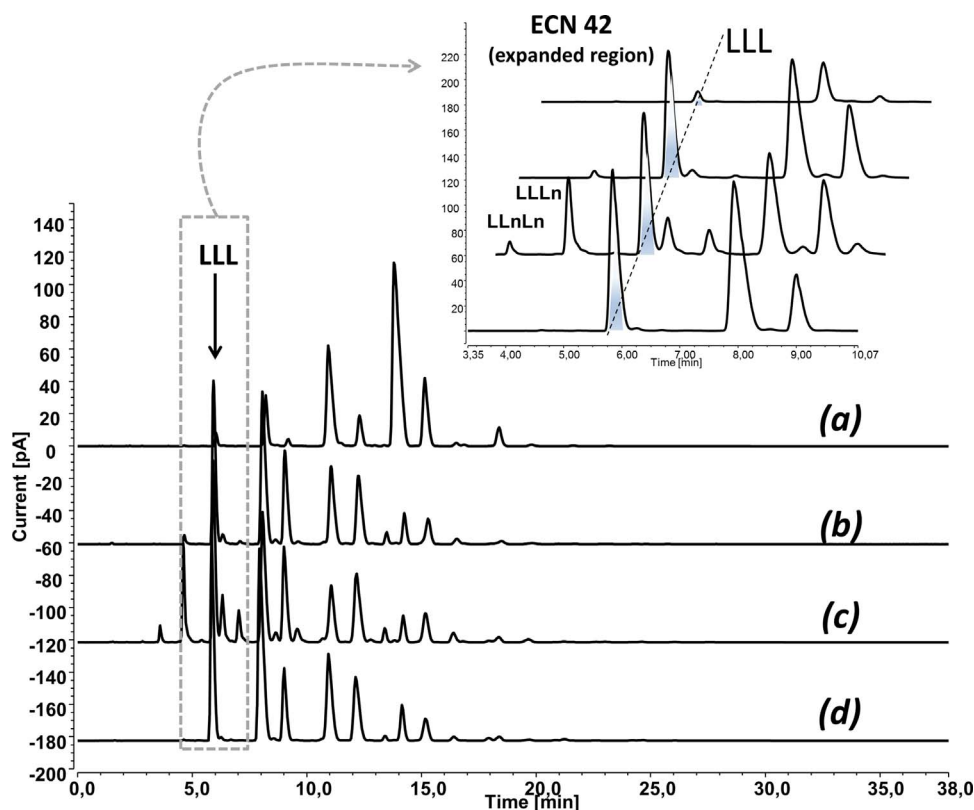


Fig. 2. Chromatograms showing the TAGs profiles of hazelnut (a), corn (b), soybean (c), and sunflower (d) oils using UHPLC-CAD. In the box, it is reported the expansion of the ECN 42 peak group. TAGs were nominated using the initials of FAs trivial names; the lists of acronyms used for FAs were as follow: L = (C18:2) linoleic acid; Ln = (C18:3) linolenic acid; ECN = equivalent carbon number (International Olive Council, 2001, 2013).

proportion, usually 50:50, v/v) that allows the separation of unsaturated species, interacting with the double bonds and the polarity of TAGs. Depending on the compatibility with the HPLC system and the detection mode, other mobile phases have been proposed such as acetonitrile-isopropanol, acetonitrile-dichloromethane, acetonitrile-chloroform, propionitrile, acetonitrile-ethanol-hexane or acetonitrile-isopropanol-hexane (Aparicio and Aparicio-Ruiz, 2000; Lisa et al., 2007; Moreda et al., 2003). However, some of these mixtures are no longer used because of their toxicity and their environmental impact. But the most critical aspect of the whole analytical methods for TAGs analysis is probably represented by the detection system and the

possibility of using gradient elution. Several different detectors have been employed to analyze TAGs by means of HPLC. And despite the advantages offered by mass spectrometry techniques for the differentiation of individual TAGs in different oils (Indelicato et al., 2017), the most common detectors used in routine analysis are still ultraviolet-visible spectroscopy (UV-vis), refractive index (RI) and evaporative light scattering (ELSD), although they all exhibit some analytical limitations. For instance, UV detector provides a good linear response, but it has low sensitivity to saturated TAGs and gives very different response factors between saturated and unsaturated ones (Holcapek et al., 2005). ELSD detector has been widely studied, however, it was shown

Download English Version:

<https://daneshyari.com/en/article/7619973>

Download Persian Version:

<https://daneshyari.com/article/7619973>

[Daneshyari.com](https://daneshyari.com)