ELSEVIER
Chemical Engineering Journal 87 (2002) 11–19
Chemical Engineering Journal
www.elsevier.com/locate/cej

Investigation in solid–liquid extraction: influence of ultrasound

M. Romdhane[a,*], C. Gourdon[b,1]

[a] *Ecole Nationale d'Ingénieurs de Gabés, Route de Medenine, 6029 Gabés, Tunisia*
[b] *Ecole Nationale Supérieure d'Ingénieurs de Génie Chimique 18, Chemin de la loge, 31078 Toulouse Cedex, France*

Received 1 October 2000; received in revised form 1 June 2001; accepted 18 June 2001

Abstract

The extraction of active principles or oils from vegetable products by means of an appropriate solvent is a classical unit operation in chemical engineering. The aim of the present work is to improve the yield or the selectivity of the extraction using ultrasound. Two solid–liquid extractions have been investigated: pyrethrines from pyrethrum flowers (Chrysanthemum cineraria) and oil from woad seeds (Isatis tinetoria). In the case of the first one, an acceleration of the kinetics and of the yield of the extraction has been obtained, probably linked to the increase of the intraparticular diffusion of the solute, which is the limiting step of mass transfer. On the other hand, ultrasound seems to have a weak effect in the case of woad seeds, probably because of the structure of the seeds. In order to distinguish the various effects acting on the ultrasound activation, a thermoelectric probe has been built for measuring the ultrasonic intensity profile. By this method, it is possible to investigate the influence of the presence of solid particles on the ultrasound wave propagation. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Ultrasound; Solid–liquid extraction; Extractor; Woad seeds; Pyrethrum flower

1. Introduction

Solid–liquid extraction with a solvent is an operation which appears in many industrial processes. The pharmaceutical industry particularly, but also industries manufacturing perfumes or pesticides have to deal with the recovery of active principles from plants. Vegetable materials contain generally only a small amount of active solute, but most of the time with high added value which justifies the development of high-performance separation processes. Consequently, there is a need for new activation methods, for instance the use of ultrasound.

Ultrasound is used in different operations in chemical engineering: waste-water treatment, drying, sonochemistry and solid–liquid extraction [1–3]. The effect of ultrasound on the extraction of saponin from ginseng has been studied [4]. The extraction of saponin was examined as a function of both irradiation time and acoustic pressure. It was observed that the yields of both total extraction and saponin are larger with ultrasonic irradiation than without ultrasonic irradiation. The increase in the yield of the total extraction is approximately 15%, and for saponin it is about 30%. In the field of agriculture, the influence of ultrasound has been studied on a great variety of solid substrates, showing positive effects in some cases. Indeed, the sonication of a seed grain increases its kinetic growth, and consequently the yield of the harvest. It seems also that the enzymes, which are the key of the biochemical reaction, increase their activity with a good proportioning of ultrasound. Primo [5] has improved the colour, the viscosity and the yield of orange juice. Spirov reported that sonication of grapes for 5–10 min ($f = 80\,000\,\text{Hz}$) improved the flavour of wine and its clarification. The application of ultrasound during short periods of maceration was effective in producing a greater yield of alkaloids from Datura stramonium [6].

In addition to the beneficial effect of ultrasound on the yield and on the kinetics of extraction, it allows change in the experimental conditions, for instance decrease of temperature and pressure. Then, the quality of the solute as in the case of protein extraction from autoclaved soybean flakes [7], can be maintained. Nevertheless, with ultrasound, the sonication time has to be carefully considered, since an excess can damage the quality of the solute [8].

Finally, many factors govern the action of ultrasound: ultrasound frequency, pressure, temperature, sonication time, etc. In this work, the influence of the different parameters will be investigated.

---

[*] Corresponding author. Tel.: +216-5-392-380; fax: +216-5-392-190.
[1] Tel.: +33-5-62-25-23-00; fax: +33-5-62-25-23-18.

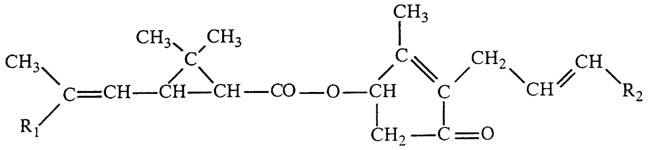
PII: S1385-8947(01)00206-6

2. Materials and equipment

2.1. Solid–liquid systems

Two solid–liquid systems have been studied: pyrethrines (solute) from pyrethrum flowers and oil from woad seeds (solid). Both are performed with hexane as solvent. The pyrethrum flowers are grown, gathered and dried in Zaïre, whereas the woad seed is gathered in the Toulouse area (France). The solid particles (flowers, seeds) are crushed in the laboratory with a blade mixer that can provide different size distributions. The analysis of the solutes can be performed as follows.

For the pyrethrines: evaporating the solvent after extraction provides a dry extract which is analysed by spectrophotometer after dilution in absolute ethanol. The global amount of pyrethrins in the extract is given via a correlation concentration versus absorbency. The pyrethrins are organic esters with insecticidal properties. They are produced by the combination of two carboxylic acids and three tertiary alcohols. Thus, six esters can coexist [1].



with

	R_1	R_2
Pyrethrine I	CH_3	$CH=CH_2$
Pyrethrine II	CO_2CH_3	$CH=CH_2$
Cinerine I	CH_3	CH_3
Cinerine II	CO_2CH_3	CH_3
Jasmoline I	CH_3	CH_2CH_3
Jasmoline II	CO_2CH_3	CH_2CH_3

Oil (triglycerides): evaporating the solvent after extraction provides an oil which is analysed by chromatography (C.P.G: Hewlett Packard 5890). In fact, it is not possible to measure the oil of the woad pastel by C.P.G; the fatty acid has to be transformed into methylic esters [1].

2.2. Equipment

The equipment considered consists of the following:

- An ultrasonic horn: the ultrasonic generator developed by UNDATIM operates with a variable power output and under an ‘automatic mode’, which seeks for an optimal frequency, ensuring maximum power transmitted to the reactor. For this study, two converters have been used corresponding to the frequencies of 20 and 40 kHz (Fig. 1).

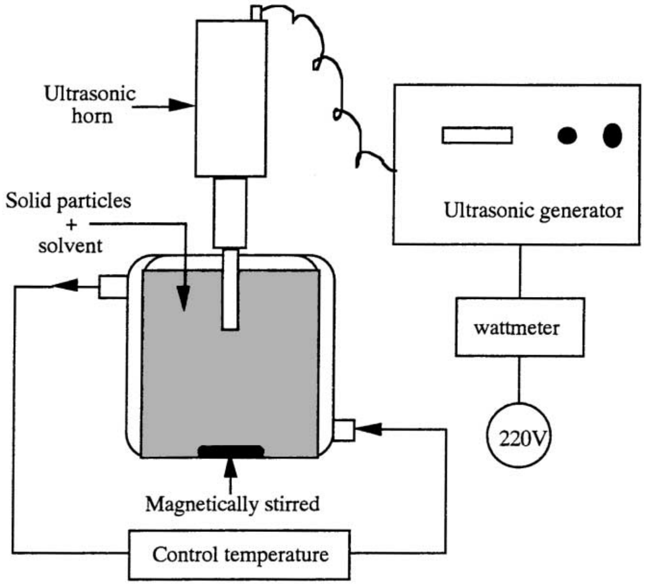


Fig. 1. Experimental device for solid–liquid extraction with ultrasound.

- An extractor: a stirred flask (0.300 l) in which solid and liquid are put into contact. The temperature of the medium is controlled by a thermostated bath.
- A watt meter used for measuring the electrical power consumed (Pcons).

The operating conditions depend on the mass and size distribution of solid particles, agitation (N), contact time (t), temperature (T), ultrasound (power, frequency), and the mass of the solid/solvent volume (ϕ).

The results are defined by the following:

- The extraction yield (E): weight of the solute extracted/weight of the initial solid particles,
- The purity of the extract (Pu): mass of the extracted pyrethrines/mass of the extract.

2.3. Thermoelectric probe

This is composed of a thermocouple embedded in an absorbent material (silicone) having the following dimensions (diameter $D = 0.004\,\mathrm{m}$; length $L = 0.005\,\mathrm{m}$ and depth $C = 0.003\,\mathrm{m}$) [9]. The thermoelectric probe is mounted on a rack gearing, which allows accurate displacement horizontally and vertically (Fig. 2). The modelling of the heat transfer allows the establishment of the relationship between the temperature signal response of the probe and the ultrasound intensity. It is shown that either the initial rate of temperature rise $(\mathrm{d}T/\mathrm{d}t)_0$ or the difference between the steady-state probe temperature T_{eq} and the medium temperature T_0 ($\Delta T = T_{\mathrm{eq}} - T_0$) is considered as being representative of the local ultrasound intensity [1,9]. A second rack gearing makes possible a precise positioning of the ultrasonic horn inside the extractor.

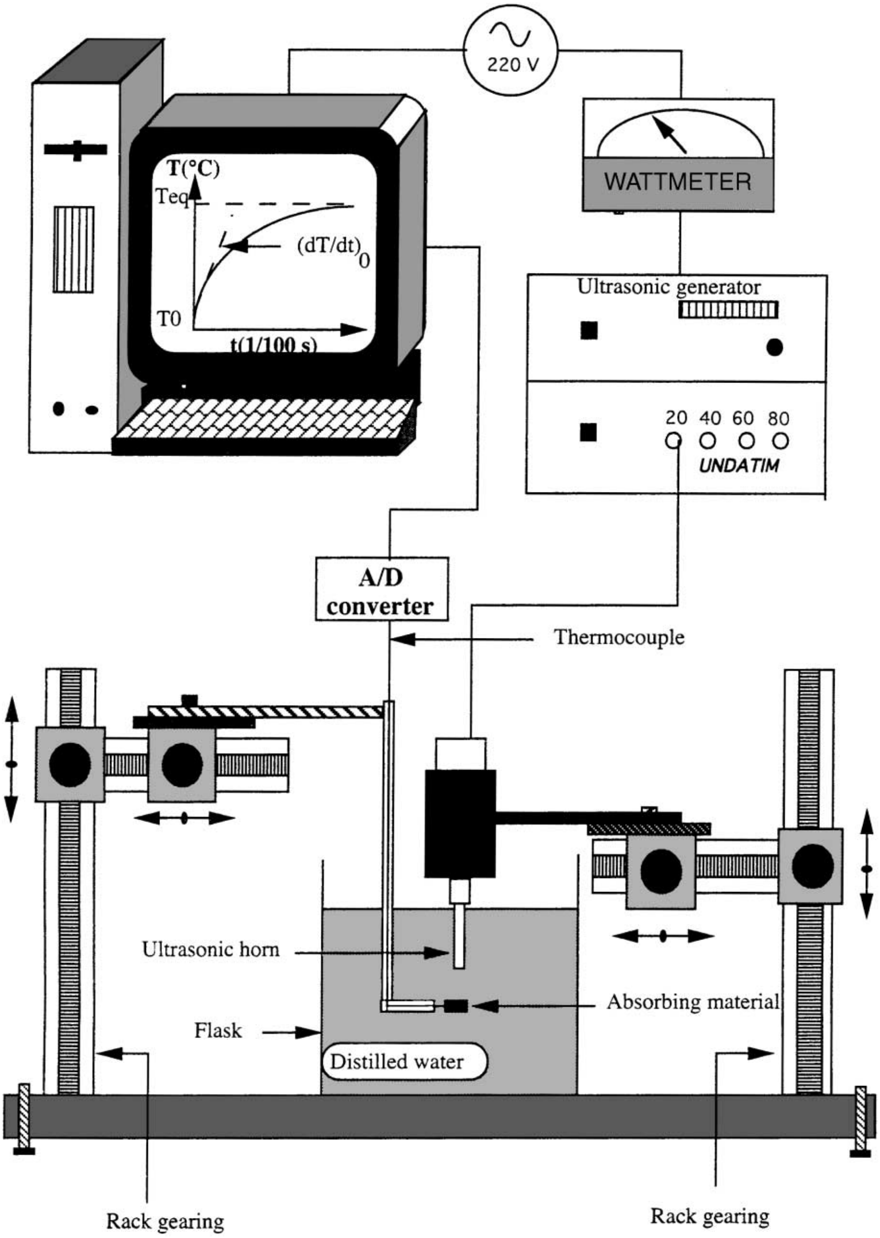


Fig. 2. Ultrasonic device for the measurement of local ultrasonic intensity.

3. Results and discussion

3.1. Extraction of pyrethrines from pyrethrum flowers

The study of the extraction of pyrethrines from pyrethrum flowers is difficult because of the many factors that can modify the results.

3.1.1. The origin of the flowers (country, area, year, etc.)

Gathering, drying and transport of the flowers can affect their initial composition [10]. In this work, three sets of flowers have been reduced to a powder and treated in the same conditions ($T = 40\,^{\circ}\mathrm{C}$, $N = 900\,\mathrm{rpm}$, $t = 60\,\mathrm{min}$). The results obtained are presented in Table 1. They are reproducible with a precision in the region of 7%. We can see that the extract is directly linked to the origin of the pyrethrum flowers. It is clearly shown that the larger extraction yield is obtained from the set No. 3, whereas no pyrethrines have been extracted in the case of the first set.

Table 1
Influence of the origin of the pyrethrum flowers on the yield of extraction

Pyrethrum flowers	Set No. 1	Set No. 2	Set No. 3
E (%)	0.05	0.60	0.80

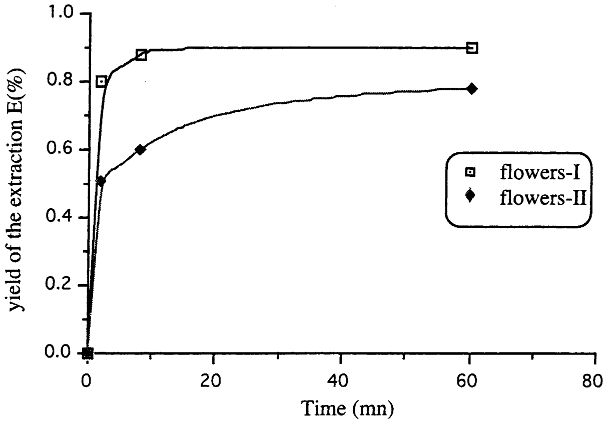


Fig. 3. Effect of ageing on the extraction kinetics of pyrethrines from crushed flowers.

3.1.2. Solid conditioning

Using flowers from the set No. 3, three series of crushed flowers have been investigated.

- Flowers I: flowers of day j, crushed and used on day j.
- Flowers II: flowers crushed on day j and used on day $j + 12$.
- Flowers III: flowers of day j, crushed and used on day $j + 20$.

In order to study the effect of the ageing of the pyrethrum flowers on the extraction yield, the results obtained with flowers I have been compared with those of flowers II, and those obtained with flowers I with those of flowers III. The results are presented in Figs. 3 and 4. In the case of crushed flowers (Fig. 3), a degradation of pyrethrines of about 13% is observed. It seems to be more important than the one obtained with the flowers not crushed (Fig. 4). Indeed, although the non-crushed flowers are older (20 days instead of 12 days), the decrease of yield is only 9%. In a previous work, it is shown that the degradation of pyrethrines can reach 20% with flowers aged 3 months [2]. This degradation is linked to the photolability of the flowers. It seems that a chemical action occurs between flowers, the air and the light; it is more important when the contact area is increasing. Consequently, in order to preserve the pyrethrum flowers under optimal conditions, any contact with air and light has to be avoided. In addition, the flowers have to be operated on immediately after crushing.

3.1.3. Sampling and size distribution

The third set has been prepared according to three different size distributions:

class 1: $1 \le d\,(\text{mm})$

class 2: $0.4 < d\,(\text{mm}) \le 0.56$

class 3: $d\,(\text{mm}) \le 0.4$

Fig. 5 shows the influence of the size distribution on the yield of extraction at 40 °C. It is clear that E increases with the decrease of the size of the solid particles. Indeed, increasing the contact area enhances the solute mass transfer.

But since the influence of the size distribution is so strong, it is possible to think that the amount of solute differs according to the size classes. This observation is based on the natural non-uniformity of the flowers. Indeed, the pyrethrum flowers are composed of different parts (stamen, corolla, stem, etc.) having different initial solute. When the flowers are crushed, these parts are not uniformity distributed among the three classes. For example, the corolla may have a great resistance to the crushing; then it will form the first class (great size). But, the quantity of the solute located on the corolla is very small; this may explain the weak yield obtained with the largest particles. On the other hand, the stamens in which the pyrethrines are concentrated, are easy to crush, will form the major part of the class 3 (smallest size).

In order to distinguish the influence of size distribution from that of the nature of the flower (stamen, corolla, etc.),

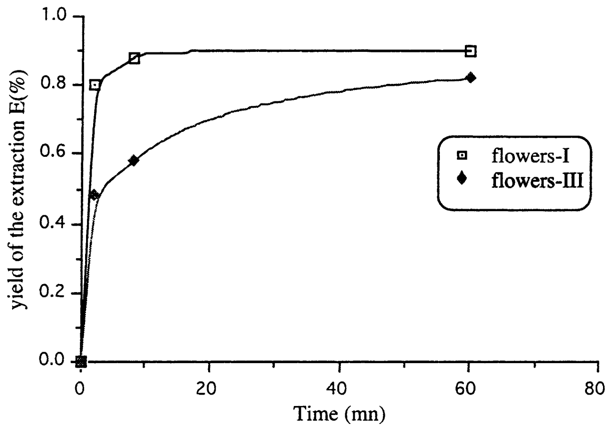


Fig. 4. Effect of ageing on the extraction kinetics of pyrethrines from uncrushed flowers.

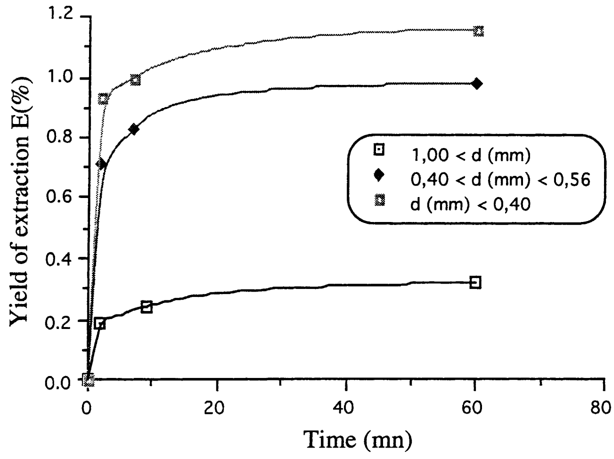


Fig. 5. Influence of size distribution on the extraction kinetics of pyrethrines from pyrethrum flowers.

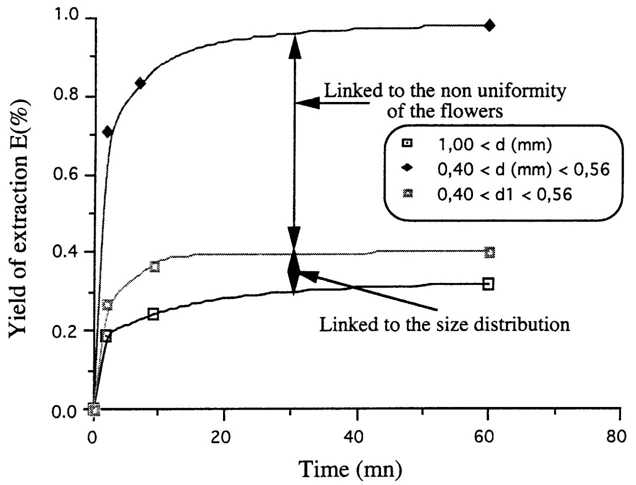


Fig. 6. Influence of distribution and the non-uniformity of the flowers on the extraction kinetics of pyrethrines from pyrethrum flowers.

the particles of class 1 have been crushed to obtain class 4 ($0.4 < d1\,(\mathrm{mm}) \leq 0.56$). Fig. 6 shows that the difference between the two lower curves is linked to the influence of the size distribution, whereas the difference between the upper curves is linked to the nature of the particles. Finally, it is possible to conclude that the difference of the yield obtained for different particle sizes (Fig. 5) is essentially caused by the nature of the particles.

The results obtained with the class 2 are reproducible. Consequently, this class has been selected for further work. Taking into the account, the different observations mentioned above, the same solid sample has been studied in order to perform valid comparisons between the results.

3.1.4. Optimal extraction conditions

The temperature, the particle size and the rotation speed of the agitator obviously have a strong influence on extraction yield E [1]. The operating conditions that lead to the optimal yield are: $T = 40\,^{\circ}\mathrm{C}$, $\phi = 40\,\mathrm{kg/m^3}$, $0.4 < d\,(\mathrm{mm}) \leq 0.56$ and $N = 1000\,\mathrm{rpm}$.

In fact, the extraction yield increases with the rise in temperature [2] and the temperature that leads to the maximum yield is 66 °C (evaporating temperature of the hexane at $p = 1\,\mathrm{atm}$). But, with ultrasound, this increase is not profitable for temperatures between 40 and 66 °C. That is why we have chose $T = 40\,^{\circ}\mathrm{C}$ as an optimal temperature.

3.1.5. Soxhlet

In order to determine the initial amount of solute, a soxhlet extraction has been made with different contact times. In Table 2, the maximum E value obtained is 1.24%. This latter result is the average of four experiments with a precision of 5%.

Table 2
Yield and purity of pyrethrins extracted from pyrethrum flowers by soxhlet

Time (min)	Yield of extraction, E (%)	Purity of the extraction, Pu (%)
30	0.67	19
180	0.70	20
1500	1.24	24
2400	1.23	25

3.1.6. Influence of ultrasound on the yield of extraction

In the extractor, different experiments were performed ($T = 40\,^{\circ}\mathrm{C}$, $V_{\mathrm{hexane}} = 25 \times 10^{-5}\,\mathrm{m^3}$, $N = 1000\,\mathrm{rpm}$, $\phi = 40\,\mathrm{kg/m^3}$). The results are presented in Table 3. They show the gain in the extraction yield which is due to the ultrasonic activation. The value of E has been increased by 40% for 60 min contact time. Using the same horn corresponding to a frequency of 20 000 Hz, a small change in frequency (≈200 Hz) can have a great effect on the extraction yield. Indeed, the increase of E is about 32%. The UNDATIM apparatus has an 'automatic mode' which seeks for the optimal frequency, thus ensuring maximum power to be transmitted to the extractor. In order to confirm this result, the following experiment has been carried out.

The flask used for the extraction was filled with distilled water ($V = 25 \times 10^{-5}\,\mathrm{m^3}$). A thermoelectric probe was placed at a distance of 0.002 m from the transmitting surface of the ultrasonic horn (Fig. 7). This latter is connected to an UNDATIM generator working in 'automatic mode'. The response of the thermoelectric probe during the search for the optimal frequency is shown in Fig. 8 [1,7]. A variation

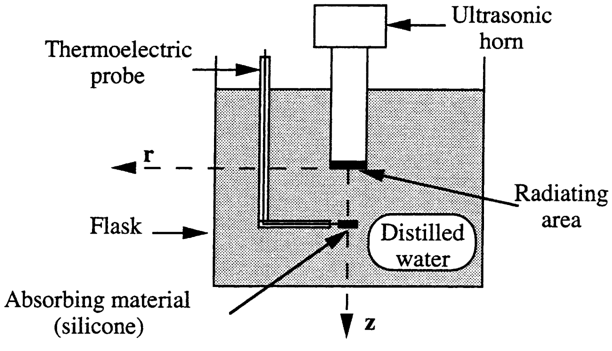


Fig. 7. Ultrasonic horn ($f = 20\,\mathrm{kHz}$).

Table 3
Influence of ultrasound on the yield of extraction E (%) ($t = 60\,\mathrm{min}$, $T = 40\,^{\circ}\mathrm{C}$, $N = 1000\,\mathrm{rpm}$)

	Without ultrasound	With ultrasound fixed frequency, $f_{\mathrm{f}} = 20\,000\,\mathrm{Hz}$	With ultrasound automatic frequency, $f_{\mathrm{a}} = 20\,223\,\mathrm{Hz}$
E (%)	0.52	0.80	1.18
Pu (%)	20	27	10

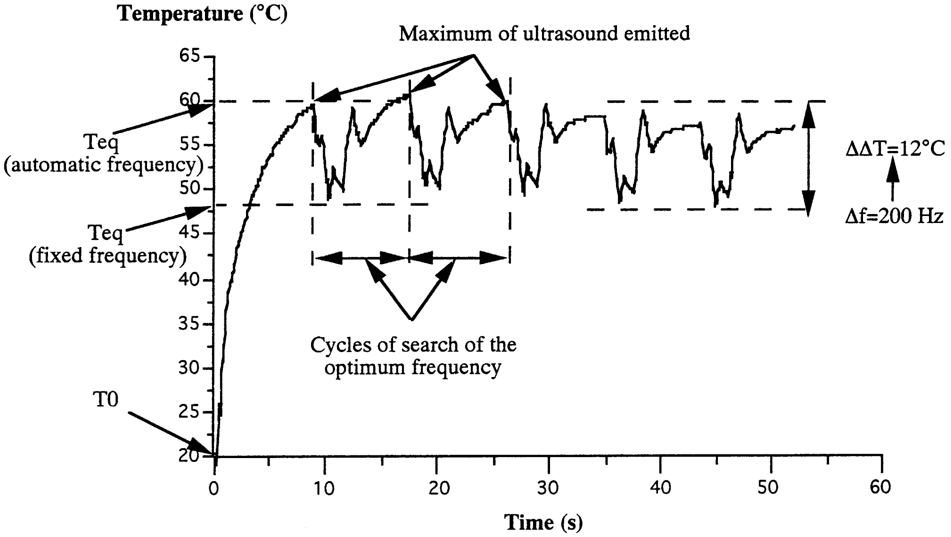


Fig. 8. Influence of frequency (automatic mode) on the response of the thermoelectric probe.

in temperature of 12 °C has been recorded in relation to a variation of 200 Hz. It can be observed that the maximum temperature is reached at the optimal frequency, corresponding to the maximum ultrasound power transmitted.

Since the ΔT measured is directly proportional to the ultrasonic power, using automatic frequency operation mode leads to an increase in power of 30% compared to the fixed frequency mode. This increase was estimated as follows:

$$\left.\begin{array}{ll} -f_{\mathrm{f}}: & \Delta T_{\mathrm{f}}=(T_{\mathrm{eq}}-T_0)=29\,^{\circ}\mathrm{C} \\ -f_{\mathrm{a}}: & \Delta T_{\mathrm{a}}=(T_{\mathrm{eq}}-T_0)=40\,^{\circ}\mathrm{C} \end{array}\right\} \Rightarrow \%\left(\frac{\Delta\Delta T}{\Delta T_{\mathrm{a}}}\right)$$
$$=\left(\frac{12}{40}\right)\times 100=30\%, \quad -\Delta\Delta T=12\,^{\circ}\mathrm{C}$$

Thus the increase of 32% in the extraction yield should be due to the increase of 30% in power. There is probably no linear relationship between the increase in power and ultrasound intensity. Indeed, in a previous work [2,10], using an ultrasonic tank, an increase in power of 50% generated only a 25% increase in extraction yield.

At 40 and 66 °C, four successive extraction steps have been performed with and without ultrasound. For an exposure of 1 h, the solids ($m = 7.27 \times 10^{-3}$ kg) are put into contact with the pure solvent ($V = 25 \times 10^{-5}\,\mathrm{m}^3$). The results in Fig. 9 show that the amount of solute extracted from the solid particles is not 1.20% as found with the soxhlet but 1.64%. It implicitly means that because of the ultrasound it is possible to reach yields larger than the one with the soxhlet, which is very often considered as a limiting value. We can also remark that without ultrasound, even if we work at a temperature of 66 °C, considered optimal, the yield obtained is less than the one achieved at 40 °C with ultrasound.

Fig. 10 shows the influence of ultrasound on the extraction kinetics of the pyrethrines from the pyrethrum flowers ($T = 40\,^{\circ}\mathrm{C}$, $V_{\mathrm{hexane}} = 25 \times 10^{-5}\,\mathrm{m}^3$, $m = 6.95 \times 10^{-3}$ kg). It is clearly demonstrated that ultrasound ameliorates simultaneously the kinetics and the yield of the extraction.

The good results obtained with ultrasound in the different experiments are probably linked to the increase in the

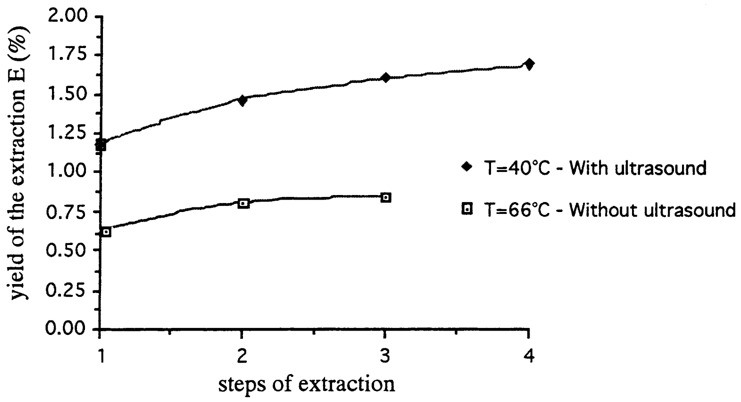


Fig. 9. Influence of ultrasound on the extraction yield of pyrethrines from pyrethrum flowers.

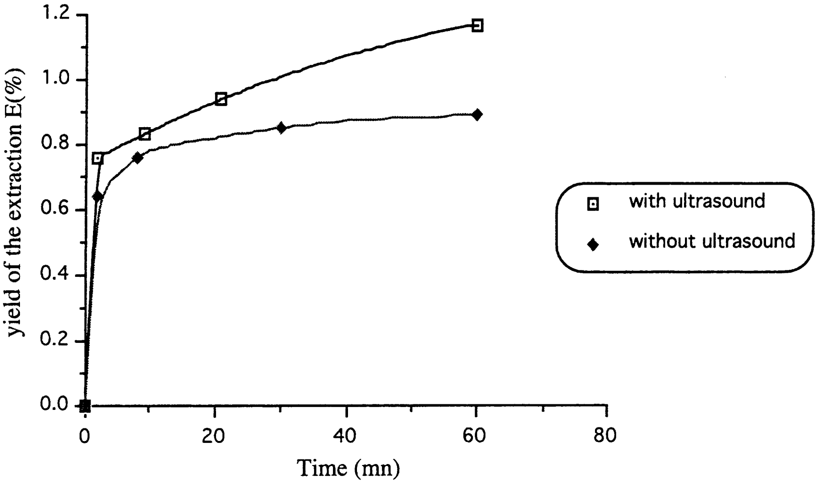


Fig. 10. Influence of ultrasound on the yield and kinetics of the extraction of pyrethrines from pyrethrum flowers ($T = 40\,^{\circ}\mathrm{C}$, $V_{\mathrm{hexane}} = 25 \times 10^{-5}\,\mathrm{m}^3$, $m = 6.95 \times 10^{-3}\,\mathrm{kg}$, $f = 20\,000\,\mathrm{Hz}$).

internal diffusion which controls the transfer of the solute to the solvent, and also to the destruction of pores in which the solute can be trapped.

3.2. Extraction of oil from woad seeds

3.2.1. Optimal conditions of extraction

In a first step, the influence of the classical parameters was studied. The results obtained show that the temperature *T*, the size of particles *d*, and the intensity of mixing have an influence on the yield and on the extraction kinetics [1]. The optimal parameters obtained are: $T = 40\,^{\circ}\mathrm{C}$, $d < 4 \times 10^{-4}\,\mathrm{m}$, $N = 900\,\mathrm{rpm}$ and $\phi = 136.4\,\mathrm{kg/m^3}$.

3.2.2. Influence of ultrasound

Fig. 11 shows that ultrasound has a weak effect both on the yield and on the kinetics of extraction. Indeed, the gain is only around 10%. This result could be linked to a wrong use of the ultrasound (frequency and intensity). Consequently, the ultrasound parameters have been studied. But the results remain unsatisfactory since the yield of extraction is practically unchanged. As an example, some results according to the power consumption, Pcons (Pcons = 60, 100 and 170 W) are plotted in Fig. 12. Similar results (Fig. 13) were obtained whether the frequency *f* is 20 000 or 40 000 Hz.

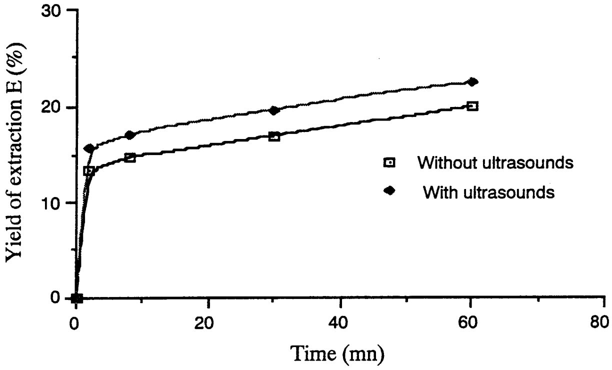


Fig. 11. Influence of ultrasound on the yield and extraction kinetics of oil from woad seeds.

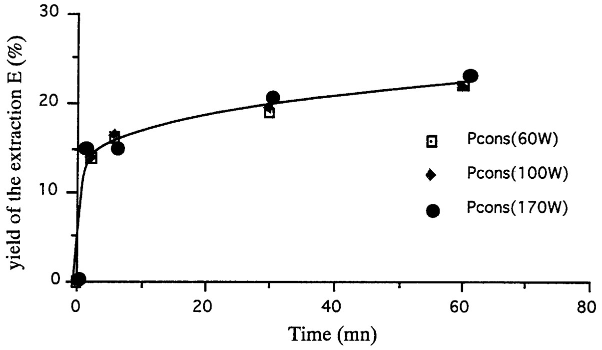


Fig. 12. Influence of power consumption on the yield and kinetics of oil extraction from woad seeds.

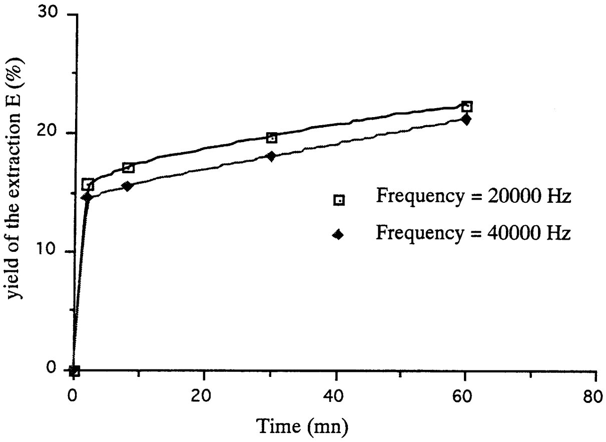


Fig. 13. Influence of ultrasonic frequency on the yield and kinetics of oil extraction from woad seeds.

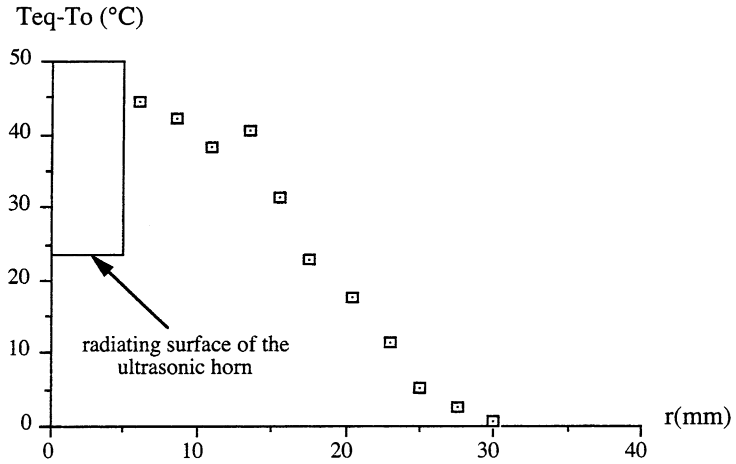


Fig. 14. Radial ultrasound intensity profile generated with an ultrasonic horn ($f = 41\,600\,\mathrm{Hz}$). Measurements were made in a flask ($V = 5 \times 10^{-3}\,\mathrm{m}^3$), in which the height of the distilled water was fixed at 17 cm.

4. Comment on ultrasonic attenuation

During the former study of extraction assisted by ultrasound, the results differ from one another according to the raw material involved (flowers or seeds). This result is probably linked to the nature of the solid: hardness, compactness and solute distribution. This is probably the main reason that conditions the interest of ultrasonic activation, but not the only one.

The key parameter lies also in the ultrasonic wave distribution inside the extractor with and without the presence of solid particles. Therefore, it seems crucial to undertake a systematic study of the coupling between the ultrasonic emitters, the sonicated medium and the reactor, in order to understand the more or less positive effect of the ultrasound and to help towards the design of an ultrasonic reactor. To this end, it is necessary to develop reliable techniques capable of measuring the local ultrasound intensity [11–14]. Among others, the thermoelectric probe is one of the most appropriate techniques. It has been studied and developed in our laboratory [1,9].

By this method, it is possible to determine the boundaries of the active zone, in which the ultrasonic intensity is the higher. Using the thermoelectric probe, we measured the radial ultrasound intensity profile distribution generated by an ultrasonic horn ($f = 20$ or $40\,\mathrm{kHz}$). The result obtained shows that the active zone is fixed in a cylinder of 6 cm in diameter (Fig. 14).

In an earlier work [1,15], the maximum ultrasound power was observed at the vicinity of the radiating surface of the ultrasonic horn. The decrease of the ultrasonic intensity is rather abrupt as the distance from the radiating surface increases.

Furthermore, the presence of solid particles can affect the ultrasonic intensity profile. Measurements have been performed in a container filled with distilled water as liquid and sunflower shells as solid. The solid is maintained in suspension, and the medium is sonicated by an ultrasonic horn (Fig. 1). Fig. 15 shows that the ultrasound intensity profiles, measured along the central axis of the horn, are attenuated with the increase of the phase ratio ϕ [16].

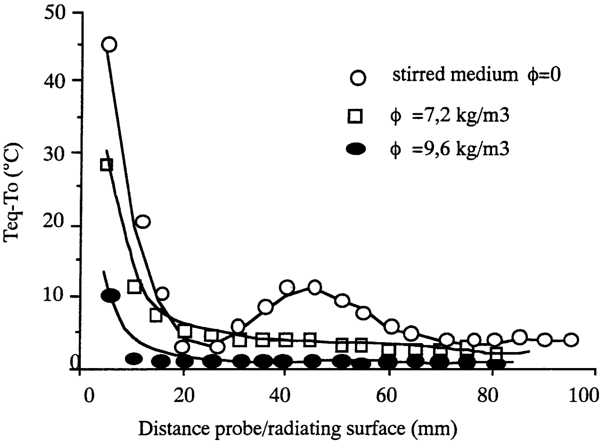


Fig. 15. Influence of solid particles (sunflower shells) on the ultrasonic intensity profile measured along the central axis of the horn.

5. Conclusion

The extraction of pyrethrines from pyrethrum flowers and oil from woad seeds has been studied. In each case, the operating conditions leading to the optimal yield have been obtained. In the case of the pyrethrum flowers, the preservation, storage, sampling and size distribution of the solid particles seem to have a great influence on the extraction yield.

The influence of ultrasound on the solid–liquid extraction process as regards yields or selectivity has also been studied here. Such influence is very difficult to predict because of the interaction of many factors, either relative to the phase system (solid/liquid/solute) or to the ultrasonic reactor itself. Two examples are given in the present paper in order to illustrate the effect of ultrasonic activation according to the nature of the solid particles (flowers or seeds). Furthermore, a technique of investigation of the ultrasonic intensity profile has been used based on a thermoelectric probe. It is shown clearly that the presence of a dispersed phase contributes to the ultrasound wave attenuation and that the active part sonicated inside the reactor is restricted to a zone located in the vicinity of the emitter. These two conclusions have great consequences for the operation of extractors and must be carefully considered in the design of ultrasonic extractors.

References

[1] M. Romdhane, Extraction solide–liquide sous ultrasons—mise au point d'un capteur de puissance ultrasonore, Thèse, Institut National Polytechnique de Toulouse, 1993.

[2] C. Haunold, Extraction des pyréthrines—analyse du procédé discontinu et de l'influence des ultrasons. Modélisation et mise en æuvre d'un procédé continu, Thèse, Institut National Polytechnique de Toulouse, 1991.

[3] M. Hiraide, Y. Mikuni, H. Kawaguchi, Solid–liquid extraction with an ammoniacal EDTA solution for the separation of traces of copper from aluminium, Anal. Sci. 11 (4) (1995) 689–691.

[4] Li. Hui, O. Etsuzo, I. Masao, Effects of ultrasound on extraction of saponin from ginseng, Jpn. J. Appl. Phys. Part 1 33 (5B) (1994) 3085–3087.

[5] Y.E. Primo, B.F. Lafuentte, R.P. Perez, C.I. Oriol, Y.E. Jorro, Spanish Patent 384 195 (1973).

[6] A.E. Demaggio, J.A. Lott, J. Pharmacical Sci. 53 (8) (1964) 945–949.

[7] L.C. Wang, J. Food Sci. 44 (1975) 549–554.

[8] G.B. Aimukhamedova, G.M. Korneva, Izv. Akad. Nauk Kirg. 4 (1962) 17.

[9] M. Romdhane, C. Gourdon, G. Casamatta, Development of a thermoelectric sensor for ultrasonic intensity measurement, Ultrasonics 33 (2) (1995) 139–146.

[10] C. Haunold, J. Berlan, C. Gourdon, G. Casamatta, G. Barale, Solid–liquid extraction assisted by ultrasound, in: Proceedings of the Ultrasonics International Conference, 1989, pp. 1289–1294.

[11] B. Pugin, Qualitative characterization of ultrasound reactors for heterogeneous sonochemistry, Ultrasonics 25 (1987) 49–55.

[12] K.S. Suslick, P.F. Schubert, J.W. Goodale, Chemical dosimetry of ultrasonic cavitation, in: Proceedings of the IEEE Ultrasonics Symposium, 1981, pp. 612–616.

[13] B. Fay, M. Rinker, A. Lewin, Thermoacoustic sensor for ultrasound power measurements and ultrasonic equipment calibration, Ultrasound Med. Biol. 20 (4) (1993) 367–373.

[14] C.J. Martin, A.N.R. Law, The use of thermistor probes to measure energy distribution in ultrasound field, Ultrasonics 18 (1980) 127–133.

[15] M. Romdhane, C. Gourdon, G. Casamatta, Local investigation of some ultrasonic devices by means of a thermal sensor, Ultrasonics 33 (3) (1995) 221–227.

[16] M. Romdhane, C. Gourdon, G. Casamatta, The influence of ultrasound in solid–liquid extraction, Value Adding Through Solvent extraction, Volume 2, Proceeding of ISEC (1996) 1245–1250.