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Microwave-integrated extraction of total fats and oils

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ABSTRACT

An improved process of Soxhlet extraction assisted by microwave, called microwave-integrated Soxhlet (MIS) is proposed for the extraction of oils and fats from different food matrixes such as oleaginous seeds, meat and bakery products. Optimal conditions for extraction were obtained using a response surface methodology and reached from a central composite design allowing us to conclude in a previous paper that the proposed process ensures complete, efficient and accurate extraction for lipids determination from olives. In this paper, the peak areas of the main fatty acids extracted with MIS from olive seeds were considered as response variables and submitted to an analysis of variance in order to determine if there was a significant link between the extraction of fatty acids and the variables required in extraction procedures. Results have shown that MIS parameters do not affect the composition of the extracts. For the generalization of the study with several food matrixes, MIS extraction results obtained were then compared to conventional Soxhlet extraction in terms of crude extract and fatty acid composition and shown that the oils extracted by MIS were quantitatively and qualitatively similar to those obtained by conventional Soxhlet extraction. MIS labstation can be considered as a new and general alternative for the extraction of lipids by using microwave energy.

1. Introduction

In analytical laboratories, Soxhlet is probably the most-used method for extraction of fats and oils from food matrices. The desired compounds are extracted by an iterative percolation of a fresh solvent, generally "*n*-hexane". After the extraction, the major solvent is eliminated in a vacuum rotary evaporator. The design of Soxhlet technique was first described by Franz von Soxhlet in 1879 for the fat determination in milk [1]. Much work has been done to improve the Soxhlet extraction to decrease the operation time and solvent use, and to eliminate the need for evaporation and concentration at the end of the extraction [2]. An important improvement of the Soxhlet extraction device has been developed by Randall who proposed a three-step extraction namely: boiling, rinsing and solvent removal [3].

Microwave energy, with a frequency of 2.45 GHz, is well known to have a significant effect on the rate of various processes in the chemical and food industry. Much attention has been given to the application of microwave dielectric heating in analytical chemistry because of the reduced analysis time, simplified manipulation, and higher purity of the final product. Several classes of compounds such as fats and oils, essential oils, aromas, pesticides, phenols, dioxins and other organic compounds have been extracted efficiently from a variety of matrices (mainly soils, sediments, animal tissues, food or plant materials). All the reported applications have shown that microwave-assisted solvent extraction (MAE) is a viable alternative to conventional techniques for such matrixes. The main benefits are the reduction of extraction time, energy and solvent used [4–6].

There are only few articles in the literature that have reported the acceleration of fats and oils extraction by microwave irradiation. The advantages of using microwave energy as a non-contact heat source for the extraction of analytes from plant materials include: more effective heating, faster energy transfer, reduced thermal gradients, selective heating, reduced equipment size, faster response to process heating control, faster start-up, increased production, and elimination of process steps [7].

In 1998, Luque de Castro et al. developed a new extraction technique called focused microwave-assisted Soxhlet extraction (FMASE), which uses two sources of energy, namely microwaves (applied on the extraction chamber of a modified Soxhlet) as auxiliary energy to accelerate the process and electrical heating (applied on the distillation flask). However, these authors have reported that the maximum recovery for oils and fats is obtained when the moisture content of the sample is between 20 and 90%,

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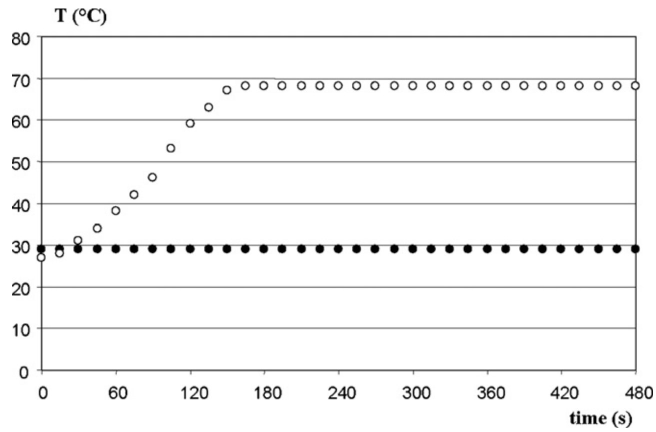

Fig. 1. Temperature evolution of n-hexane as a function of time: (●) n-hexane + 600 W, (○) n-hexane + Weflon + 600 W.

preferably about 30%. When using dried samples, the recovery yield drops dramatically [2,8–14].

Recently, an original process for extracting oils and fats by using microwave energy has been developed in our laboratory [15]. Based on a relatively simple principle, this process involves the use of a polytetrafluoroethylene/graphite compound (Weflon, Sorisole, Bergamo, Italy) in the form of a stir bar with a new concept of Soxhlet assisted by microwaves. Weflon allows diffusion of heat created by the microwaves to the surroundings and is particularly useful in the case of transparent solvent to microwave radiations such as hexane (Fig. 1). MIS extraction combines the advantages of the extraction performed with Soxhlet (extraction repeated by a pure solvent) and the heating by microwaves (reduction of extraction time). The process ensures complete, rapid and accurate extraction of the samples. No extra heat generator is required; dry (whatever the percentage of dryness) as well as wet material may be extracted. Moreover, the apparatus concentrates the final extract and thus eliminates the evaporation step under reduced pressure. A second-order central composite design (CDD) has been used to investigate the performance of the device. MIS technology has appeared as a good alternative for the extraction of fats and oils from olive seeds.

The aim of the present study was to investigate MIS ability to extract fats and oils from an expanded group of foodstuff. Sunflower seeds and peanuts were studied in the oleaginous seeds group. Fatty acids were then extracted from beef to represent meat products. The same extractions were also performed on croissant to study the bakery products. Optimal conditions obtained using a response surface methodology (RSM) and reached from the CCD from previous experiments [15] were used as settings for the generalization study. The peak areas of the main fatty acids extracted in various experiments of the CCD were also considered as response variables. MIS extracts obtained in various experiments have been compared with those provided by conventional Soxhlet extraction in terms of quantitative (yield) and qualitative (fatty acid composition) studies for the different food matrixes. To complete the study, this paper gives an overview of current knowledge on the mechanism of MIS extraction.

2. Experimental

2.1. Chemicals and materials

n-Hexane and n-heptane used for extraction experiments and fatty acids derivatives preparation respectively were all of analytical grade and were purchased from VWR International (Darmstadt, Germany). BF_3-methanol reagent (20% solution in methanol), NaOH and NaCl of analytical grade, used for fatty acids derivatives preparation, were also supplied by VWR International.

2.2. Samples studied

Olives *Aglandau* (Vaucluse, France) samples were collected from local oil mill. Foreign materials such as leaves, earth or stones were then removed from olives samples before being randomized in plastic flask and stored at −18 °C until use. Sunflower seeds, peanuts, croissants and beef steaks were purchased from local grocery store. Sunflower seeds and peanuts were kept in their own safety pocket and stored in dark until use. Croissants and beef steaks were randomized in plastic flask before storage at −18 °C until use.

2.3. Determination of the moisture and volatile matter content

In order to respect the official procedure [16], samples were dried before extraction and ground for less than 0.5 h before investigations. 10 g of samples weighed to the nearest mg were placed in an electrical oven at 80 °C. Samples were then weighed each 2 h after cooling in a desiccator until the ratio (m/m, %) was less than 10%. The average moisture contents found for each samples are listed in Table 1.

2.4. Extraction procedures

Both conventional Soxhlet extraction and MIS extraction procedures have been carried out as described in previous investigation [15].

For convention Soxhlet extraction, 30 g of dried and ground samples were weighed to the nearest 10 mg of sample. The amount was transferred in a cellulose thimble and placed after in the extraction chamber. The Soxhlet apparatus, fitted with a condenser, was placed on a distillation flask containing 300 mL of solvent. Samples were thus extracted under reflux with n-hexane during 4 h. Thereafter, the cellulose cartridge was cooled to room temperature in a desiccator and its content was then milled before being transferred again in the thimble. The described procedure was thus repeated during 2 h until a total extraction of 8 h (4 h + 2 h + 2 h). The content of the distillation flask was then concentrated to dryness with a vacuum rotary evaporator and the flask was then cooled to room temperature in a desiccator and weighed to the nearest mg.

For MIS extraction (Fig. 2), 30 g of dried and ground samples were weighed to the nearest 10 mg. Weflon particles were placed in the base vessel and a Teflon filter support was added. The sample was loaded onto the support and 300 mL of hexane was added in order to immerse the sample. Then, the base vessel was placed in

Table 1
Moisture content of the samples used in various experiments

	Sample studied				
	Olive seeds	Sunflower seeds	Peanuts	Beef steaks	Croissants
Moisture content (%)	55.5	4.0	3.6	68.7	19.0