Ultrasonically Assisted Extraction (UAE) of Natural Products Some Guidelines for Good Practice and Reporting

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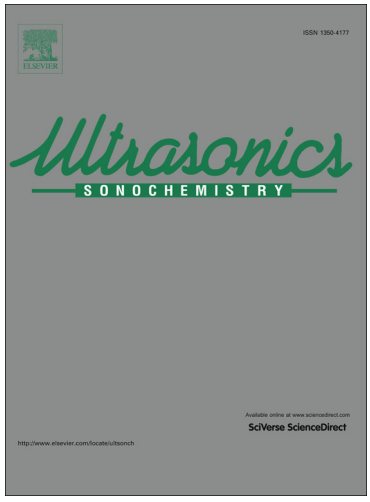

Short communication

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Ultrasonically Assisted Extraction (UAE) of Natural Products
Some Guidelines for Good Practice and Reporting

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Abstract

Over the years I have reviewed a large number of papers concerning the ultrasonically assisted extraction (UAE) of natural products and many of them suffer from common problems that make them unsuitable for publication. In this article I will identify these problems in the hope that new manuscripts can avoid such pitfalls.

Many authors submit papers that describe just one herb which was investigated under UAE. They rationalise that the paper is worthy of publication simply because this particular herb was never before been extracted using ultrasounds. On its own this is definitely not a sufficient reason for the acceptance of a paper dealing with UAE unless something outstanding was found during the investigations. Even if the paper describes more than one herb it is also again important to note that simply because the herbs were extracted ultrasonically for the first time it is not an adequate scientific reason for publication. In both cases there must be some substantial findings to make such papers valuable to the scientific community.

It is frustrating for anyone who wishes to pursue the published UAE research to find that a published paper does not adequately describe the nature of the herbal material. It is not sufficient simply to name the species and origin of the sample it is essential to describe the physical properties of the herbal materials:

- Part of the plant used: entire herb; leaves; bark; stems; roots; rhizomes; etc.
- Its physical state: chunks of herb; crushed herb; powdered herb. In the last two cases the particle size range used for UAE experiments is also necessary since this has a significant influence on solvent extraction.
- How the herb was dried is also of importance i.e. forced air/oven, natural in full sun or shade, etc.
- Residual moisture in herbs should be determined and reported. After any form of drying of a plant material there will always be some moisture remaining and this will

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enter the solvent and alter its composition (particularly if the solvent is water soluble e.g. ethanol).

- Finally and if possible, it is very useful to report not only how the plant material was powdered and what type of grinder was used but also whether the sample was sieved. It is good practice in the extraction the herbal powder to use material that has been subjected to a screening procedure in which the herb powder has a well-defined, size through the use of calibrated granulometric sieves.

In some papers the ultrasonic equipment used in the experimental part is very poorly described. It is definitely not sufficient to state simply that the extraction was performed using an ultrasonic probe or cleaning bath. In addition to this the manufacturer must be named and it is also necessary to mention the ultrasonic frequency of the machine and the real power used for the extraction (which is definitely not the power output of the equipment as quoted by the manufacturer). When an ultrasonic bath is used this will be generally for indirect sonication [1]. In order to properly describe the experimental work, the volume of the extraction vessel, its geometrical features (round or flat bottomed and in the latter case the diameter of the base of the flask) must be stated. In all cases the position extraction of the vessel in the bath should be carefully chosen to be above one the transducers and at a specified distance above it to ensure an acoustic field entering into the vessel as constant as possible. The positioning can be determined simply using the aluminium foil cavitation activity determination [2].

Similar care and attention should be taken when describing an ultrasonic probe system which is normally used for direct sonication [1]. In this case the probe diameter as well as tip area (for many probe system sonicators the tip is not circular and so its area is smaller than that calculated simply from the probe diameter).

As mentioned above a very important parameter for UAE, or indeed in any study of sonochemistry, is the acoustic power which enters the extraction mixture. The most common method of assessing this is by a form of calorimetry which was introduced when sonochemistry was in its infancy [3]. It must be remembered of course that this laboratory method is only an approximation of the true acoustic energy entering the mixture because a more precise quantitative determination would require an accurate calorimeter system which is not easy to use (or indeed find) in any normal working laboratory [4]. For this reason sonochemists decided that some form of simple method should be reported in order to compare results from different laboratories. There have been refinements in the methodology but the underlying principles remain the same [5, 6].

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In laboratory scale UAE the volume of the suspension of herb in solvent is known and it is this volume that can be used to estimate the ultrasonic power entering the extraction volume by calorimetry. To do this it is useful to use the same solvent as used for the extraction and volume as for real experimental work but in the absence of herbal material. However because the specific heat of the solvent is required for the calculation it is permissible to use the same volume of water as long as this is mentioned in the paper. The results obtained should be reported as W/ml, value that could be used by anyone who wants to reproduce the research work at the same scale or larger. Other methods such as the calculation of ultrasonic power by dividing the total output power (as stated by the equipment manufacturer) by the proportion of that maximum indicated by the control or dial position during the experiment is not an acceptable. This could differ with the probe system used and would change as a function of probe age, an old probe often has an eroded tip which delivers different power from a new tip at the same vibrational amplitude.

Many herbal compounds have medicinal properties and this is the reason for interest in their extraction but some UAE papers deal with mainly the medicinal properties of the extracts. Any concentration of these aspects is outside of the scope of Ultrasonics Sonochemistry which is dedicated to applications of ultrasound not medicine. A similar situation occurs when the paper concentrates on analytical methodology of the extracts which is again beyond the needs of this journal. In my opinion authors of either of the above types of paper, where ultrasound technology is not the main thrust of the extraction undertaken, should target their manuscripts at journals dedicated to such kinds of research results. In the case of Ultrasonics Sonochemistry it will be sufficient if only a brief description of the analytical methods used was included in enough detail so that another scientist to reproduce the work.

One of the very important parameters mentioned above is the temperature used in the extraction. It is a common fault for the authors who use ultrasonic baths to quote the temperature in the water in the bath surrounding the extraction vessel. This is not the temperature inside the extraction vessel which can be 2 to 5°C higher than of cooling bath surrounding it (the heat exchange is not fast enough to reach the equilibrium, especially for glass vessels). With the help of a thermocouple plunged inside of the extraction vessel the temperature could be measured with a good degree of confidence and it can be used for indirect or direct sonication.

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Many papers reporting laboratory scale experiments use volumes of extraction mixture in the range 10 to 50 ml. There is nothing wrong with this but often the authors will then claim that this is a technology scalable to industrial level. This is not true and the error probably comes from the increasing number of groups who use computer software for the "optimization" of the laboratory results and the subsequent generation of so called 'optimal parameters'. In almost all of these papers the "optimization" often neglects or does not pay sufficient attention to the:

- correct method of measuring ultrasonic power input;
- difference in acoustic field in laboratory versus large scale vessels which is an important parameter;
- distribution of cavitational bubbles in the reaction vessel and their behavior in the extraction media;
- huge difference between the behavior of cavitational bubbles in a small volume (50 ml) compared with their behavior in a large volume.

UAE is a very complex process, what is occurring in a 10 ml extraction volume is not linearly scalable to even 50 or 100 ml, to say nothing of a scale up to 100 L level. If the extraction needs 0.5 W/ml and that was 'optimally' determined for 50 ml extraction volume experiments, then for 10 or 100 L the ultrasonic power to match the laboratory experiment should be 5 or 50 kW!!! If this was a correct estimate then the excessive energy requirement would invalidate the potential of using the method reported for industrial extraction. Fortunately and as all good sonochemists know, large scale extraction is possible using specially designed equipment and at much lower powers than any such optimization programmes might predict.

In many cases the proportion of herb to solvent used at laboratory scale is too high. Good phytopharmaceutical technology practice suggests ratios of 1:5, 1:10 and sometimes 1:20 as the accepted herb/solvent (w/v) ratio. Ratios higher than 1:20 (i.e. 1:50 or more) are useful and accepted only for the extraction of very valuable compounds, their value overcoming the costs of using high level of herb/solvent ratio.

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Highlights

ultrasonically assisted extraction (UAE)
guidelines
good practice

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