*Int. Agrophysics, 2008, 22, 133-137*

INTERNATIONAL
**Agrophysics**
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# Dry matter extraction from valerian roots (*Valeriana officinalis* L.) with the help of pulsed acoustic field

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*Received March 4, 2008; accepted April 18, 2008*

A b s t r a c t. The paper presents results concerning ultrasonically assisted extraction of bioactive compounds from roots of valerian. Pulsed ultrasonic field was applied. The investigation was accomplished by varying irradiation time and sonic power. The results were compared with classical (silent) extraction. The study included evaluation of the coefficient of rehydratation, yield of extraction, dry matter content in extract and residue, and unit energy consumption. The utilisation of pulsed ultrasound was proved to be a more efficient technique than the classical method for extraction of bioactive components from dried valerian roots.

K e y w o r d s: pulsed ultrasonic field, extraction, dry matter, valerian

## INTRODUCTION

Valerian (*Valeriana officinalis* L.) is one of the basic and widely cultivated herbal plants in Poland. Its medicinal properties have been already known in ancient Greece and Rome. Extracts from valerian have a broad range of applications. The first main use of valerian is as a tranquillizer for people with hyperexcitability. The second use of valerian is as a smooth-muscle relaxing agent. This action is applied to treatment of stomach and intestine cramp and so-called vegetative neurosis (Leśniewicz *et al*., 2006). Valerian is also a component of many herbal mixtures which are widely used to treat sleeping disorders (Bent *et al*., 2006).

The method for obtaining bioactive compounds from valerian is maceration. Due to the instability of active compounds the process is conducted at temperatures between 20 and 30°C. The low temperature causes that the extraction process is of a small yield and time-consuming. Hence, methods are being sought which will reduce treatment time and simultaneously increase the yield of extraction. One of them may be the use of an ultrasound field (Mason *et al*., 1996; Povey and Mason, 1998; Sališová *et al*., 1997; Śliwiński, 2001; Mason and Lorimer, 1989). The mechanism of intensification of extraction process in the presence of ultrasound is that the ultrasonic waves break the cells and release the contents of cells into the extraction solvent (Toma *et al*., 2001; Wilson and Walker 1995). Recently, the use of ultrasonics for solid-liquid extraction is becoming increasingly popular (Ebringerovà and Hromàdkovà, 1997; Hromadkova *et al*., 2002; Paniwnyk *et al*., 2001; Romdhane and Gourdon, 2002; Śliwiński, 2001; Toma *et al*., 2001, Valachovic *et al*., 2001; Vinatoru, 2001; Vinatoru *et al*., 1997).

However, continuous ultrasounds cause fast heating of treatment mixture and require intensive cooling during extraction. One of the ways to limit the very fast increase of temperature is the use of pulsing operation. This technique was applied in medicine (Crisci and Ferreira, 2002; Qin *et al*., 2006). The pulsing operation prevents the overheating of tissues during ultrasonic therapy. Apart from that, in some conditions, the pulsing operation can be even more effective in comparison with continuous ultrasounds (Leighton, 1994; Mitome and Hatanaka, 2002). Application of pulsed ultrasound slows down the increase of temperature in the treatment mixture and, as a consequence, reduces costs of cooling.

Until now pulsed ultrasounds have been investigated only in sonochemical reactions (Henglein, 1995). There is no information about the use of pulsing operation to obtain extracts from herbal plants in scientific literature. Because of this, the aim of this study was to investigate the influence of pulsed ultrasonic field on extraction of dry matter from valerian roots and to compare the results with the classical method (maceration).

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MATERIALS AND METHODS

The research materials were roots of valerian (Fig. 1). The raw material came from a plantation located in the Lublin Upland, where it is widely cultivated.

The research was conducted according to the scheme presented in Fig. 2.

Dried valerian was ground in colloid mill WŻ-1. Then, the material was divided into fractions using a laboratory sieving device. One fraction with particle size of 0.25-0.5 mm was taken for testing.

Classical extraction was performed in a thermostat by heating the solid-liquid mixture in an Erlenmeyer flask in a temperature of 25ºC. The solution was not stirred during heating. The time of extraction was 15, 30, 45 and 180 min. The control extraction is represented by the abbreviation c.e. in the Figs 3-6.

Ultrasonication was carried out using an ultrasonic processor (Sonic VCX 750) at a frequency of 20 kHz. The generator was a horn-type (25 mm diameter and 122 mm length). All experiments were performed on samples of 10 g dispersed in 100 ml of water without additional stirring. The parameters of ultrasonic treatment were as follows:

– radiated sonic power – 3.2, 4.8, and 6.2 W $cm^{-2}$;
– length pulse – 2 s, interval between pulses – 1 s;
– times of ultrasonic treatment – 10, 20, 30 and 120 min that correspond with total times of extraction of 15, 30, 45 and 180 min, respectively.

The temperature of the medium during extraction was kept at 25±2°C.

Coefficient of rehydratation *(R)* was calculated using the following formula:

$$R = \frac{\text{initial of the dried valerian (g)}}{\text{mass of the dried valerian after steeping (g)}} 100\%. \quad (1)$$

Yield of extraction *(Y)* was calculated using the following formula:

$$Y = \frac{\text{dry matter content in the exctract (g)}}{\text{mass of the intial solid particles (g)}} 100\%. \quad (2)$$

**Fig. 1.** Outlook appearance of valerian roots.

Dry matter content in the extract was determined by drying at 105°C till constant weight was achieved in accordance with the Polish Standard PN–90/A75101/03.

The unit energy consumption *(N)* was calculated using the following formula:

$$N = \frac{\text{total amount of emitted energy (J)}}{\text{dry matter content in the extract (g)}}. \quad (3)$$

The total amount of emitted energy was obtained from the energy monitor of the VCX 750.

The experiments were carried out in 3 replications. Achieved results were subjected to statistical processing applying variance analysis. The difference significance was tested by means of the Tukey test. All computations were made using Statistica 6.0 software.

RESULTS AND DISCUSSION

**Influence of ultrasound on rehydratation coefficient**

Swelling of material is the first stage of extraction in the case of dried material. The capacity of dried material for absorption of water can be expressed by a rehydratation coefficient. The quantity of absorbed water has a great influence on leaching velocity of solute from matrix of solid. Ultrasound can accelerate the swelling process, so the influence of sonication on the rehydratation coefficient was investigated. The influence of ultrasonic treatment on rehydratation coefficient is shown in Fig. 3.

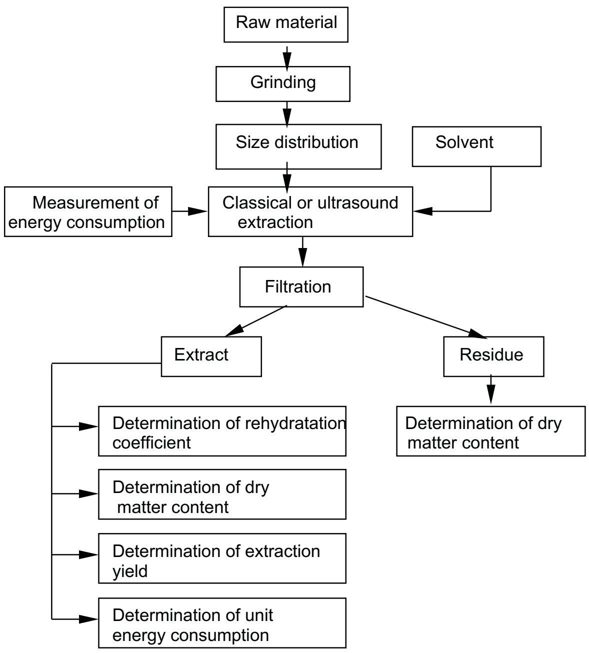


**Fig. 2.** Scheme of research schedule.

In the case of dried valerian, pulsed ultrasound had a weak influence on the rehydratation coefficient. There were only small significant differences in the rehydratation coefficient between ultrasound samples obtained after 45 min and 180 min at 6.2 W cm$^{-2}$ sonic power. There were no significant differences between other ultrasound and control samples.

It is worth to notice that changes of the rehydratation coefficient in time were very small. Dried valerian absorbed water very fast, immediately after immersion, during the first 15 min. Further soaking of dried material caused only a small increase of absorbed water. Because of this, pulsed ultrasound has a small influence on rehydratation coefficient.

### Influence of ultrasound on the yield of extraction

The influence of sonic power and time of treatment on the yield of dry matter from valerian roots is presented in Fig. 4. The sonication effect on yield of extraction was dependent on the intensity of ultrasound and time of irradiation. There were significant differences between the classical and ultrasonic extraction for each time of treatment. Influence of sonic power on yield of extraction was dependent on time of irradiation. For short time of treatment (15 and 30 min), there were no significant differences in the yield of extraction. For longer time of treatment there were differences between the sample at 6.2 W cm$^{-2}$ sonic power and the samples at 3.2 and 4.8 W cm$^{-2}$ sonic power. The highest yield of extraction was obtained after 180 min of treatment at 6.2 W cm$^{-2}$ sonic power.

The higher efficiency of the ultrasound-assisted extraction can be explained by mechanical action of acoustic field on the cell walls, resulting in increased extractability of dry matter from valerian. In the case of continuous ultrasounds, such effects were known to facilitate improved extraction of various components from plant materials (Romdhane and Gourdon 2002).

### Influence of ultrasound on dry matter content in extract

The influence of ultrasound on dry matter content in the extract is presented in Fig. 5.

The dry matter content in the extract increased after ultrasonic treatment. There were significant differences between classical and ultrasonic extraction. The differences were visible with the naked eye. The extracts from the classical extraction were light brown and from the ultrasonic extraction were dark brown coloured. The influence of ultrasound intensity on dry matter content was dependent on time of irradiation. The sonic power had an influence on dry matter content in the extract only for the longest time of treatment (180 min). There were no significant differences between ultrasonic samples for other times of sonification. The highest dry matter content in the extract was obtained after 180 min of treatment at 6.2 W cm$^{-2}$ sonic power.

### Influence of ultrasound on dry matter content in the residue

To confirm the intensifying effect of pulsed acoustic field during extraction the dry matter content in the residue was investigated. The dry matter content in the residue decreased with increasing time period of ultrasonic treatment. There were significant differences between classical and ultrasonic extraction. The highest decrease of dry matter

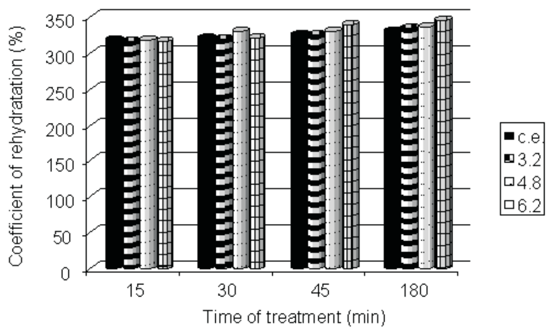


**Fig. 3.** Influence of sonic power and time of irradiation on rehydratation coefficient.

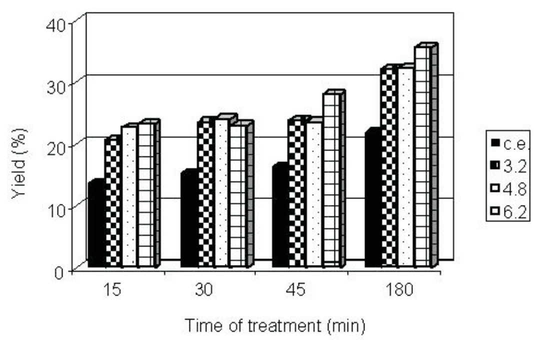


**Fig. 4.** Influence of sonic power and time of irradiation on yield of extraction.

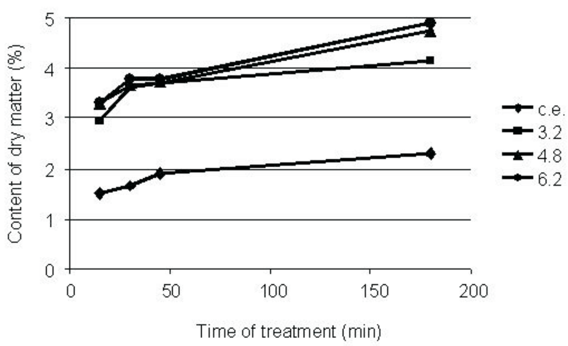


**Fig. 5.** Influence of sonic power and time of irradiation on dry matter content in extract.

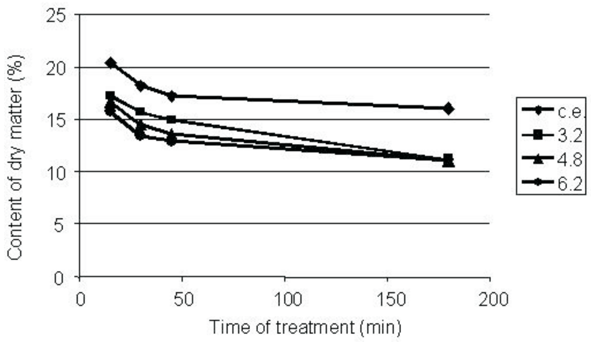


**Fig. 6.** Influence of sonic power and time of irradiation on dry matter content in residue.

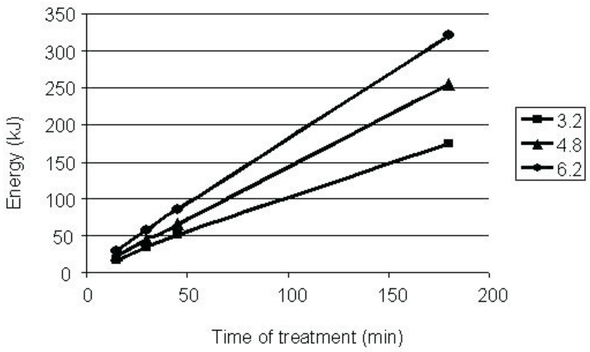


**Fig. 7.** Energy emitted during ultrasonic extraction.

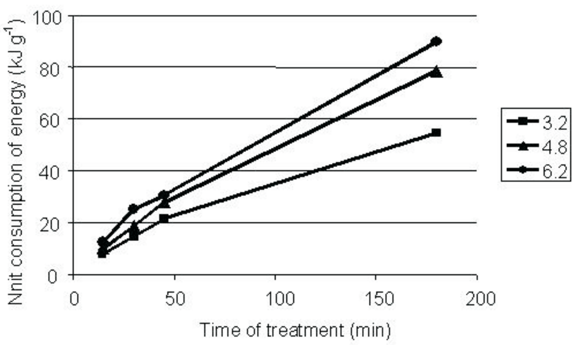


**Fig. 8.** Unit consumption of energy during ultrasonic extraction.

content in residue was obtained after 180 min of ultrasonic treatment. There were no differences between intensities of ultrasounds for this time. Similar effects were also observed by other authors in the case of continuous ultrasounds (Hromadkova *et al*., 2002). They affirmed lower yield of extraction from residue obtained after ultrasonic treatment in comparison with residue obtained from classical extraction. The decrease of dry matter content in residue confirms the effectiveness of pulsed acoustic field in extraction of valerian roots (Fig. 6).

**Influence of ultrasound on unit consumption of energy**

The quantity of emitted energy during sonication is dependent on various factors, such as viscosity and temperature of medium, size and concentration of solids immersed in liquid. Some of these parameters can be changed during treatment. Apart from that, some ultrasonic devices can adjust sonic power to the resistance of sample by keeping a constant amplitude of ultrasonic vibrations. Because of this it is important to determine the total quantity of energy emitted to treatment medium. The energy emitted by the ultrasonic device during extraction is presented in Fig. 7.

The dependence between time of treatment and energy consumption is a straight line, so it can be assumed that during sonication the resistance of the sample was constant.

Figure 8 demonstrates unit energy consumption related to the amount of dry matter extracted from dried valerian (*N*). This parameter describes well the effectiveness of extraction from energy point of view. There were significant differences in unit energy consumption between ultrasonic treatments. The lowest energy consumption was obtained at the lowest sonic power.

CONCLUSIONS

1. The utilisation of pulsed ultrasound has proved to be a more efficient technique than classical operation in extracting bioactive components from dried valerian roots.

2. The use of a pulsed acoustic field allowed to reduce the intensity of mixture cooling during the irradiation.

3. Pulsed ultrasounds accelerated the leaching of bioactive compounds from a matrix of solids.

4. Because the differences between ultrasonic and classical extraction in the coefficient of rehydratation were small, whereas the differences in yield of extraction were higher, it is supposed that the pulsed ultrasound affects more the second stage of extraction.

5. The yield of extraction depended on the sonic power and the time of treatment. The highest yield was observed after 180 min at 6.2 W $cm^{-2}$ sonic power.

6. Pulsed ultrasonic treatment increased also dry matter content in obtained extract. Apart from that, the effectiveness of ultrasonic field was also confirmed by the decrease of dry matter content in residue.

7. Taking into account the unit energy consumption related to the amount of dry matter extracted from dried valerian, the best results were obtained at the shortest time of ultrasonic treatment and the lowest sonic power.

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