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Soxhlet extraction of solid materials: an outdated technique with a promising innovative future

M.D. Luque de Castro*, L.E. García-Ayuso

Department of Analytical Chemistry, Faculty of Sciences, University of Córdoba, E-14004 Córdoba, Spain

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Abstract

An overview of the evolution of Soxhlet extraction of solid materials and its comparison with the performance of other conventional and new extraction techniques is presented. First, a discussion on both conventional Soxhlet as compared with other conventional extraction techniques and some minor improvements of the former for specific applications is done. Secondly, a critical comparison of conventional Soxhlet with the new extraction techniques such as supercritical fluid extraction, microwave-assisted processes and microwave-assisted solvent extraction shows the reasons why major, recent improvements of this technique (namely Soxtec® System HT, Soxwave-100 and focused microwave-assisted Soxhlet extraction) have been proposed, aimed at overcoming most of the shortcomings of conventional Soxhlet and converting it into an updated tool for leaching which competes advantageously with the most recent alternatives in the extraction field. © 1998 Elsevier Science B.V. All rights reserved.

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1. Introduction

Sample pretreatment is often one of the most time consuming steps of the analytical process, particularly when solid samples are involved. The search for modification of the present devices, the design of new devices and the use of auxiliary sources of energy which shorten and/or enable automation of sample pretreatment have been the aim of analytical chemists in the last decades.

Solvent extraction of solid samples, which is commonly known as solid–liquid extraction, but which should be referred to, in a more correct use of the physicochemical terminology, as leaching or lixiviation, is one of the oldest ways of solid sample pretreatment. Among the techniques used for implementation of this step, Soxhlet has been the leaching technique mostly used for a long time. This assertion is supported by the fact that Soxhlet has been a standard technique during more than one century and, at present, it is the main reference to which the performance of other leaching methods is compared. In conventional Soxhlet, originally used for the determination of fat in milk [1], the sample is placed in a thimble-holder, and during operation gradually filled with condensated fresh solvent from a distillation flask. When the liquid reaches the overflow level, a siphon aspirates the solute of the thimble-holder and unloads it back into the distillation flask, carrying the

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*Corresponding author. Tel.: +34 57 218615; fax: +34 57 218606; e-mail: qa1lucam@uco.es

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extracted analytes into the bulk liquid. This operation is repeated until complete extraction is achieved. This performance makes Soxhlet a hybrid continuous–discontinuous technique. Inasmuch as the solvent acts stepwise, the assembly can be considered as a batch system; however, since the solvent is recirculated through the sample, the system also bears a continuous character.

The most outstanding advantages of conventional Soxhlet are as follows: the sample is repeatedly brought into contact with the fresh portions of the solvent, thereby helping to displace the transfer equilibrium. The temperature of the system remains relatively high since the heat applied to the distillation flask reaches the extraction cavity to some extent. No filtration is required after the leaching step. Sample throughput can be increased by simultaneous extraction in parallel, since the basic equipment is inexpensive. It is a very simple methodology which needs little specialized training, has the possibility to extract more sample mass than most of the latest methods (microwave-extraction, supercritical fluids, etc.), and is non-matrix dependent. There is a wide variety of official methods involving a sample preparation step based on Soxhlet extraction [2–7].

The most significant drawbacks of Soxhlet extraction, as compared to the other conventional techniques for solid sample preparation are, the long time required for the extraction and the large amount of solvent wasted, which is not only expensive to dispose off but which can itself cause additional environmental problems. Samples are usually extracted at the boiling point of the solvent for a long period of time and the possibility of thermal decomposition of the target compounds cannot be ignored, when thermolabile analytes are involved. The conventional Soxhlet device is unable to provide agitation, which would accelerate the process. Due to the large amount of solvent used, an evaporation/concentration step after the extraction is mandatory. The technique is restricted to solvent selectivity and is not easily automated.

Due to these advantages and disadvantages associated with Soxhlet, a series of authors have tried to improve the conventional Soxhlet device, either by modifying its design or the operational procedure. Other Soxhlet changes have been aimed at developing special applications prohibited to the conventional extractor.

The aim of this review is both to outline the importance of Soxhlet as a model to which the performance of other methods is referred and offer to the readers a critical overview of the evolution of this technique from the most simple to the most innovative and useful modification thus enabling to predict its promising future.

2. Soxhlet vs. other conventional leaching techniques

Methods based on Soxhlet extraction have been and are at present used as reference when conventional methods based on other principles have to be evaluated. In this context the methods most frequently compared with Soxhlet have been those based on the use of ultrasounds, shaking or stirring and on a combination of both of them. Most of these conventional methods have in common with Soxhlet, the fact that they are time-consuming and require large amount of solvent. On the other hand, they are relatively simple both in performance and fundamentals, so their development does not require specialized personnel. Finally, these methods are cheap, which has favoured significantly their widespread use particularly both in industries and routine laboratories.

Ultrasound-assisted extraction is, together with Soxhlet, the most accepted conventional leaching technique. Ultrasound-assisted methods are usually developed in a discontinuous, batch mode, and the shortening of the extraction time (with respect to that in the absence of ultrasounds), is due to an increase of both pressure (which favours penetration and transport), and temperature (which improves solubility and diffusivity), both increasing the transport phenomena and displacing the partitioning equilibrium. Two major shortcomings of the ultrasound-assisted extraction are: (i) its inability to renovate the solvent during the process, which causes its efficiency to be a function of the partition constant, and (ii) the mandatory filtration and rinsing steps after extraction, which involve a longer time for the overall process, a higher solvent consumption and the danger of both loss and/or impurification of the extracted species during manipulation. The increase in polarity of the system (including solvents, analytes and matrix) increases efficiency, which can either surpass [8–11] or can be similar to [12] that obtained by conventional Soxhlet. A co-solvent is sometimes added in order to increase the polarity of the liquid phase [13,14]. The use of ultrasound-assisted extraction is advisable for thermolabile analytes which are altered under Soxhlet working conditions [15]. In a number of comparisons, the efficiency of both the alternatives is similar [16–20], but Soxhlet extraction provides better reproducibility [21,22], and in other comparisons the efficiency of Soxhlet is higher than that of ultrasound-assisted extraction [23–27]. Therefore, a wider field application, better reproducibility and efficiency and less sample manipulation are the advantages of Soxhlet extraction vs. ultrasound-assisted extraction, but at the expense of a longer extraction time.

Shaking extraction and stirring extraction (or Vortex extraction) methods have also been compared with their Soxhlet counterparts. The former are in general less effective than Soxhlet [28–32] as they involve most of Soxhlet disadvantages, but none of its advantages. Some additional help (namely, an enzymic reaction [33,34], ultrasound radiation [35], solvent mixtures [36–38], etc.) has sometimes been coupled to the shaking or stirring step in order to improve the overall efficiency, it hardly surpasses that of Soxhlet extraction.

Only devices based on one of these principles but especially designed for specific purposes surpass Soxhlet extraction performance. For example, distillation with solvent reflux, frequently used in organic chemistry has only been compared to Soxhlet extraction in the exhaustive extraction process [39] with hot thermal degradable samples [40,41]; perforation in H_2SO_4 [42], which avoids artefacts formation, common in some Soxhlet extractions; azeotropic distillation [43], useful for the removal of analytes from environmental matrices as most of the interferences are unable to form azeotropes which cannot be distilled and therefore remain in the extraction flask [44]; homogenization [45–47]; thermal degassing in special devices [48]; Gerber extractor for fat [49,50], Gulbaran extractor of oil from seeds [51], and Mojonnier method [52] for the extraction of fat from foods, are some of the concrete approaches which provide better results than Soxhlet extraction at the expense of a more limited application.

In short, Soxhlet extraction is a general, well-established technique which clearly surpasses in performance other conventional extraction techniques and which is only surpassed by specific designs referring to a limited field of applications.

3. Minor soxhlet improvements

The majority of simple modifications developed from the original Soxhlet device [1] has been focused to pre-established goals. These changes consisted of minor alterations aimed at applying them to a particular type of sample (liquid, thermolabile, etc.) and new designs of basic units such as the thimble-holder, siphon, condenser, etc. which slightly improved the features, application field and/or results of the methods thus developed.

In order to develop the liquid–liquid extractions, the conventional Soxhlet extractor has been adapted for the continuous extraction of a liquid with either a lighter or heavier solvent. Its performance is similar to a combination of functions of a separating funnel and a Soxhlet extractor, working in a manual [53], or automated way [54]. The extraction of lipids under constant moisture and vacuum conditions requires a sealed Soxhlet extractor [55], while the extraction of biological materials without heating has been carried out by a modified extractor in which liquid–liquid extraction is carried out with percolated rather than with the fresh solvent [56]. Combination of heating under reflux and percolation has been used for fat extraction from the vegetable samples [57] and a hybrid Soxhlet extractor and Dean-Stark distillator has enabled the removal of chlorinated dibenzo-*p*-dioxins and dibenzofurans from matrices with high water content [58].

The units which constitute a Soxhlet extractor have been modified with different aims. Thus, changes in the thimble-holder have been aimed at, (a) the simultaneous performance of extractions using a glass cylinder with a stainless-steel wire platform on which eight porous plastic cartridges stand [59]; (b) the development of room temperature extractions for thermolabile species by locating the extraction thimble-holder separated from the solvent flask in such as way that the heat from the heating source never reaches the sample zone [60]; (c) avoiding turbulence in the sample zone during the siphoning step by positioning a glass support into the thimble-holder, where a small weighing bottle is mounted in [61]; and (d) the distillation and re-use of the solvent, minimizing both the solvent losses and the time taken for the removal of the solvent from the extract [62].

The Soxhlet siphont has also been modified by putting a sintered-glass disc at the bottom of the extraction chamber and an outlet with a PTFE stopcock below the disc. The stopcock has twofold objective of controlling the flow of the solvent, thus maintaining a constant level above the solid to be extracted, and avoiding dropping of the toxic solvent from the chamber by closing it when the flask is exchanged [63]. Another modification which enables constant solid–solvent contact is achieved by converting the siphon tube into a constant-level device by leading a tube from its upper bend back into the extractor and then to the atmosphere [64]. In situ evaporation of the solvent after extraction has been achieved by inserting a stopcock in the siphon tube [65]. A no-siphon Soxhlet, fitted with either a sprinkler device or a cylindrical glass tube with a serrated bottom end (depending on the use of solvents lighter or heavier than water) has been designed and used for the drugs extraction from biological fluids [66].

Both the geometry of the condenser and its performance have been optimized in order to maximize both the boiling rate and the solvent temperature in the extraction chamber [67,68]. Also the condenser has been modified for increasing the safety by minimizing or avoiding losses of boiling liquid which result from bumping in a superheated solution [69]. A faster extraction in compact samples has been achieved when the extractor was modified for fitting a stirrer inside the filter cartridge and the reflux condenser was sealed into the side of the extractor making an angle of 30° with the vertical axis [70]. Some other less noticeable modifications [71–74], have also been reported.

4. Soxhlet vs. new leaching techniques

Recent leaching methods have as a common feature an improvement of the extraction efficiency due to the use of solvents at high temperature and pressure. In addition, they require shorter extraction time, use less amount of solvent, allow simultaneous extraction of several samples most of the times, and either work automatically or are easy to automate. Among a pleiad of new methods, those based on the use of microwaves as auxiliar energy source, supercritical fluids as extractants and the so called 'accelerated solvent extraction' are most relevant. In order to perform a better sustained comparison with Soxhlet the fundamentals and the more salient aspects of these methods are commented.

The usefulness of microwave irradiation as a heating source for the assistance of wet ashing techniques was demonstrated in 1975 [75]. Microwaves have assisted mainly digestion steps [76–80]; nevertheless, they have also been successfully used to help leaching steps. The first in this field were Ganzler and Salgo [81,82], who used a domestic microwave oven in order to demonstrate a higher extraction of polar compounds by this leaching as compared to the Soxhlet method for these compounds. In order to avoid degradation of the target analytes the authors carried out their process in several short heating and cooling cycles. After the pioneers, a number of researchers have used microwaves, either in cyclic mode [83] or in the continuous mode [84] for helping extraction, but it has been only in the late 1980s and the beginning of the 1990s after the development of the commercial devices, this method for sample pretreatment has been widely accepted. Noticeable contributions to expand the use of microwaves as an alternative to conventional methods have been reported by Paré and López-Ávila (microwave-assisted processes MAP™ [85,86] and microwave-assisted solvent extraction MASE [13,23]) using focused and multimode microwaves.

The advantages of microwave-assisted leaching vs. conventional Soxhlet result from the performance of this heating source. Unlike conventional heating based on the conduction and convection phenomena, that is, by exterior contact, microwave heating is based on dielectric loss. The heat appears in the bulk of the irradiated material, thus giving rise to an inverse temperature gradient; that is, volume rather than surface heating. The high temperatures reached by microwave heating reduce dramatically both the extraction time and the volume of solvent required. Absorption of microwave-energy is proportional to the relative sample or solvent permittivity [87], so the extraction efficiency improves when the polarity of the analytes increases; that is from PAHs [88–91] to more polar compounds such as organochlorinated [83,23,89] and organophosphorous pesticides [23], phenols [89,92], etc. and with the solvents with a high dielectric constant.

The main difference in the use of open vs. closed microwave-based extractors is the high pressure to which the sample–solvent system is subjected later [PTFE, hermetically closed vessels] in a microwave oven. An open vessel (glass or quartz) to which the monomode radiation is focused is used for the former type, thus achieving a precise, localized and controlled heating vs. a drastic heating. The positive aspects of both modes of microwaves for assisting leaching processes looks like the panacea for the problems with Soxhlet, the only advantage of the latter being its lower acquisition cost. More in depth studies [93–95] have demonstrated that the efficiency of microwaves can be very poor when either the target analytes or the solvents are non-polar or of low-polarity, when they are volatile and/or when the solvents used have low dielectric constants. In these cases, very common in organic chemistry, Soxhlet extraction is superior to microwave-assisted extraction.

Accelerated solvent extraction (ASE) constitutes a leaching technique which is based on the principles similar to those of MAP and MASE, but microwave-energy is replaced in ASE by conventional heating in an oven [96]. Commercial devices are automated and the extraction step is developed in a very short time with small volume of organic solvents at elevated temperatures (>200°C) and pressures higher than 200 bar in order to keep the solvent in liquid state [97]. This approach has been tested by extracting PCBs [98], PAHs [99], chlorinated herbicides [100] and pesticides [101], etc. from soils, showing as main drawback a strong background interference and high detection limits [102]. Most of the applications of ASE are screening methods. In cases where this alternative has been used for quantitative purposes and the results are compared to those from conventional Soxhlet, an acceptable agreement has been found [102] despite Soxhlet extraction has not been performed under optimal conditions.

Supercritical fluid extraction (SFE) is one of the most successful and recent contributions to leaching techniques [103]. The very special properties of fluids under supercritical conditions, between those of liquid and gases (i.e. solvation power similar to liquids, but low viscosity and high diffusivity similar to gases) dramatically facilitate transport phenomena, thus making extraction faster and more effective. Despite other supercritical (SC) fluids such as freons, ammonia, organic solvents, etc. have been used as extractants, the most common SF extractant used at present is CO_2. This fact is due to its low toxicity and inflammability, reasonable critical conditions and its chemical inertness. The most important shortcoming of SC–CO_2 is its weak interaction with both analytes and matrices which, when pure, provide poor efficiencies in the extraction of environmental persistent pollutants (namely, PAHs [104], PCBs [105], chlorinated dioxines [106], etc.) from complex matrices as soils or sludges. This problem has been overcome either by addition of co-solvents (namely methanol [107], pentane[108], toluene[109]), so as to increase the polarity of the extractant, or by reducing the polarity of the analytes to be leached by complex or ion pair formation, esterification and reverse-micelle formation. Both alternatives have been used separately or in combination, whereas a third one consisting of addition of polarity modifiers plus active components which decrease matrix effects on SFE [109] has also been proposed. The advantages of SFE vs. both Soxhlet and other conventional leaching techniques are time reduction, facility for on-line coupling with either detectors or chromatographs [110], less necessity for cleanup due to the high selectivity achieved by manipulating pressure and temperature [111], and, most of all, suppression of solvent removal steps as the extractant is released from the leached species after depressurisation. Between the three trapping systems usually employed for analyte collection after extraction [112], namely, liquid collection, cryogenic trapping and solid-phase trapping, the last is the most effective as it allows simultaneous collection, cleanup and concentration prior to either individual chromatographic separation or direct detection. In spite of the advantages of SFE and the wide display of commercially available apparatuses and instruments which have stimulated the development of new applications, this technique has not fulfilled the expectations of traders and users so far owing to the following facts: (a) the big discrepancies in efficiency between spiked and natural samples [104,105,113,114]; (b) the number of methods reported in the literature in which the efficiency is lower than that provided by Soxhlet methods [108,115]; (c) the poor ruggedness of SF extractors, particularly the restriction and trapping units [116], which hinders transfer of an optimized–validated method from one extractor to another; (d) the scarce knowledge about both the way of overcoming analyte–matrix interactions and the use of the most appropriate modifier.

Enhanced-fluidity liquids is a mode of SFE in which the extractant consists of a big proportion of organic solvents associated to a liquid of low viscosity such as CO_2. The resulting mixture is used in a single phase region of the corresponding phase diagram at temperature below the critical value of the mixture. The approach has been used for the extraction of phenolic pollutants with results similar to those provided by conventional Soxhlet [117]. The advantages and disadvantages of this leaching mode with respect to the existing ones have not been clearly stated yet.

The use of sub- and supercritical water for continuous leaching takes advantage of the excellent features of this universal solvent such as price, environmental compatibility, facility for achievement of a high purity quality, etc. However, the most important feature of water from the point of view of its use as a general extractant is the high manipulation of its dielectric constant by changing the temperature under moderate pressure. Thus, from a dielectric constant close to 80 under normal conditions, this parameter decreases to 27 at 250°C and 40 bar, and to less than 10 under SF conditions. This allows the range of polarity of the extracted analytes to be dramatically expanded. Thus, compounds such as hydrocarbons, PAHs, PCBs, etc., which are not extractable by water under normal conditions [118,119], are efficiently extracted when the temperature is raised and the appropriate dielectric constant is reached [120–123]. The most important shortcoming of water as extractant vs. SC–CO_2 is its liquid state under normal conditions, which yields diluted solutions of the extracted species. This shortcoming is circumvented by in-line connection of a preconcentration device after the restrictor (namely, absorption or ion-exchange minicolumn) [124,125]. This approach has not been systematically compared with Soxhlet because of the very different operating conditions required by both techniques especially when it comes to the extractants used.

A remarkable aspect of these new leaching techniques when applied to some natural samples is the obtainment of efficiencies higher than 100% referred to conventional Soxhlet (even 200%, despite Soxhlet has been continued for 80–90 h). This behaviour can be explained by the non-drastic conditions used in Soxhlet: when some fraction of the target analyte is strongly bound to the matrix there is not enough energy involved in the Soxhlet process for its separation, which is achieved under the working conditions of these new alternatives. This fact has called for improvements of the conventional Soxhlet approach in order to circumvent this serious negative aspect which had not been identified before.

5. Present, major soxhlet improvements

Most of the modifications of the conventional Soxhlet extractor developed in the last few years have been aimed at making its performance more similar to that of the recent techniques for solid sample treatment: namely shortening the time for the leaching step, using auxiliary energies and automation. Thus, the most essential alterations of conventional Soxhlet have led to the design of high-pressure, automated and microwave-assisted Soxhlet extractors.

High pressure in a Soxhlet extractor has been achieved by constructing it either of heavy Pyrex-glass [126], or stainless-steel [127], or by placing the Soxhlet extractor in a cylindrical stainless-steel autoclave [128]. The aims of this modified Soxhlet extractor have been the removal of highly reactive compounds with solvents of low boiling point [126] and gases under normal pressure and temperature [128], thus keeping the extractant in liquid state, as in the extraction of PAHs from aerosols using fresh liquid–CO_2 at 45–50 bar which passed through the sample cartridge for 3–6 h [129], or at 60 bar for the extraction of essential oils [130]. Also Soxhlet extraction with supercritical CO_2 has been carried out using stainless-steel equipment both laboratory-made [127] and commercial one [131]. The former device was used among others for the removal of morphine alkaloids from seeds and biological fluids. Despite the device was described, used and shown as more efficient than other extraction alternatives, further applications have not been reported by the authors [127]. A commercial high-pressure Soxhlet stainless-steel extractor was used by Bernal et al. [131] for the removal of PCBs from different foodstuffs-certified reference materials using liquid CO_2 at a working pressure of 55 bar and at a temperature between 42°C and 46°C. Working conditions close to those corresponding to the supercritical state of the extractant dramatically complicated the performance of the extractor as the cycles were halted. That is a result of solvent evaporation. So, despite the fact that supercritical fluid Soxhlet extraction could, in principle, seem simple and with lower maintenance costs than conventional continuous supercritical fluid extraction [103] due to fresh solvent recycling with minimal consumption, and with higher efficiency than recirculating conventional SFE, the change from supercritical state to liquid and vice versa adversely affects Soxhlet performance. This limitation hinders the expansion of supercritical fluid-based Soxhlet.

A mandatory step of Soxhlet extraction evolution is automation. The commercial equipment called Soxtec® System HT is the more widespread device in the field of automated Soxhlet as it performs extraction with similar precision as conventional Soxhlet, but with a significant saving of time [132]. The approach employs a combination of reflux boiling and Soxhlet extraction (both assisted by electrical heating), in such a way that the overall process involves two extraction steps: a boiling and a rinsing step. Finally, a step for the recovery of the solvent is also developed. Statistical comparison of Soxtec methods with Soxhlet ones [133] has shown correlation coefficients (r) higher than 0.98. Since its commercialization in 1982, Soxtec methods have been developed and put into practice in a large number of applications [134–136]. Despite the fact that Soxtec is mainly used as a way of shortening the leaching time [137–139], it also improves the efficiency of the process in strongly bound analytes, whose transfer to the liquid phase is highly favoured during the boiling step [140,141]. The efficiency of extraction is, at least, equivalent to that obtained with conventional Soxhlet.

Microwave-assisted Soxhlet extraction shows four main differences concerning 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; (3) the extraction process is totally or partially performed as in conventional Soxhlet, i.e. with permanent sample-fresh solvent contact; (4) subsequent filtration is not required. Therefore, these techniques maintain the advantages of conventional Soxhlet overcoming the limitations of the latter such as the long extraction time, incapability for both automation and quantitative extraction of strongly retained analytes, etc. The two alternatives of microwave-assisted Soxhlet are the Soxwave-100 extractor, patented and commercialized by Prolabo (Paris, France) and the focused microwave-assisted Soxhlet extractor (FMASE), designed by the authors of this review and constructed by Prolabo. The main differences between both are as follows: (a) the principle of Soxwave-100 is similar to Kumagawa extraction, but with operational performance similar to Soxtec® System HT [132]; that is, a three-step extraction. The FMASE device works as a conventional Soxhlet; that is, a series of cycles in which the extractant is completely renewed but with sample irradiation by microwaves at preset time within each cycle. (b) While the Soxwave-100 extractor uses a single heating source (i.e., focused microwaves) which acts both on the sample and the solvent, the FMASE uses two energy sources: microwaves for sample irradiation and electrical heating of the extractant. This latter distinction in performance leads to a number of differences in behaviour, namely:

1. the dielectric constant of the solvent used as extractant is of paramount importance in the Soxwave-100 extractor as the heating source of the solvent is microwaves; so polar solvents are more favourable than the non-polar or low-polar ones. Solvent distillation in FMASE is achieved by electrical heating, which is independent of the polarity;
2. as far as the energy required by the solvent is different from that required by the sample for removal of the target analytes, the adoption of a compromise is mandatory in Soxwave-100 extraction. This is not the case with FMASE, in which each temperature can be optimised independently;
3. the Soxwave-100 performance involves a first step in which the sample is immersed into the boiling solvent, followed by lifting of the cartridge over the solvent and continuous dropping of the condensate on the cartridge. In this step, a matrix-solvent partitioning equilibrium of the extractable species is established while the microwave radiation acts on both the sample and the solvent; in the second

step the partitioning equilibrium is displaced to extraction completion due to the contact of the fresh solvent with the sample, which is not subjected to microwave energy during this step. In FMASE clean solvent and microwave irradiation are simultaneous, thus favouring the mass transfer and shortening the extraction time as a result.

Soxwave-100 applications described so far are restricted to 'application sheets' from Prolabo (namely, environmental [142–144], polymers [145], drugs [146] and food samples [147–149]). The results have been compared with those provided by conventional Soxhlet and a lower efficiency than by the latter has been found in all the instances. Applications of FMASE to the extraction of PAHs, alkanes and herbicides [150], as well as to fat in foods (edible oilseeds, cheese and milk) [151,152] show efficiencies comparable to or higher than conventional Soxhlet.

It is worth emphasizing that the Soxhlet-100 is an automated extractor but the FMASE is a prototype which can be easily automated, as proposed by the authors of this review to the prototype manufacturer, by introducing the following changes: a multiposition carrousel for automated placing and removal of the cartridge; a distillation flask assembly including a multichannel valve for automated flask changeover; an optical sensor for monitoring both the solvent level in the extraction chamber and the analyte contents in the solvent; and a computer for fully controlling the extractor operation (lifting, lowering and rotating of the cartridge in the carrousel; switching the selecting valve; setting the time and duration of microwave irradiation through the sensor signal, etc.

6. Conclusions

Conventional Soxhlet extraction has been the most used extraction technique worldwide for a number of decades, surpassing the performance of other extraction alternatives and being used as an efficiency reference for the comparison of its conventional and new counterparts. A series of minor changes have allowed conventional Soxhlet to be applied to specific applications prohibited to the unchanged device.

The design and use of new, fast extraction techniques such as supercritical fluid extraction, accelerated solvent extraction and microwave-assisted solvent extraction, among others, had relegated conventional Soxhlet as an old-fashioned, time consuming and manual technique which has been updated by incorporating auxiliary energies such as microwaves, endowing Soxhlet with features which enable it to be advantageously compared with its recent counterparts.

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