Comparative evaluation of Microwave-assisted processes for fatty acid methyl esters analysis in food matrices

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The analysis of fatty acids can yield meaningful insights in many field of applications, such as clinical, forensic, and biological. In the particular case of food analysis profiling the fatty acids, in relation or not to the total fat content, can provide useful information regarding their impact on nutrition and health.

This kind of analysis may involve a first extraction step of the lipids from the sample matrix and subsequently transforming the fatty acids into fatty acid methyl esters (FAMEs) to profile them in the following gas chromatographic analysis. Many of the commonly used techniques may need long processing times and involve the use of hazardous chemicals, usually BF₃ for derivatization. Recently, we developed a method known as microwave-assisted extraction and derivatization (MAED) with the aim of reducing the sample preparation time. This approach uses acidic methanol, a less hazardous solvent, and has been shown to be equivalent to the official AOCS Ce2b-11, which involves a simultaneous alkali hydrolysis and methylation procedure.

In this study, we extended the comparison to the results obtained with the AOCS Ce2c-11 official method, which involves prior acid digestion followed by alkali hydrolysis and finally methylation (the last part as for AOCS Ce2b-11). These data were also compared with the common procedure in routine laboratories to perform an extraction, to determine the total fat content, and then continue with the derivatization for FAMEs profiling. Specifically, two different kinds of extractions supported by microwave technology were used: solvent extraction and extraction + hydrolysis. After that, both the extracts were derivatized using i) BF3 and ii) an acidic methanol solution in the microwave device. Overall, seven distinct procedures were applied to seven distinct food samples —including dairy, meat, and ready-to-eat foods.

In the end, a reversed fill/flush flow modulation comprehensive multidimensional gas chromatography (GC×GC-FID) was used to identify and quantify the FAMEs, and AGREEPrep measures were used to evaluate all the single methods from both greenness and FAMEs profile perspectives.