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New microwave-integrated Soxhlet extraction An advantageous tool for the extraction of lipids from food products

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Abstract

A new process of Soxhlet extraction assisted by microwave was designed and developed. The process is performed in four steps, which ensures complete, rapid and accurate extraction of the samples. A second-order central composite design (CCD) has been used to investigate the performance of the new device. The results provided by analysis of variance and Pareto chart, indicated that the extraction time was the most important factor followed by the leaching time. The response surface methodology allowed us to determine optimal conditions for olive oil extraction: 13 min of extraction time, 17 min of leaching time, and 720 W of irradiation power. The proposed process is suitable for lipids determination from food. Microwave-integrated Soxhlet (MIS) extraction has been compared with a conventional technique, Soxhlet extraction, for the extraction of oil from olives (*Aglandau*, Vaucluse, France). The oils extracted by MIS for 32 min were quantitatively (yield) and qualitatively (fatty acid composition) similar to those obtained by conventional Soxhlet extraction for 8 h. MIS is a green technology and appears as a good alternative for the extraction of fat and oils from food products.

Keywords: Soxhlet; Microwave; Extraction; *n*-Hexane; Oil; Food

1. Introduction

In general, analytical procedure for oils or fats from food products comprises two steps: extraction (Soxhlet extraction with hexane as solvent) and analysis (gas chromatography with flame ionization detection or coupled to mass spectrometry). Whereas the last step is finished after 30 min to 1 h, extraction takes at least several hours. It is frequently done by the Soxhlet extraction method, based on an iterative percolation of a fresh solvent generally "*n*-hexane" [1,2].

The Soxhlet extraction technique was invented in 1879 by Franz von Soxhlet for the fat determination in milk [3]. Then, it has been generalized for extraction in agricultural chemistry before becoming the most used tool for solid–liquid extraction in many fields like environment [4,5], foodstuffs [6–8], and also pharmaceutics [9–11]. Nowadays, Soxhlet apparatus is still commonplace in laboratories and has been the standard and reference method for solid–liquid extraction in most cases [12]. Soxhlet extraction nevertheless has some disadvantages such as long operation time required (several hours), large solvent volumes, evaporation and concentration needed at the end of the extraction, and inadequacy for thermolabile analytes [13].

Many works have been done to improve Soxhlet extraction [13]. In 1974, Randall developed an improved Soxhlet extraction device, which proposed a three-step extraction, namely: boiling, rinsing and solvent removal [14]. It was labelled later with full-automation by different companies, under the name of Soxtec System HT (Gerhardt, Bonn, Germany) or Soxtherm (Foss, Eden Praire, MN, USA). Randall method was faster than classical Soxhlet method because the sample to be extracted was immersed in hot solvent. It induces an enhancement in mass transfer, an increased solubility and diffusion rate, and a better kinetic of desorption and solubilization [15]. Another recent alternative to improve on the technique has been proposed.

In 1998, Luque de Castro and co-workers developed a new extraction technique called focused microwave-assisted Soxhlet

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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) [16,17]. Different devices were designed and constructed, allowing each additional advantages such as automation, possibility of recovering most of the extractant, possibility of using water as extractant and selection of the extractant volume in each extraction cycle. This system has been used for the determination of the oil content and the fatty acid composition of oleaginous seeds [18,19], lipids from sausage products [20], fat from cheese [21] and bakery products [22]. However, it has been found that the moisture content of samples to be extracted is a defining parameter for the recovery yield, when carrying the methods of the previous art [19].

In view of the above, there is a need to provide an improved microwave-assisted extraction process, which overcomes at least some limitations of the known processes.

The aim of this work was to develop a new method, "microwave-integrated Soxhlet" (MIS), for the extraction of fats and oils. A Response Surface Methodology obtained from a multivariate study was used to investigate the performances of MIS and to study the relevance of factors required during operating extraction. Optimal parameters were then obtained from a second-order polynomial equation. The results then obtained were compared with those obtained by conventional technique, in order to introduce this advantageous alternative in the analytical procedure of lipids in food products. To investigate the potential of MIS, comparisons have been made with conventional Soxhlet extraction for the extraction of oil from olives (*Aglandau*, Vaucluse, France) in terms of extraction time, yield and fatty acid composition.

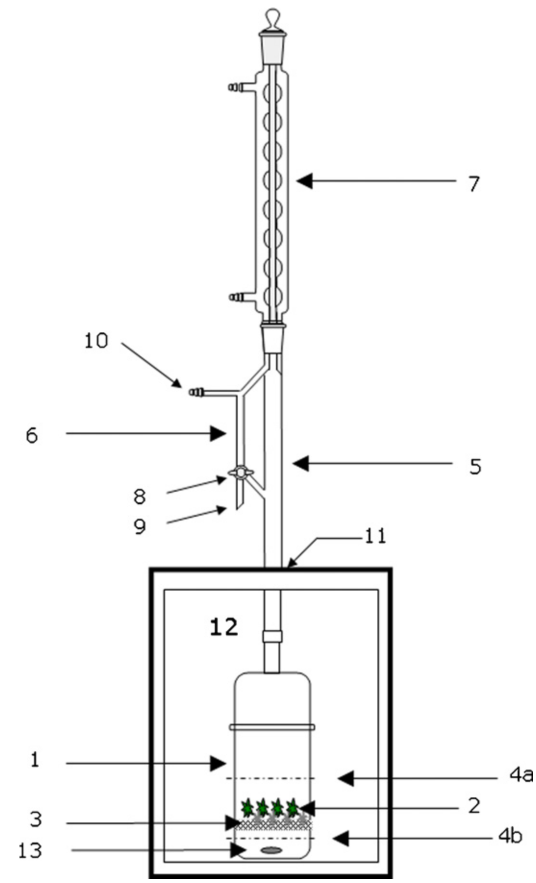

Fig. 1. The basic principle of the new process of Soxhlet extraction assisted by microwaves. 1: base vessel; 2: solid material; 3: support; 4a: *n*-hexane level immersing the sample; 4b: *n*-hexane level below the sample; 5: extraction tube; 6: side arm; 7: condenser; 8: 3-way valve; 9: side arm opening to collect *n*-hexane; 10: side arm opening to pull a vacuum in the system; 11: opening on upper surface of the microwave oven; 12: microwave oven; 13: Weflon magnetic stirrer.

2. Experimental

2.1. Reagents

n-Hexane and *n*-heptane of HPLC grade were supplied by VWR International (Darmstadt, Germany). Sodium chloride and sodium hydroxide of analytical grade were purchased from VWR International. BF_3-methanol reagent (20% solution in methanol for synthesis) was also supplied by VWR International.

2.2. Plant material

For the development of this research, olives *Aglandau* (Vaucluse, France) samples were collected from local oil mill and were removed from foreign materials such as leaves, earth or stones. Olives were then 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 ISO 659-1988 procedure [12], samples were ground less than 1.5-h before extraction procedures. Amounts of about 10 g of olive sample were placed in an electrical oven at 80 °C. Samples were then weighted each 2 h after cooling in desiccators until the ratio m/m (%) was less than 10%. Thereby, we found an average moisture of about 55%.

2.4. MIS apparatus and procedure

The basic principle of the new process of Soxhlet extraction assisted by microwaves is illustrated in Fig. 1. Microwave-integrated Soxhlet extraction has been performed in a Milestone ETHOS microwave oven. The multi mode microwave reactor having a twin magnetron (2 × 800 W, 2.45 GHz) with a maximum delivered power of 1000 W variable in 10 W increments. Time, temperature, pressure and power were controlled with the "easy WAVE" software package during experiments. Temperature was monitored by a shielded thermocouple (ATC-300) inserted directly into the sample container and by an infrared sensor outside the reactor. The base vessel is a traditional glass round-bottom flask. The flask (1) for containing the solid material is a flask suited for microwave reactions. The base vessel (1) forming part of the device contain a polytetrafluoroethylene/graphite stir bar capable of absorbing microwaves at the