A rapid and efficient method for dialkyl ketones and sterols determination in fat

Steven Mascrez[1], Sabine Danthine[2], Giorgia Purcaro[1]
[1] Analytical Chemistry Lab,; [2] Department of Food Science and Formulation,
Gembloux Agro-Bio Tech, University of Liège, Gembloux, 5030, Belgium

Presented at the 2021 AOCS Annual Meeting & Expo ONLINE

Introduction

The present work deals with the optimization of a fast method for the determination of dialkyl ketones (DAKs) in interesterified fats. Microwave-assisted saponification was optimized, followed by purification on a lab-packed silica solid-phase extraction (SPE) cartridge. The method proposed may be used for the determination of DAKs or both DAKs and sterols by simply eluting an additional fraction from the SPE cartridge. The final determination was performed by gas chromatography- flame ionization detector (GC-FID) for quantification and gas chromatography-mass spectrometry (GC-MS) for confirmation purposes. The proposed method showed good recoveries (>80%) and limit of quantification (0.04-0.07 µg/g for the 4 standard DAKs and of 0.07 µg/g for α-cholestanol). Repeatabilities (n=3) were below 15% for DAKs and generally lower than 6% for sterols. Accuracy on the entire sterol profile was confirmed in comparison to the International Olive Council reference method. The method was finally applied to real-world samples before and after chemical interesterification.

Material and Methods

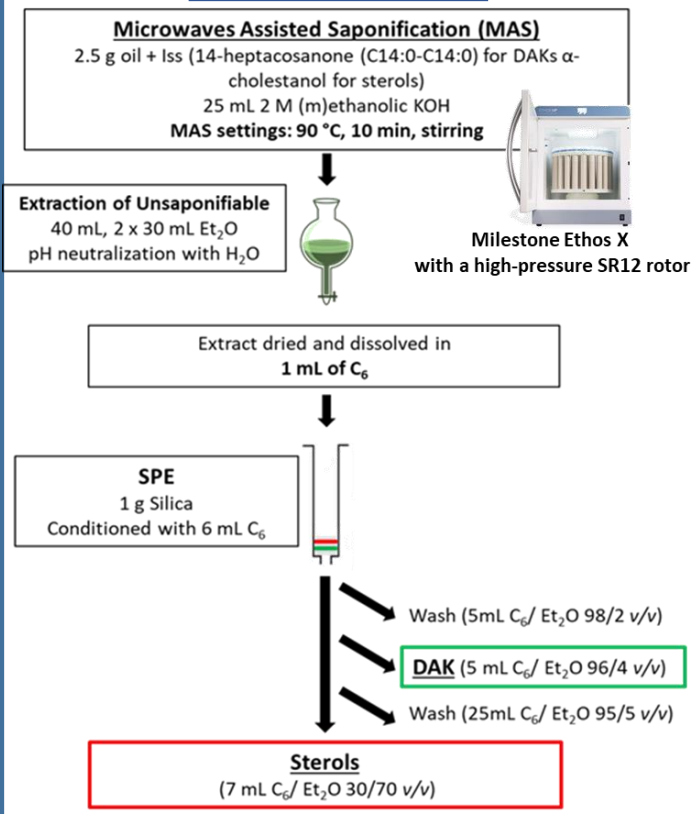

***Figure 1**: Scheme of the sample preparation procedure.*

DAKs

The DAK profile was studied using GC-MS before quantification with GC-FID. Identification of the different DAKs was not possible based only on the NIST Database; therefore, additional information was obtained by a careful evaluation of the MS fragmentation and by the MS data and elution order. **Figure 2** depicts the profile of the chemically interesterified rapeseed oil along with the MS spectra of all the DAKs detected.

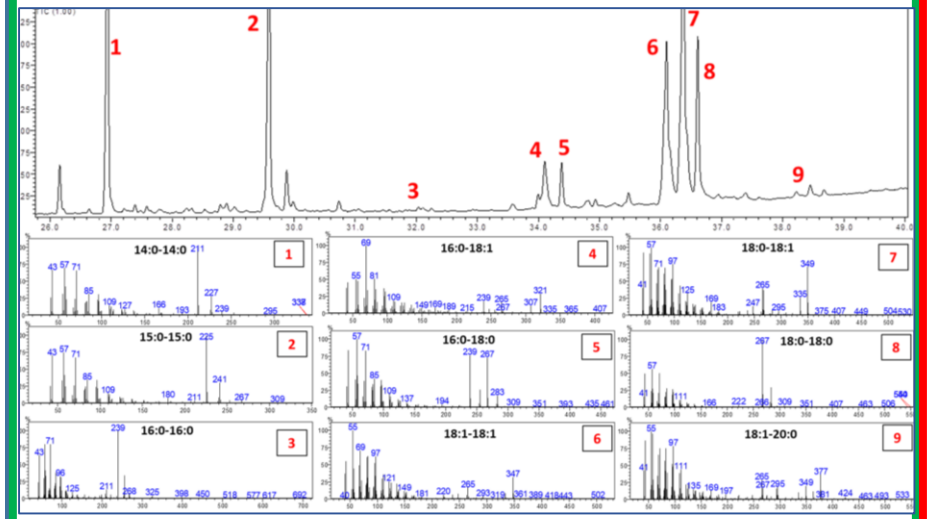

***Figure 2** : GC trace of the chemically interesterified (CIE) rapeseed oil. The MS spectra of each of the identified peak are reported labeled with the same number of the corresponding peak and reporting the identification of the DAK.*

Sterols

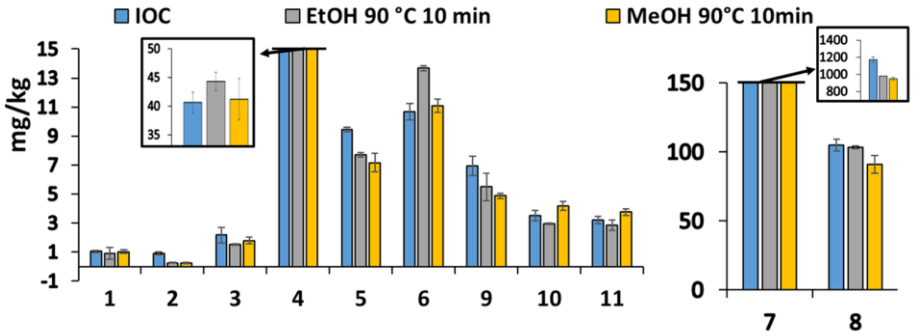

***Figure 3** : Barplot of the sterols from olive oil using the IOC method and the optimized method using MeOH or EtOH for saponification.*

This research was carried out during the first Covid-crisis, therefore to face the shortage of EtOH, MeOH was tested and used providing it gave similar performance (**Figure 3**). The sterols profile was evaluated before and after CIE (**Figure 4**).

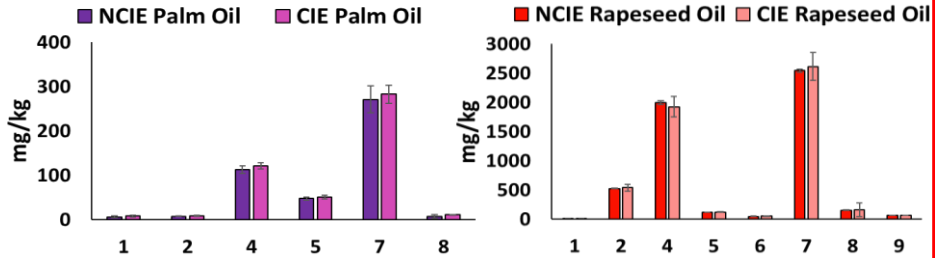

***Figure 4**: Sterols content of no chemically interesterified (NCIE) and chemically interesterified (CIE) of palm oil and rapeseed oil*

1: Cholesterol; 2: Brassicasterol; 3: 24-Methylene-cholesterol; 4: Campesterol; 5: Stigmasterol; 6: Clerosterol; 7: β-Sitosterol; 8: Δ-5-Avenasterol; 9: Δ_5-24-Stigmastadienol; 10: Δ-7-Stigmastenol; 11: Δ-7-Avenasterol

Acknowledgement
We thank Milestone and Shimadzu for their supports.

References

[1] S.Mascrez, S. Danthine, G. Purcaro, Foods, 2021, 10, 445.