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STUDIES UPON THE ULTRASONIC EXTRACTION PROCESS ON AN EXAMPLE OF DRY MATTER EXTRACTION FROM DRIED CARROTS

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Summary. The research deals with an analysis of an influence of acoustic field intensity and dried material's breaking up during the extraction process. The obtained data were compared to results from classical extraction. A process of ultrasonic extraction was performed without cooling of the tested water – dried carrots system. It was found that in the case of intensities ranging from 6.8 to 11.2 W/cm^2 and up to 20 minutes of processing time, the ultrasonic extraction gave better effects referring to separation of soluble substance than the classical method.

Key words: ultrasounds, extraction, dried carrots

INTRODUCTION

Carrot is one of the most common and worldwide-spread root crops. Its share in the global vegetable production exceeds 3%. Also in Poland, it is commonly cultivated and utilized in foodstuff processing. Carrot is most important due to its high level of vitamins and minerals. It serves for production of juice, meals, frozen products as well as a component of many fresh and pasteurized salads. Dried carrots are used for production of many vegetable-based spices. Its application in cosmetics and phytotherapy is also known – it is used as a component of face masks for skin regeneration, shampoos for hair nutrition and creams production. Extracts made from carrot are valuable products as well. Substances contained in carrot show strong action that stimulates the metabolism and urine production. They also increase a general organism's resistance. Therefore, carrot extracts may be applied for treatment of many disturbances of digestive tract, namely at children and infants [Kołota and Orłowski 1993, Pijanowski *et al.* 1978].

Producing the extracts from plant material is difficult due to their complex structure, which results in low efficiency and long time of the process. Therefore, new methods of extraction intensification are searched for.

Application of ultrasounds is a potential way for extraction efficiency improvement. Ultrasonic field invokes many phenomena that may positively affect the process kinetics: increase of processed medium temperature, viscosity decrease, micro-floats and in the case of properly selected parameters, also cavitation. Thus, ultrasonic extraction finds its broad application for separation of various organic substances [Mason *et al.* 1996, Ebringerovà and Hromàdkovà 1997, Sališová *et al* .1997, Vinatoru *et al.* 1997, Hromàdkovà *et al.* 1999, Vinatoru *et al.* 1999, Valachovic *et al.* 2001, Vinatoru 2001, Hromadkova *et al.* 2002, Hromàdkovà and Ebringerovà 2003].

Toma *et al*. [2001] studied an influence of acoustic field at 33 kHz frequency and 1 W/cm^2 intensity on extraction efficiency and structural changes of various plant materials: peppermint, hop, dill, pot marigold and lime. They found an increase of the efficiency as compared to classical extraction in almost all cases.

Romdhane and Gourgon [2002] conducted experiments upon an influence of ultrasonic field on extraction of pyrethrines from *Pyrethrum* flowers and oils from woad seeds. Their studies revealed that the breaking up level and type of raw material subjected to the process had a great effect on ultrasonic extraction efficiency. Hromàdkovà and Ebringerovà [2003] using hemicellulose from buckwheat shells observed an influence of temperature and solvent concentration on ultrasonic processing efficiency. Paniwnyk *et al.* [2001] in his study upon rutin extraction from *Saphora japonica* found the impact of solvent type, and Entezari *et al.* [2004] in his attempts to achieve juice from dates, recorded an influence of acoustic field intensity on the process efficiency.

Many factors affect the course and efficiency of extraction using ultrasounds. They are such parameters associated with acoustic field as: wave frequency, ultrasound intensity, acoustic energy density; raw material: structure, breaking up level, type and amount of extracted substance; solvent's physical properties; as well as the process itself: duration, temperature, pressure, etc.

Ultrasounds intensity is detrimental, because it determines many secondary effects due to acoustic field presence such as thermal effects (temperature increase), cavitation, dispersion, etc.

The aim of the present paper was to study an influence of various acoustic field intensities and the material's breaking up level on the efficiency of extraction process. In addition, a comparison of the obtained results to those from the classical extraction method was made.

MATERIAL AND METHODS

Material

Dried carrots of Jarga cv. were the material for this study. Its initial moisture content was 12%.

Comminution

Dried carrots were ground in colloid mill WŻ-1. Then, the material was divided into fractions using laboratory sieving device. Three fractions of the dried material were taken to tests: 0.25-0.5 mm, 0.5-1 mm of particle diameter as well as the not broken up fraction, in which the particle size was within 2–10 mm range.

Ultrasonic processing

Before ultrasonic treatment, the material was wetted with water in 1:10 ratio (dried material : water). Sonication process was carried out in a glass vessel of 250 cm^3 capacity in ultrasonic device VCX 750 (Sonics) that made possible to continuously control the vibration amplitude and module for recording the current energy amount emitted by ultrasonic tip. One-inch diameter ultrasonic probe was used in the experiments. The treatment times were 10, 20, and 30 minutes at the following ultrasound intensities: 6.8, 11.6 and 16.2 W/cm^2. Temperature changes of the processed material were recorded during sonication (Fig. 1).

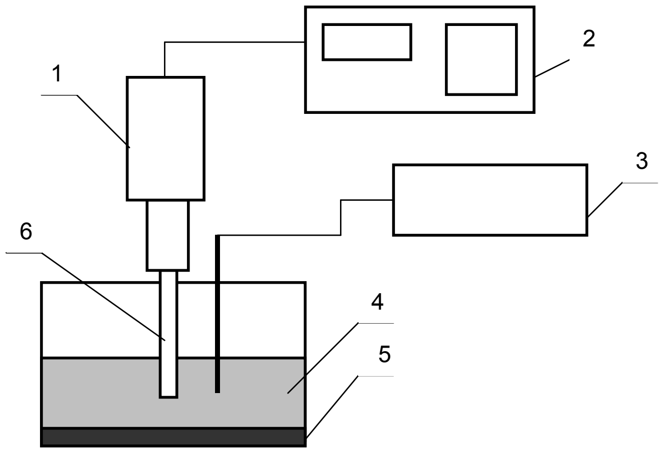


Fig. 1. Scheme of the set for ultrasonic processing: 1 – ultrasonic converter, 2 – generator with watt-meter, 3 – thermoelement, 4 – solvent, 5 – dried carrots, 6 – ultrasonic probe

Classical extraction (control)

Classical extraction was performed in thermostat by heating of diluted dried material in Erlenmeyer's flask till the temperature corresponding to that during ultrasonic processing. Solution was not stirred during heating.

Filtration

Separation of the achieved phases was made by means of gravitation method using metal perforated plate with 0.25 mm of mesh diameter, funnel and Erlenmeyer's flask.

Temperature measurments

The temperature was measured both during ultrasonic and classical extraction using APAR-type thermoelement (accuracy 1°C). Readings were made every 1 minute.

Calculation of extraction efficiency

Extraction efficiency was calculated using the following formula:

$$E = \frac{m_e}{m_s} 100\%$$

where:

m_e – weight of extracted substance (dry matter) from dried carrots;
m_s – initial weight of dried carrots.

Dry matter determination

Dry matter content in the extract was determined by drying at 105°C till constant weight in accordance to PN-90/A 75101/03.

Statistical processing

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

RESULTS AND DISCUSSION

Temperature changes

The ultrasounds action is inseparably associated with heat emission within the tested medium. The phenomena responsible for an increase of temperature in the processed material are: friction of boundary and interface surfaces, absorption of ultrasound energy and, in the case of high intensities, cavitation [Śliwiński 2001]. Almost 75% of acoustic energy transmitted by ultrasonic converter may be subsequently transformed into thermal energy of a system processed [Löning and Horst 2002] which is the reason of an decrease of intensifying effects of acoustic field in many processes [Mason and Lorimer 1989]. Therefore, monitoring of thermal changes within the tested system along with searching for optimum temperatures for extraction process performing are very important at ultrasonic processing.

Figures 2–4 present the temperature changes in the processed dried carrot – water system depending on initial intensity of acoustic field and the material's breaking up level.

The initial temperature of sonicated material was 16°C. At 6.8 W/cm^2 of initial intensity of ultrasound field, the studied solution began warming up achieving 58°C after 30 minutes of treatment. In the case of 11.6 W/cm^2 intensity at an analogous processing time, the temperature of the solution was 75°C. The highest temperature was recorded for the highest ultrasound field intensity level (16.2 W/cm^2), which was 82°C. Such temperature increase was observed for not broken up dried carrots (2–10 mm). Breaking up of the material did not affect the kinetics of the tested system's warming up.

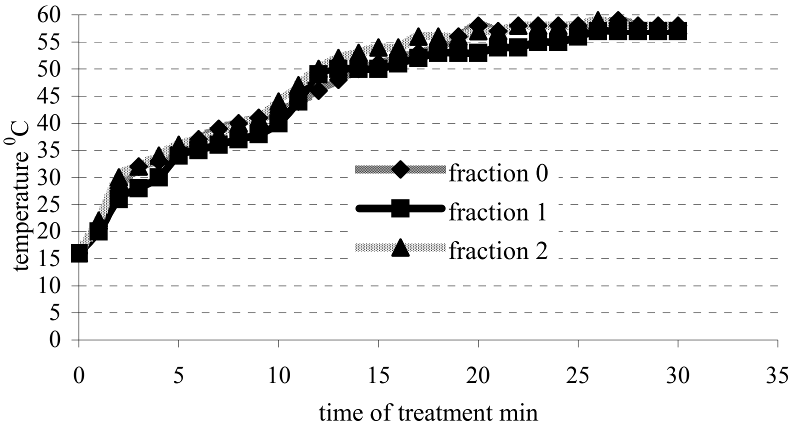


Fig. 2. Temperature changes in the tested system during processing at 6.8 W/cm^2 of ultrawave field intensity

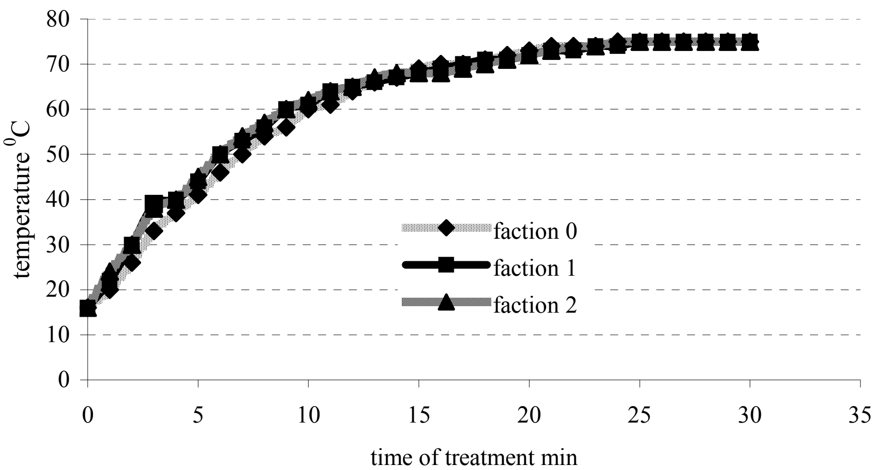


Fig. 3. Temperature changes in the tested system during processing at 11.6 W/cm^2 of ultrawave field intensity

Regression equations with similar coefficients (Table 1) were defined for all the studied fractions. No significant differences between warming up of materials containing dried particles of various diameters were observed. These equations were the basis for an evaluation of the temperature conditions in which the classical extraction should be performed (for comparisons).

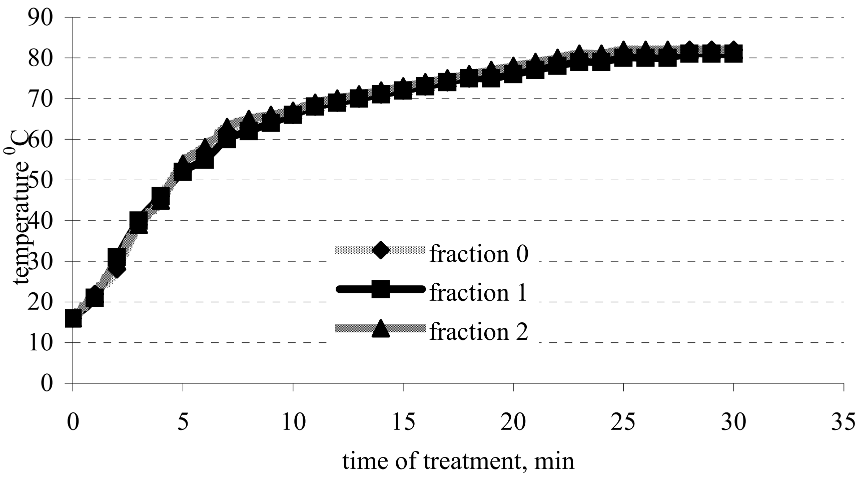


Fig. 4. Temperature changes in the tested system during processing at 16.2 W/cm^2 of ultrawave field intensity.

Table 1. Regression equations describing the temperature changes during water-dried material system sonication.

Intensity	Particle diameter	Regression equation	R^2 coefficient
6.8 W/cm^2	2–10 mm	Y = 12.502 ln t + 14.889	0.96
	0.5–1 mm	Y = 12.585 ln t + 14.947	0.96
	0.25–0.5 mm	Y = 12.268 ln t + 18.436	0.95
11.6 W/cm^2	2–10 mm	Y = 17.543 ln t + 20.322	0.97
	0.5–1 mm	Y = 17.081 ln t + 20.226	0.98
	0.25–0.5 mm	Y = 16.837 ln t + 21.033	0.98
16.2 W/cm^2	2–10 mm	Y =18.503 ln t + 21.537	0.98
	0.5–1 mm	Y = 18.142 ln t + 22.021	0.99
	0.25–0.5 mm	Y = 18.152 ln t + 22.227	0.99

Changes of dry matter content

Only total content of dry matter in the extract with no analysis of the type and share of particular components was determined in the experiment. Such an attempt is often used when achieving the bio-active substances from dried materials.

Results referring to the influence of acoustic field on dry matter contents are presented in Figures 5–7. The following symbols were applied c_0 – classical extraction, particle diameter 2–10 mm; c_1 – classical extraction, particle diameter 0.5–1 mm, c_2 – classical extraction, particle diameter 0.25–0.5 mm, u_0 – ultrasonic extraction, particle diameter 2–10 mm, u_1 – ultrasonic extraction, particle diameter 0.5–1 mm; u_2 – ultrasonic extraction, particle diameter 0.25–0.5 mm.

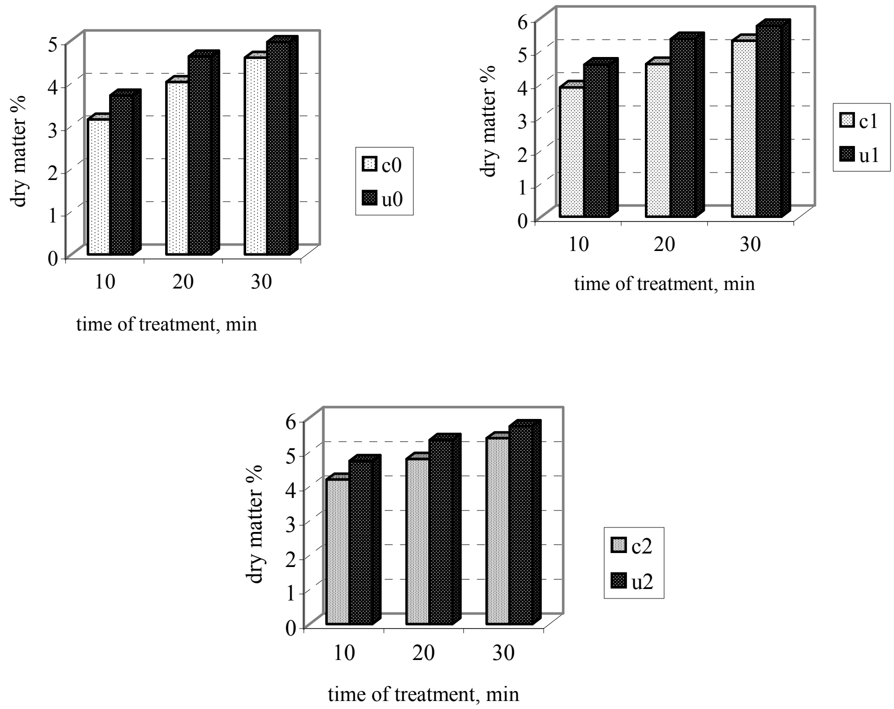


Fig. 5. Comparison of dry matter content changes in extracts after classical and ultrasonic (6.8 W/cm^2 of ultrasound field intensity) extractions

Extraction of dried carrots made using water and at presence of ultrasound field caused a statistically significant increase of dry matter content in the extract as compared to samples not treated with ultrasounds.

The difference was 15% after 10 minutes of extraction and 13% and 7% for 20 and 30 minutes, respectively, for not broken up fraction. In the case of 0.5–1 mm diameter fraction, differences between ultrasonic and classical samples were: for 10 minutes – 15%, for 20 minutes – 14% and for 30 minutes – 8%. Similar tendencies of dry matter content changes in the extract were observed for the smallest diameter fraction.

Increasing the ultrasound field intensity to 11.6 W/cm^2 resulted in a total increase of dry matter content in the extract. Amount of dry matter in extract after ultrasonic treatment was still higher than in samples achieved from classical extraction.

A further increase of acoustic field intensity up to 16.2 W/cm^2 did not cause a significant increase of dry matter content. Disappearance of differences of dry matter contents between sonicated and control samples was also recorded.

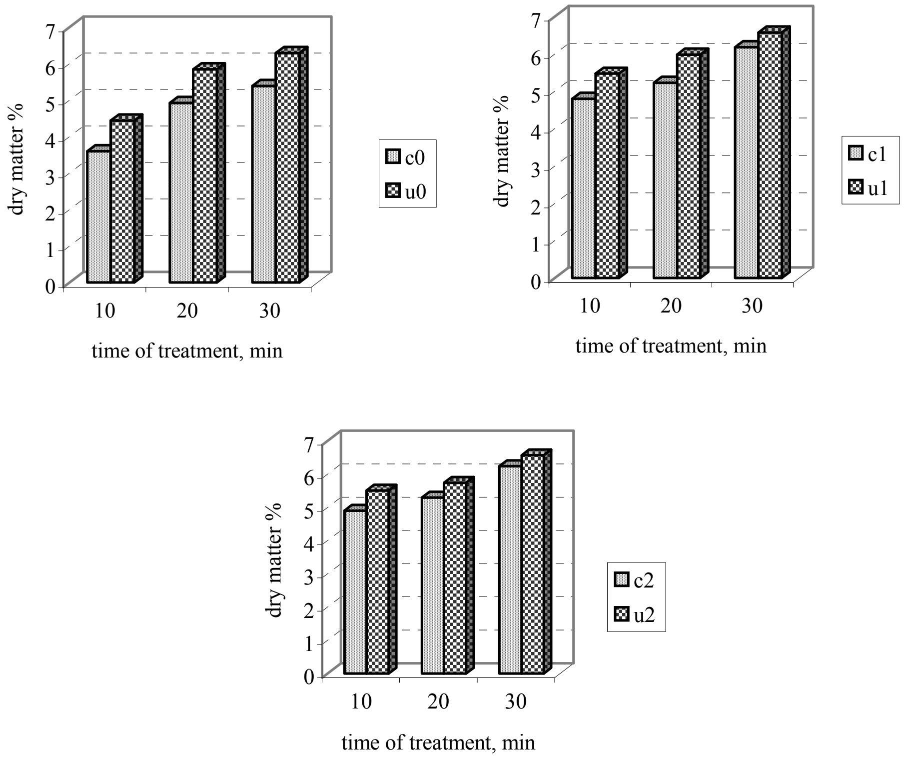


Fig. 6. Comparison of dry matter content changes in extracts after classical and ultrasonic (11.2 W/cm^2 of ultrasound field intensity) extractions

The highest differences of dry matter content between sonicated and classically extracted samples were achieved in the case of 6.2 and 11.6 W/cm^2 intensities. Increasing the acoustic field intensity resulted in an increase of total dry matter content in the extract, but at the same time the decrease of the difference between control and sonicated samples.

The elucidation of such dry matter content changes may be made on the basis of an analysis of the temperature influence on phenomena occurring at the presence of ultrasound field. At temperatures near ambient ones, the ultrasound action causes various secondary phenomena: micro-floats, disintegration, or cavitation. These effects are not the reason for mass exchange intensification. However, they make the diffusion layer thickness decrease, which in consequence contributes to the increase of diffusion rate. Elevation of the tested system's temperature (that occurs at no-cooling condition) reduces the influence of some secondary effects such as cavitation [Paniwnyk *et al.* 2001]. Temperature increase also causes higher absorption of ultrasound vibrations energy through the liquid and decreases the coefficient of the electric into mechanical energy transformation, which results in a decrease in the ultrasound system's working efficiency. Therefore, at higher temperatures, the effect of ultrasound interaction towards the tested system is mainly of thermal character. Thus, no dry matter content differences between classical and ultrasound extractions at higher temperatures were observed in the present study.

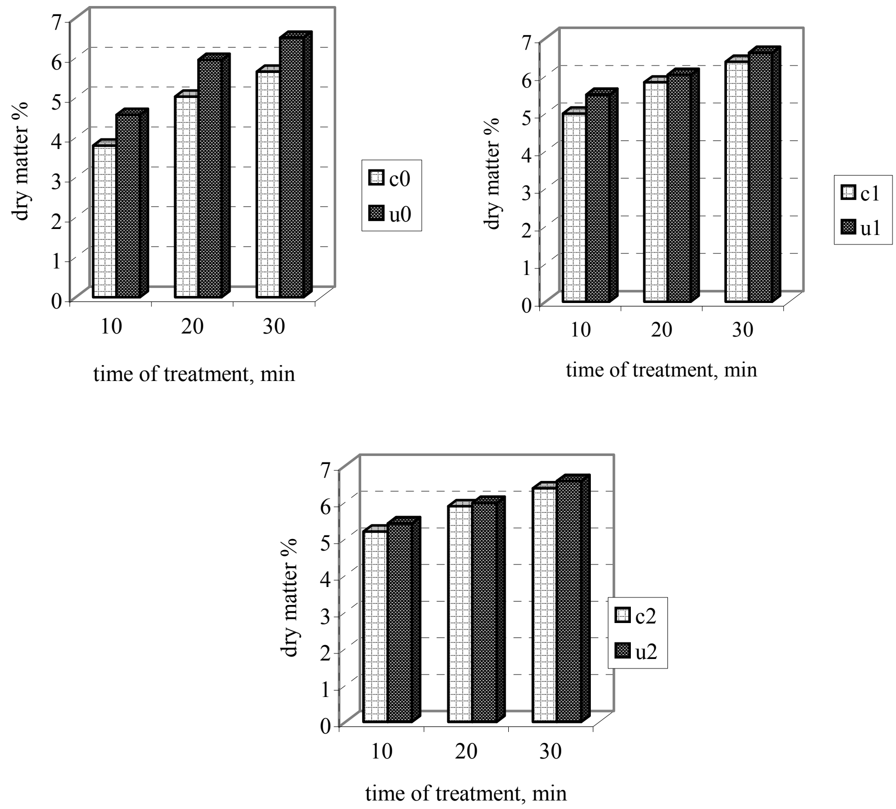


Fig. 7. Comparison of dry matter content changes in extracts after classical and ultrasonic (16.2 W/cm^2 of ultrasound field intensity) extractions

Changes of extraction yield

Yield is one of the general parameters describing the extraction efficiency. The achieved extraction yields are of a very complex character, thus they were analyzed separately for particular intensities of acoustic field.

Intensity of 6.8 W/cm^2

Result comparison from classical and ultrasonic extraction depending on dried material breaking up level is presented in Figures 8a–c.

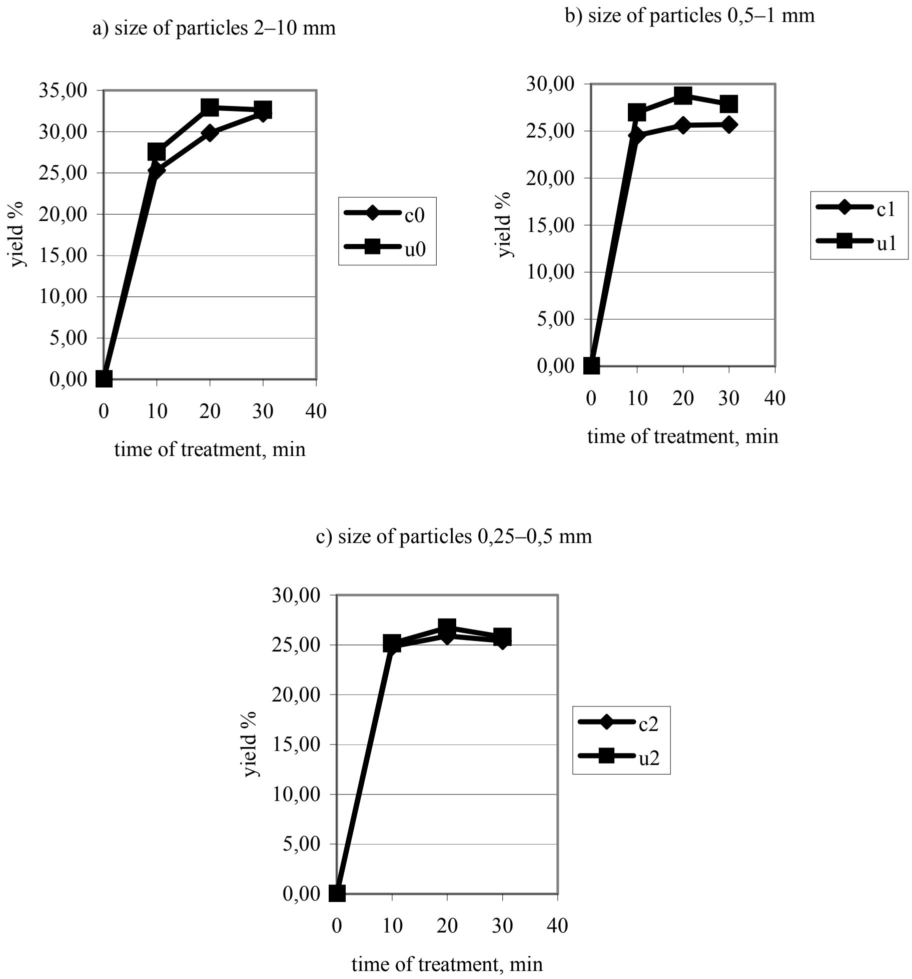


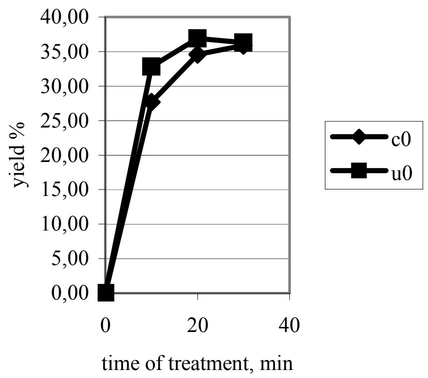
Fig. 8. Yields of classical (c) and ultrasonic (u) extractions applying 6.8 W/cm^2 of ultrasound field intensity

The extraction yield changed depending on the dried material's breaking up and processing time. For the largest particles (Fig. 8a), extraction yield increased along with the treatment time. The highest value of the parameter was achieved for that level of the material's breaking up. For fraction of 0.5–1 mm particle diameter (Fig. 8b), extraction yield increased till 20 minutes then remained at a constant level. For 0.25–0.5 mm fraction (Fig. 8c), the extraction yield reached the maximum value after 10 minutes of processing and then began to decrease. Referring to the whole studied range, higher or at least equal extraction yield was achieved at ultrasonic as compared to classical extraction.

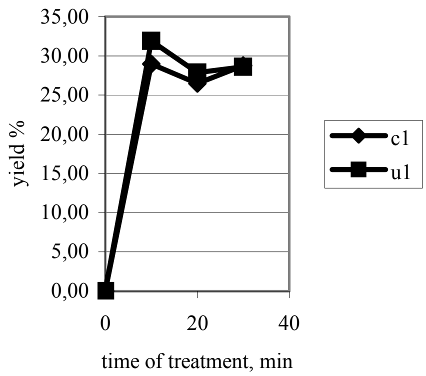
Intensity of 11.2 W/cm^2

Figures 9a-9c present the changes of extraction yield at 11.2 W/cm^2 of ultrasound field intensity. An increase of intensity caused an increase of extraction yield. The maximum extraction yield reaching 36.9% determined for not broken up fraction at 20-minute ultrasonic treatment was achieved, which was the highest value for the whole analyzed range. Higher or at least equal yield due to the application of acoustic field as compared to the classical method was achieved here, similar to the previous ultrasound field intensity.

a) size of particles 2–10 mm



b) size of particles 0,5–1 mm



c) size of particles 0,25 –0,5 mm

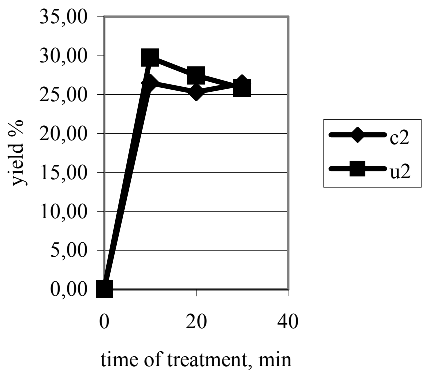
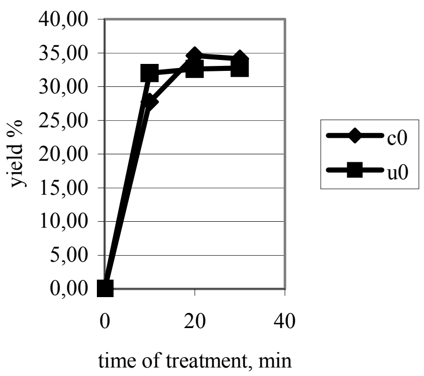


Fig. 9. Yields of classical (c) and ultrasonic (u) extractions applying 11.2 W/cm^2 of ultrasound field intensity

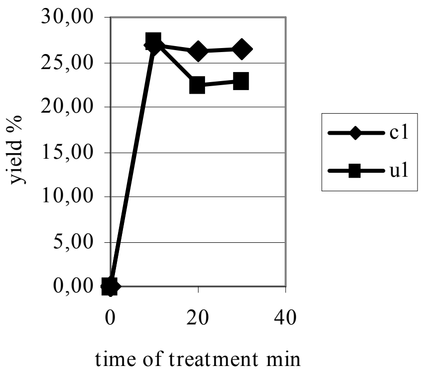
Intensity of 16.8 W/cm^2

Figures 10a–c present the changes of extraction yield at 16.8 W/cm^2 of ultrasound field intensity. A further increase of ultrasound intensity did not positively affect the extraction yield. Only in a single case better ultrasonic than classical extraction yield was achieved. Total decrease of numerical values for the extraction yield was observed both for sonicated and classically treated samples.

a) size of particles 2–10 mm



b) size of particles 0,5–1mm



c) size of particles 0,25–0,5 mm

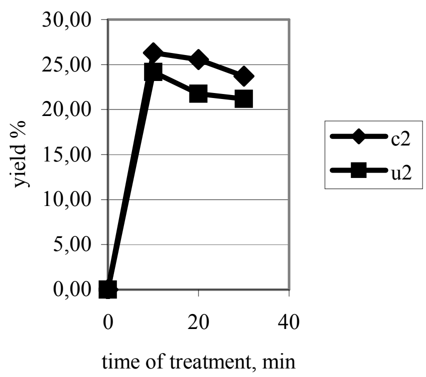


Fig. 10. Yields of classical (c) and ultrasonic (u) extractions applying 16.2 W/cm^2 of ultrasound field intensity

Complex character of yield changes both in the case of classical and ultrasound extraction may be elucidated by the dependence between the amount of filtered solution and dry matter content. It can be described by the following formula:

$$E = \frac{ym}{m_s} \ \%$$

where:

E – extraction yield %;
m – weight of filtered solution g;
y – content of dry matter in filtered solution %;
m_s – initial weight of dried material g.

Prolongation of the processing time caused an increase of the extracted substance concentration, and on the other hand, absorption of more and more solvent (water) by dried material. Increase of the concentration exceeded the losses due to solution absorption by dried material at the initial stage, which resulted in yield increase. Along with the treatment time, relative gains of extracted substance decreased, which affected the yield decrease at constant absorption of solution by the dried material. Breaking up of the dried material accelerated both the extraction of soluble substance and swelling of the dried material. A decrease of the yield occurred after some time, because the amount of the absorbed solution exceeded the relative gains of the extracted substance concentration. Ultrasounds not only make the soluble substances extraction faster, but also accelerate the dried material's swellin [Toma *et al.* 2001]. Therefore, the amount of absorbed solvent was higher in the case of classical extraction after longer sonication of the dried material. If the extracted substance concentration achieved at the same time is similar, in consequence it leads to lower yield of ultrasound-assisted process. Such effect was observed during ultrasonic extraction at 16.8 W/cm^2 intensity.

It should be kept in mind that extraction yield determined in such a way is not apparent and it may be misleading. To fully describe the course of dried carrots extraction, it would be necessary to study what part of soluble substance was re-absorbed by the extracted material due to the swelling process. It would be done by means of pressure separation after mixture extraction, e.g. by extrusion of the extract from the raffinate.

CONCLUSIONS

Ultrasonic extraction of dried carrots is a complex process affected by many factors such as: ultrasound field intensity, correlated temperature increase, processing time and raw material's breaking up level. In the case of the dried material's extraction, the yield is a sum of two processes: dissolution and diffusion of soluble substance into the solution as well as solvent penetration along with part of soluble substance into the solid interior. The excess of one of the above processes, at short treatment times, determines the final value of the achieved extraction yield. Introduction of acoustic field intensifies both the processes, which in consequence contributes to the change of substance extraction efficiency and makes the changes occurring within the process more complicated.

At high swelling coefficient, the yield changes are biased due to the solvent absorption by dried material. Therefore, also dry matter content achieved in the extract should be taken into account when considering the process efficiency.

The highest differences of dry matter contents in the extract were achieved between sonicated and control samples for short processing times. At this time, the temperature increase was low, which did not significantly confuse other effects invoked by ultrasounds.

On the basis of the performed experiments it was found that extraction of dried carrots gave the best results using acoustic field intensities in the range of 6.8–11.2 W/cm^2 at treatment times below 20 minutes. Application of such working parameters of ultrasound converter allows to achieve better effects than using the classical extraction method.

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