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Review

Soxhlet extraction: Past and present panacea

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ABSTRACT

An overview of Soxhlet extraction, the advantages and shortcomings of this centenary technique as well as the attempts to improve its performance and achievements reached is here presented. Assistance of high pressure, ultrasound or microwaves has decreased or minimized the negative characteristics of the conventional extractor. Automation of Soxhlet performance opened the door to commercialization of a number of different approaches. The evolution of Soxhlet extractor is here critically discussed, and the conclusion from this overview is that the adoption of new technologies to improve its performance converts Soxhlet extraction in almost a "panacea" in this field.

Contents

1. Introduction

Sample preparation is most often a necessity as even the simplest samples are frequently unsuitable for direct analysis because of excessive dilution or concentration of the target analytes or incompatibility with instrument operation procedures. For these reasons, sample preparation is, most times, the bottleneck of analytical methodologies as it constitutes the principal source of error and remains as one of the most time-consuming steps [1], particularly with solid samples. Solid samples are the most difficult to process as most analytical instruments cannot handle them. Therefore, the first operation in the preparation of solid samples involves transferring the target analytes to a liquid phase.

Solvent extraction of solid samples, which is commonly known as "solid–liquid extraction" but should rather be named as "leaching" or "lixiviation" to more strictly adhere to its physical–chemical foundation, is one of the oldest techniques of solid sample preparation. It serves, not only to remove and separate compounds of interest from insoluble high-molecular-weight fractions, but also from other compounds that could interfere with subsequent steps of the analytical process. Classically, leaching has been widely carried out by maceration, based on the correct choice of solvents and the use of heat and/or agitation to increase the solubility of compounds and the rate of mass transfer. Despite the extensive use

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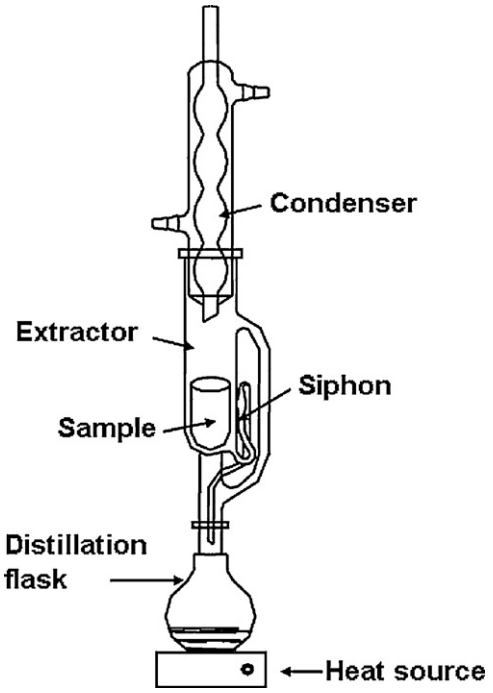

Fig. 1. Conventional Soxhlet extractor.

of maceration, particularly for isolation of natural products, this is characterized by long extraction protocols with low efficiency.

In 1879, von Soxhlet developed a new extraction system (Soxhlet extractor) which has for a long time been the most widely used leaching technique [2]. In fact, Soxhlet extraction has been a standard technique for over a century and the methods based on it remain the primary references against which performance in new leaching methods is measured. The advantages and shortcomings of Soxhlet extraction have been used as starting points for the development of a variety of modifications intended to alleviate or suppress the latter while keeping or even improving the former. Most of the modifications reported over the last few decades have been aimed at bringing Soxhlet closer to that of the more recent techniques for solid sample preparation, by shortening leaching times with the use of auxiliary energies and automating the extraction assembly.

The purpose of this review is both to outline the current position of Soxhlet extraction as a model to which the performance of other extraction techniques is referred and offer an overview about the evolution of this technique with a discussion about the different technical versions developed to accomplish a more competitive extraction technique.

2. Conventional Soxhlet extraction

In its classical implementation, which was originally used to determine fat in milk [2], the sample is placed in a thimble-holder that is gradually filled with condensed fresh extractant (term used to refer to the solvent used for extraction) from a distillation flask (see Fig. 1). When the liquid reaches the overflow level, a siphon aspirates the solute from the thimble-holder and unloads it back into the distillation flask, thus carrying the extracted analytes into the bulk liquid. This operation is repeated until extraction is complete. Operationally, Soxhlet extraction is thus a continuous–discrete technique. In fact, since the extractant acts stepwise, the assembly operated as a batch system; however, extractant is recirculated through the sample, so the system also operates in a continuous manner somehow.

Conventional Soxhlet extraction has some attractive advantages. Thus, the sample is repeatedly brought into contact with fresh portions of extractant, which facilitates displacement of the transfer equilibrium. Also, the system remains at a relatively high temperature by effect of the heat applied to the distillation flask reaching the extraction cavity to some extent. In addition, no filtration is required after leaching and sample throughput can be increased by performing several simultaneous extractions in parallel, which is facilitated by the low cost of the basic equipment. Moreover, Soxhlet extraction is a very simple methodology that requires little training, can extract more sample mass than most of the latest alternatives (microwave-assisted extraction, supercritical fluid extraction, etc.) and seemingly subject to no matrix effects – this assertion is not strictly true as seen when Soxhlet extraction is compared with supercritical fluid extraction of analytes strongly bound to their matrix [3]. There is a wide variety of official methods involving a sample preparation step based on Soxhlet extraction [4–8].

The most serious drawbacks of Soxhlet extraction as compared to other techniques for solid sample preparation are the long time required for extraction and the large amount of extractant wasted, which is not only expensive to dispose off, but also the source of additional, environmental problems. Samples are usually extracted at the solvent boiling point over long periods, which can result in thermal decomposition of thermolabile target species. Also, a conventional Soxhlet device provides no agitation, which would help to expedite the process. In addition, the large amounts of extractant used call for an evaporation–concentration step after extraction. Finally, the Soxhlet technique is limited by extractant and difficult to automate.

Conventional Soxhlet extraction, with its advantages and shortcomings, has been used as starting point for the development of a variety of modifications intended to alleviate or suppress the latter while keeping or even improving the former. Most of the modifications reported over the last few decades have been aimed at bringing Soxhlet closer to that of the more recent techniques for solid sample preparation, by shortening leaching times with the use of auxiliary forms of energy and automating the extraction assembly.

3. High-pressure Soxhlet extraction

Soxhlet extraction under a high pressure is achieved by placing the extractor in a cyclindrical stainless-steel autoclave [9] or by the use of either commercial or laboratory-made supercritical fluid-Soxhlet extractors [10]. The particularity of high-pressure Soxhlet extraction is that the extractants do not reach supercritical conditions. Examples of them can be low-boiling solvents or gases under normal pressure and temperature, but in liquid state under high pressure. The development of the Soxhlet process under high pressure (1000–1500 psi) should short the time required and reduce solvents consumption. High-pressure Soxhlet extraction has been used to isolate organochlorine pesticides and polychlorinated biphenyls (PCBs) prior to determination in certified potato, carrot, olive oil and lyophilized fish tissue samples. In this application, carbon dioxide was used as extractant medium at the peak popularity of this extractant. The extraction set-up was immersed in a thermostated bath with a cooling water (0 °C) pumping system to condense the extractant [11].

Another application was fractionation of low-molecular-weight polyethylene. In this study, liquid CO_2 was found to be a suitable solvent for the lowest molecular weight hydrocarbons but failed to solubilize hydrocarbons with molecular weights higher than C-40–C-50. Liquid pentane was found to be an effective solvent for hydrocarbons insoluble in liquid CO_2 [12].

Fig. 2. Soxtec® System HT equipment initially commercialized by Tecator.

The main drawback accompanying to these extraction systems is associated to their operational principles. The Soxhlet process should not be affected by its performance under high pressure, which adds an extra level of complexity and reduces the robustness of the extractors.

4. Automated Soxhlet extraction

Automation of Soxhlet extraction was initially implemented on the commercial equipment Soxtec® System HT (see Fig. 2), which provided substantial savings in time and extractant [13]. This apparatus uses a combination of reflux boiling and Soxhlet extraction (both assisted by electrical heating) to perform two extraction steps (boiling and rinsing), followed by extractant recovery. Exchange from one to other step is achieved by switching a lever. The Soxtec counterpart B811 extractor, able to perform the same steps as a Soxtec device, emerged to implement the possibility to operate also as a conventional Soxhlet apparatus. The overall performance of the B811 extractor is computer-controlled [14].

Similar systems, currently commercialized by Foss, are the automated Soxtec™ 2050, the semiautomated Soxtec™ 2055, or the more economic versions Soxtec™ 2043 and 2045. One other version is the SoxCap™ 2047 that includes an acid hydrolysis step in the operation protocol used for total fat analysis [15]. Based on the use of these devices there are about 80 thoroughly tested methods available in the form of Application Sub Notes within the agricultural, food and industrial sectors, ranging from total fat extraction in meat to extraction of PCB in soil and sludge. Soxtec Systems have been used in officially approved methods such as AOAC 2003.05 and 2003.06 (crude fat in feed, cereal grain and forage using diethyl-ether and hexane extraction methods), AOAC 991.36 (fat crude in meat and meat products), ISO 1444:1996 (free fat content in meat and meat products) or EPA 3541 (extraction of PCBs in soil and sludge). Despite the implementation of commercial extractors in reference analysis methods, their compact configuration does not improve the scarce versatility of the conventional Soxhlet device.

5. Ultrasound-assisted Soxhlet extraction

An extractor based on the physical–chemical principles of Soxhlet by taking advantage of ultrasound effects [16] was designed, constructed and applied by the authors' team to the extraction of total fat from oil seeds such as sunflower, rape and soyabean [17]. The approach uses the conventional Soxhlet glasware, but has the Soxhlet chamber accommodated in a thermostatic bath

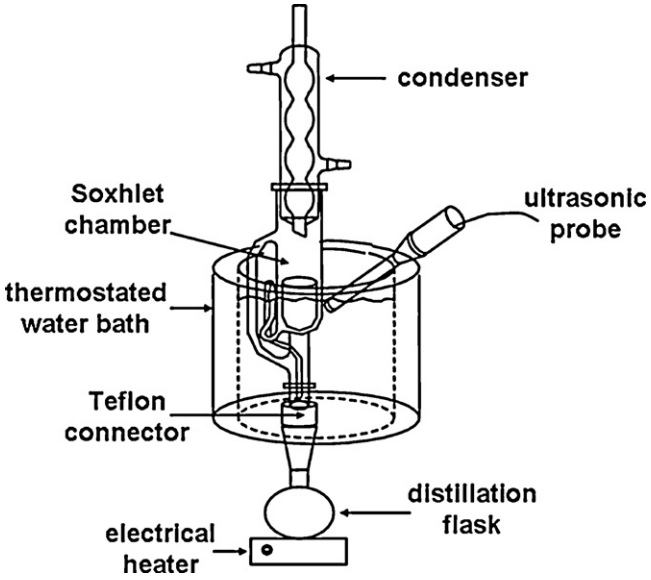

Fig. 3. Experimental set-up designed for ultrasound-assisted Soxhlet extraction (adapted from Ref. [17], reproduced with permission of Elsevier).

through which ultrasound is applied by means of an ultrasonic probe, as shown in Fig. 3. The application of ultrasound to the sample cartridge provides results similar to, or even better than, those obtained by conventional Soxhlet leaching (official ISO method); however, it enormously decreases the number of Soxhlet cycles needed in conventional procedures. But the most important result of ultrasound application is the decompaction effect it produces, which avoids typical steps of grinding several times between Soxhlet cycles to diminish the increased compactness produced by the dropping extractant. Despite the reported oxidative effect of ultrasound [18] under drastic conditions, the mild conditions used in this extractor do not degrade the extracted oil.

6. Microwave-assisted Soxhlet extraction

Between the attempts to improve Soxhlet performance, the most successful has been the use of microwaves, which has provided the wider variety of approaches. In fact, microwave-assisted Soxhlet extraction remains the most interesting improvement of conventional Soxhlet extraction.

Microwave-assisted Soxhlet extraction differs mainly in some or all of four aspects from other microwave-assisted extraction techniques, namely: (1) the extraction vessel is open, so it always works under normal pressure; (2) microwave irradiation is focused on the sample compartment; (3) the extraction step is totally or partially performed as in the conventional Soxhlet technique (*i.e.* with permanent sample–fresh extractant contact); (4) no subsequent filtration is required. Therefore, these approaches retain the advantages of conventional Soxhlet extraction while overcoming its limitations, as regards throughput, automatability and ability to quantitatively extract strongly retained analytes, as the most important.

6.1. Commercial microwave-assisted Soxhlet extractors

The most used commercial microwave-assisted extractor is the Soxwave-100 apparatus, which was patented and made commercially available by Prolabo (Paris, France). The principle behind the Soxwave-100 is similar to Kumagawa extraction and its operation similar to that of the Soxtec® System HT [13], the process involving extraction in three steps: a first step where the sample is immersed

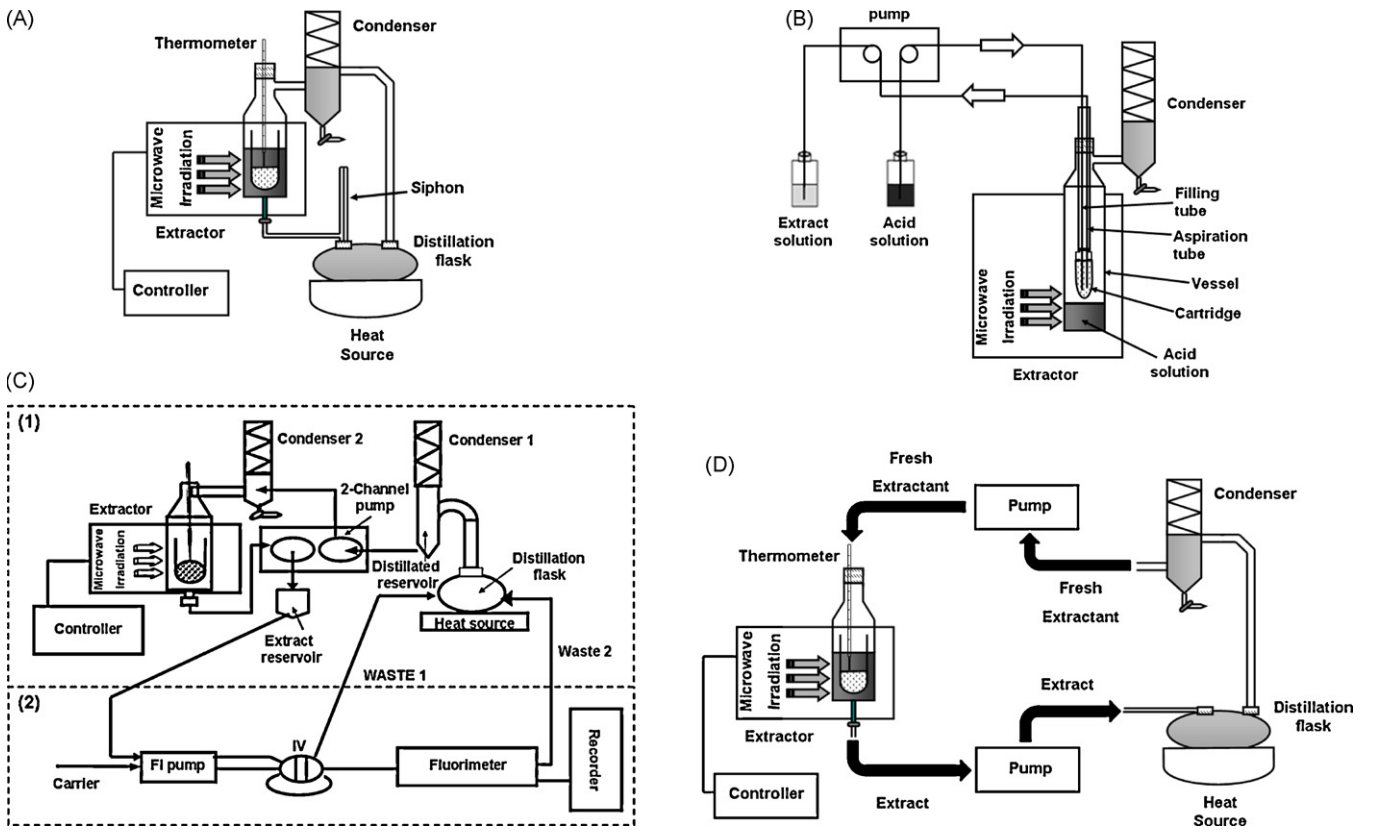

Fig. 4. (A) First FMASE prototype (adapted from Ref. [29], reproduced with permission of Elsevier). (B) Reverse configuration of the FMASE (adapted from Ref. [30], reproduced with permission of Royal Society of Chemistry). (C) Coupling of the extraction unit with a dynamic manifold to monitor the leaching process: (1) controlled FMASE; (2) flow injection manifold to interface extraction with detection (adapted from Ref. [31], reproduced with permission of American Chemical Society). (D) Complete automatic configuration of the first prototype (adapted from Ref. [32], reproduced with permission of American Chemical Society).

in the boiling extractant, followed by lifting of the cartridge over the solvent and continuous dropping of the condensate on the cartridge. In the first step, a matrix–extractant partitioning equilibrium of the extractable species is established while the microwave radiation acts on both the sample and extractant. In the second step, the partitioning equilibrium is displaced to extraction completion by effect of the sample coming into contact with fresh extractant in the absence of microwave irradiation.

The Soxwave-100 extractor uses a single heating source (*viz.* focused microwaves), which acts on both the sample and solvent. This fact makes the dielectric constant of the solvent used as extractant of paramount importance in this extractor; therefore, polar solvents are more efficient here than non-polar and low-polar solvents. Because the amount of energy required by the solvent is different from that required to remove the target analytes from the sample, a compromise must inevitably be made in this respect.

The Soxwave-100 has retained its original commercial design and its uses have been restricted to Prolabo "application sheets" (namely, environmental [19–21], polymer [22], drug [23] and food samples [24–26]).

It is worth mentioning here the attempts by the Chemat team to develop a microwave-assisted extractor (the name they give to the device is "microwave-integrated Soxhlet extractor"), which they consider to be similar to a Soxhlet extractor but in fact differs markedly from it in operational terms [27]. Thus, there is no contact of the sample with fresh extractant and no siphoning of the extract; also the extractant is heated by microwaves (similarly to the Soxwave-100), and a filtration step is required. Low-polar and non-polar extractants are heated to their boiling points by using microwaves while stirring with a Weflon magnetic stirrer to absorb microwave radiation. In this way, solvent vapours penetrate through the sample and are condensed on arrival at the condenser. Then, the condensate is dropped down onto the sample by adjusting a 3-way valve. Obviously, this operation is not based on the Soxhlet principle that exploits contact between the sample and fresh extractant in each leaching cycle; therefore, displacement of the partitioning equilibrium to complete extraction is impossible. Extraction must be inevitably followed by filtration in order to separate the remaining solid matrix from the extract. Despite the name used by the authors, the device does not integrate Soxhlet and microwaves. This extractor has been used to isolate lipids from foods [27] and oily seeds [28].

6.2. The focused microwave-assisted Soxhlet extractor (FMASE)

This extractor was designed by the authors' group and its first prototype was also constructed by Prolabo (see Fig. 4A). Contrarily to the Soxwave-100, the FMASE works like a conventional Soxlet apparatus; thus, it performs a series of cycles where the extractant is completely renewed but the sample is irradiated with microwaves for a preset time each cycle. It uses two energy sources (microwaves for sample irradiation and electrical heating of the extractant), which leads to the following behaviour: (i) extractant heating is non-dependent on the solvents polarity; (ii) the energy for solvent heating and that required to remove the target analytes from the sample can be optimized independently at each temperature; (iii) in FMASE, clean extractant and microwave irradiation are simultaneous, which facilitates mass transfer and shortens extraction times as a result [29].

Three prototypes have been designed and constructed since focused microwave-assisted Soxhlet extraction was first proposed as a sample preparation approach in 1998. The prototypes were

sequential improvements (particularly as regards efficiency and flexibility) over their previous incarnations. Therefore, each prototype had new advantages – and some disadvantages – over its older siblings. Below are described the most salient features of each.

6.2.1. First, simplest prototype: advantages and shortcomings

The prototype (Fig. 4A) was constructed by Prolabo (Paris, France) in 1998 and consisted of a modified Microdigest A301 focused microwave digestor (200 W maximum power) where a hole was made at the bottom of the irradiation zone to connect the cartridge compartment with the distillation flask through the siphon. This adaptation allowed the cartridge compartment of a conventional Soxhlet unit to be accommodated in the irradiation zone of the microwave oven. Operationally, the extractor is identical to a conventional Soxhlet apparatus except that it affords irradiation of the cartridge with focused microwaves for a preset time during each extraction cycle while fresh extractant (condensed vapours from the distillation flask) is dropped on and passed through the solid sample. In this way, breaking of analyte–matrix bonds is facilitated by application of the appropriated energy. A Prolab "Megal 500" thermometer was used to monitor the extraction temperature. Also, two controllers were used for the microwave unit and thermometer, and an electrical isomantle furnished with a rheostat was used as heating source for the distillation flask. The operational variables amenable to optimization in the FMASE are the irradiation power, irradiation time and number of cycles.

The device retains the advantages of conventional Soxhlet extraction while overcoming restrictions such as its long extraction times, non-quantitative extraction of strongly retained analytes – which is enabled by easier cleavage of analyte–matrix bonds by effect of interactions with focused microwave energy–, difficult of automation – which is made easier by replacing glassware with pumps– and the large volumes of organic solvent that are wasted. Unlike a conventional Soxhlet extractor, the microwave-assisted Soxhlet system allows up to 75–85% of the total extractant volume to be recycled by evaporation–collection of most of the extractant volume. Electrical heating of the extractant, the efficiency of which is independent of the extractant polarity, is also crucial for this step.

This prototype, which is especially flexible, has been the subject of the following modifications and/or combinations:

Reverse configuration: The name given to this configuration comes from the sample location, which is not the cartridge, but rather the extraction vessel (see Fig. 4B). This modification enables the use of acid extractants, which can destroy the cartridge upon contact with them under microwave irradiation. Thus, the cellulose cartridge is used as a filter rather than as a sample container. The main drawbacks of this configuration are the inability to *in situ* recycle of the extractant – hence it is only applicable to aqueous extractants, and the dilution effect on analytes; in any case, it is very useful for removal of metals from coal [30].

Coupling for monitoring extraction: A flow-injection (FI) interface between the FMASE (in modified form) and an appropriate detector allows the extraction process to be independent of the sample matrix [31]. Fig. 4C shows the overall system, which comprises the following parts:

(a) The extractor, which is connected to the distillation flask in addition to the Microdigest A301 with the orifice at the bottom, and the quartz sample container in which the Megal 500 thermometer is inserted. The distillation flask is connected to condenser 1, with a reservoir for condensed vapour, from which fresh extractant is pushed to the quartz sample container. A second condenser (number 2) directly connected to the quartz container condenses vapour from it. No siphon is used and the extract is led from the orifice at the bottom of the quartz vessel to a graduated reservoir after each cycle. A two-channel low-pressure pump is used to transfer the distilled extractant from the reservoir in condenser 1 to the quartz vessel and, following contact between the sample and extractant, to lead the extract to its reservoir.

(b) The dynamic FI manifold for on-line monitoring of the extraction process consists of a low-pressure pump and injection valve, a flow-cell located in a fluorimeter and transport tubes to lead the effluents from the outlets of the injection valve and flow-cell to the distillation flask.

Each cycle involves filling the quartz vessel containing the cartridge and sample with fresh extractant from the distillate reservoir, irradiation with focused microwaves and unloading of the extract in its reservoir, the graduation in which allows measurement of the aspirated extract volume.

Simultaneously with the start of each cycle, the channel of the FI pump, which is used to aspirate the extract from the previous cycle, is enabled to have the stream circulate through the 500-μl loop of the injection valve IV onto the distillation flask. The outlet of the flow-cell in the fluorimeter also reaches the distillation flask, thus avoiding losses of the extract used for monitoring.

This combined system fulfills the following objectives: (1) operation as a screening system (yes/no answer); (2) monitoring of extraction kinetics; (3) semi-quantitation of the analytes in routine analyses when the sample composition is approximately known. It is also very useful with a view to establishing the refractivity of samples without times losses, and overcomes the most significant limitation of extraction techniques in general when the yields of specific compounds to be extracted are dependent on the bulk composition of the sample (matrix effects).

This configuration can be modified in order to couple extraction to other steps of the analytical process such as derivatization, pre-concentration, clean-up or any type of high-resolution separation (*e.g.* gas or liquid chromatographs, capillary electrophoresis) or detection.

Automatic configuration: This configuration is similar to part A of the previous one (see Fig. 4D). In this case, two single-channel piston pumps equipped with flexible tubes are used to aspirate the extractant and replace the siphon. In this way, more strict control of the contact time between sample and fresh solvent is achieved by aspirating the latter at preset intervals and introduction of fresh extractant into the cartridge at the preset flow-rate is facilitated. Operationally, the process consists of a number of cycles each involving the following three steps: (1) filling of the extraction vessel with fresh extractant delivered by pump 1; (2) microwave irradiation; (3) unloading of the extraction vessel and delivery of the extract to the distillation flask with the help of pump 2 [32].

As stated above, the main drawback of the first FMAS prototype was the difficulty of using high-boiling extractants. This excluded "green" applications based on the use of water as extractant. This shortcoming could have been circumvented by replacing the glassware with piston pumps and Teflon tubing; however, it promoted the development of a new prototype intended to expand its scope of application with other types of extractant [33].

6.2.2. Automated, flexible prototype overcoming the shortcomings of the first

This second prototype was constructed by SEV (Puebla, Mexico) and called MIC II (Fig. 5). It is based on the same principles as the previous FMAS extractor and consists of a single unit where shortening of the distillation glassware allows reception of the extractant vapour on a condenser connected to the top of the sample cartridge vessel with minimal losses in the way, its condensation, and dropping on the solid sample. The siphon has been replaced with a valve that allows filling of the vessel to the desired level or its draining to the distillation flask. The short glassware distillation path used

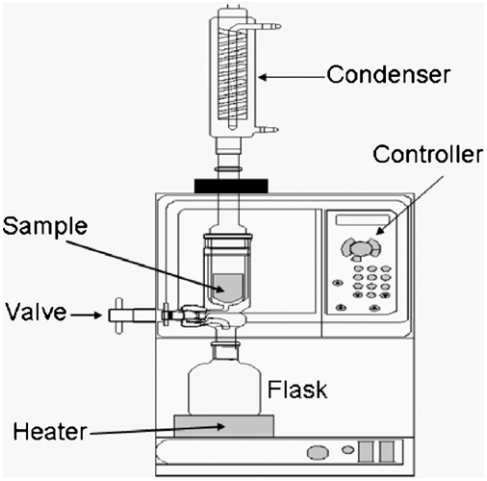

Fig. 5. Scheme of the MIC II FMASE (adapted from Ref. [34], reproduced with permission of American Chemical Society).

affords the use of water or other high-boiling extractants. Since unloading of the extract from the sample vessel can be controlled via a switching valve, a new operational variable named "delay time" (*viz.* interval during which the sample is in contact with the solvent after microwave irradiation and before draining from the irradiation vessel) can also be optimized for improved extraction [34,35]. The device operates at a microwave power between 100 and 400 W with irradiation time control ranging from 1 s to 1 h. The main limitation of this prototype is that the extraction process cannot be completely automated; thus, the valve must be switched by hand, and so must microwave irradiation. One other limitation is inability to recycle the extractant, which is desirable with extractants other than water. Complete automation and extractant recycling were thus two objectives to be fulfilled with a new prototype.

6.2.3. A dual-operation automated prototype: advantages of the definitive prototype (commercial availability)

A fully automated focused microwave-assisted Soxhlet extractor was designed and constructed also by SEV. This extractor (Fig. 6), called MIC V, uses two extraction units, which allow the simultaneous processing of two samples for replicated extraction. Automation is accomplished by using an optical sensor, a solenoid valve and control via microprocessor software. An 18-cm long siphon houses the optical sensor, which is positioned at a given siphon height to have the magnetron start irradiation of the sample when the solvent reaches the preset level. The higher the position of the optical sensor along the siphon is, the higher the extractant volume that is brought into contact with the target sample in each cycle. The solenoid valve, inserted at the bottom of the siphon, is automatically switched at the end of the irradiation step to empty the sample vessel. One parameter related with extractant volume, and hence dependent on the position of the optical sensor, is the unloading time, which is the time during which the solenoid valve remains in its unload position. This prototype can be coupled to other steps of the analytical process via an appropriate FI interface by introducing a Teflon tube in the distillation flask. This final prototype overcomes the limitations of its predecessors and affords fully automatic extraction of two samples at once [36,37].

6.2.4. New incoming prototype

A new, more compact prototype called Accesox (Barcelona, Spain) has recently been developed to reach a wider market. This device has the additional choice of the maximum temperature to be reached in the sample–extractant medium during microwave irradiation. In this way, the temperature of the leaching process can be effectively controlled, which can be indispensable for applications involving thermolabile compounds.

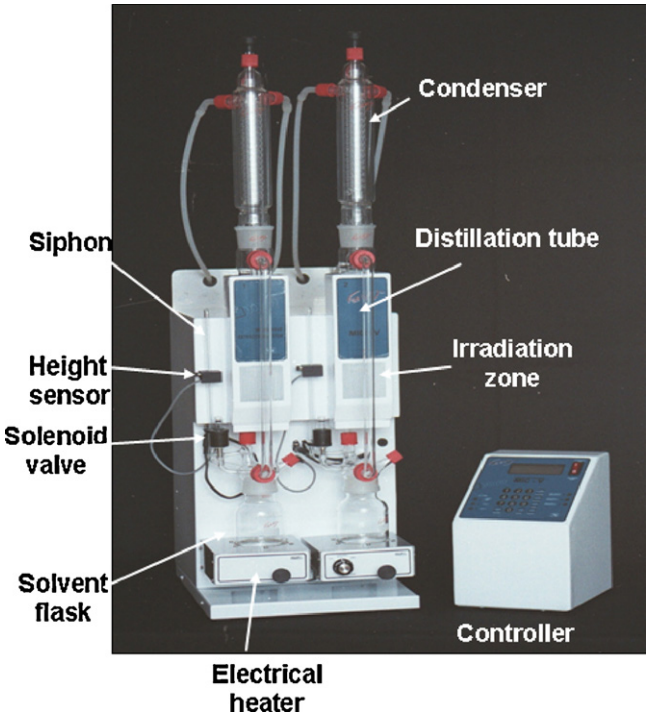

Fig. 6. Automatic FMASE prototype (adapted from Ref. [36], reproduced with permission of Elsevier).

7. Conclusions

Soxhlet extraction has for more than a century demonstrated its advantages, which have surpassed in most cases its shortcomings. The latter have been more or less successfully overcome in the following ways:

(1) By increasing the pressure into the sample cartridge, thus favouring extractant penetration into the solid and shortening the extraction time as a result; and also decreasing the extractant volume. Nevertheless, working at high pressure complicates the experimental set-up.
(2) By automating extraction using different approaches that have given place to a number of commercial extractors with different characteristics but with a common denominator: shortening of the extraction time, decreasing of the extractant volume and providing simultaneous extraction of several samples. Maybe the most significant shortcomings of these devices are relatively high acquisition costs and lack of versatility.
(3) By assisting extraction with auxiliary energies. There is at present no commercial extractors based on this principle. Nevertheless, the use of ultrasonic energy and, mainly that of microwaves, looks very promising and is, in the authors' opinion, the best alternative so far to surpass Soxhlet shortcomings.

It is clear that conventional Soxhlet has for long time been the best leaching alternative. Improvement of the conventional extractor by incorporation of present technologies allows its adaptation to the present necessities; so, it can be said that Soxhlet extraction has been, and it is, almost a panacea in this area.

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