

## ULTRASOUND-ASSISTED EXTRACTION OF SUNFLOWER OIL FROM THE CAKE AFTER SUNFLOWER SEED PRESSING

**Dragan D. Milenković<sup>1\*</sup>, Milutin M. Milosavljević<sup>2</sup> and Aleksandar Lj. Bojić<sup>3</sup>**

<sup>1</sup>High Technological Technical School for Professional Studies,  
Kosančićeva 36, 37000 Kruševac, Serbia

<sup>2</sup>Faculty of Technical Science, University of Priština,  
Kneza Miloša 7, 38220 Kosovska Mitrovica, Serbia

<sup>3</sup>Faculty of Science and Mathematics, University of Niš,  
Višegradska 33, 18000 Niš, Serbia

**Abstract:** The influence of ultrasound application on the extraction (UE) of the cake after sunflower seed pressing was studied. Three different solvents were used for extraction in the Soxhlet apparatus: n-hexane, petroleum ether and extraction petrol. Petrol for extraction showed the highest yield. It has been shown that the using of ultrasound improved the extraction kinetics in the initial period – washing, and in the second period – the diffusion of oil from the mass of the cake. The parameters of the non-stationary diffusion model with and without the use of ultrasound were  $k' = 0.039 \text{ min}^{-1}$ ,  $k' = 0.026 \text{ min}^{-1}$  and  $b' = 0.713$ , i.e.  $b' = 0.589$ , respectively. Oil yield was also slightly higher in the ultrasonically supported extraction.

**Key words:** ultrasound, sunflower, extraction.

### Introduction

The production of sunflower oil is one of the most characteristic examples of the application of extraction on an industrial level. Therefore, this process is well developed and is the subject of research in many studies (Bäumler et al., 2016; Rai et al., 2016). The production of sunflower oil involves several stages: separating the grain from impurities, moistening the grain in the water vapor for easier separation of the shell, pressing, extracting the residual oil from the cake, and final treatment of the oil (dearomatization and decolorization). The cake, after extraction, contains a certain amount of residual oil and as such is used for the animal food production. Preferably, the oil content of the cake after extraction at an acceptable time interval is as low as possible.

---

\*Corresponding author: e-mail: dragan956@gmail.com

The using of ultrasound, with known effects of heterogeneous systems (cavitation), can have a positive effect on the extraction kinetics and yield (Amirante et al., 2017; Zhang et al., 2017). Many results of the studies in the field of extraction confirm the following: extraction of oil from the fennel seeds (*Foeniculum vulgare L.*) (Moubarik et al., 2011), mandarin orange (*Citrus reticulata*) (Ma et al., 2008), rose hip (*Rosa canina L.*) (Szentmihaályi et al., 2002) as well as sunflower seeds (*Helianthus annuus L.*), (Zardo et al., 2017), tobacco (*Nicotiana tabacum L.*) (Stanisavljević et al., 2009) and sage (*Salvia officinalis L.*) (Veličković et al., 2006; Milenović et al., 2002).

The results presented in this study are preliminary research results obtained within the framework of the project for improving the efficiency of the technological process of extracting sunflower oil in the oil company “Plima” in Kruševac. The goal of the project was to find out the rationality of the adaptation of the existing equipment.

## Materials and Methods

### Materials

As an extraction material in this study, a cake of the mechanically pressed sunflower seeds was used. The crushing of the cake was done in an electric grinder with rotary blades (15 000 rpm.). The resulting particles were then separated through standard sieves. For the extraction, a fraction from 0.4 mm to 0.5 mm was used. The content of moisture in the samples is given in Table 1. The n-hexane, petrol ether and high-purity petrol were used for the extraction.

Table 1. The moisture content in the samples, % m/m.

Sunflower seed	Cake before extraction	Cake after extraction
7.31	4.82	13.30

### Extraction in a Soxhlet apparatus (SA)

In this study, two extraction techniques were applied. The ultrasound-assisted extraction in a modified Soxhlet apparatus (SA) (Luque-Garcia and Luque de Castro, 2004), as shown in Figure 1, then a classical batch reactor for extraction with stirring, and an ultrasonic generator (BE) were used in this research.

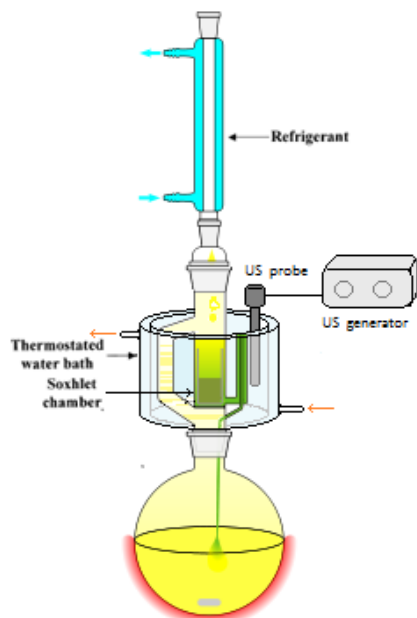


Figure 1. A modified Soxhlet apparatus for ultrasound-assisted extraction (Luque-Garcia and Luque de Castro, 2004).

The (SA) technique with and without the use of ultrasound was used to determine the maximum yield. The technique (BE) with and without ultrasound was used to monitor the extraction kinetics. For the (SA) technique, a sample of 10 g and 100 mL of the appropriate solvent was used. The extraction time was 6 hours (24 cycles). After the final extraction, the solvent was eliminated in a vacuum evaporator at 50°C.

#### Extraction in the batch reactor

The extraction kinetics in this study was investigated in a classical batch reactor with stirring and the ultrasonically assisted extraction in the batch reactor. In both cases, high-purity petrol was used as a solvent. A 1:10 hydromodule was applied, and a sample of 5 g of the sunflower cake was placed in an Erlenmeyer of 100 mL with 50 mL of the solvent. The set-up consisted of an ultrasonic cleaner (EI, Niš, Serbia; total nominal power: 2 x 50 W; and internal dimensions: 300 x 220 x 155 mm), operating at 40 kHz frequency. The Erlenmeyer was then immersed in an ultrasonic bath filled with water up to 1/3 height. The operating temperature was constant at 25°C ( $\pm 0.2^\circ\text{C}$ ). The extraction times were 2.5 min, 5 min, 10 min, 20 min, 40 min and 60 min. After completion of the extraction, the solvent was removed in a vacuum evaporator at 50°C.

## Results and Discussion

### Extraction in the Soxhlet apparatus (SA)

The results of the sunflower oil extraction from the pressed sunflower seeds are shown in Table 2 and Figure 2. There was a positive contribution to the use of ultrasound. Compared to n-hexane and petrol ether, petrol showed the best yield, both without and with ultrasound. The contribution of ultrasound to all samples ranged from 1% to 1.5%.

Table 2. The sunflower oil residue in samples after pressing in operating conditions, determined without and with ultrasonically assisted extraction, % m/m.

	n-hexane		Petrol ether		Petrol for extraction	
	Without ultrasound	With ultrasound	Without ultrasound	With ultrasound	Without ultrasound	With ultrasound
1	19.02	19.97	19.50	20.67	19.73	21.11
2	21.95	23.05	22.31	23.65	22.44	24.01
3	20.01	21.01	20.08	21.29	20.19	21.60

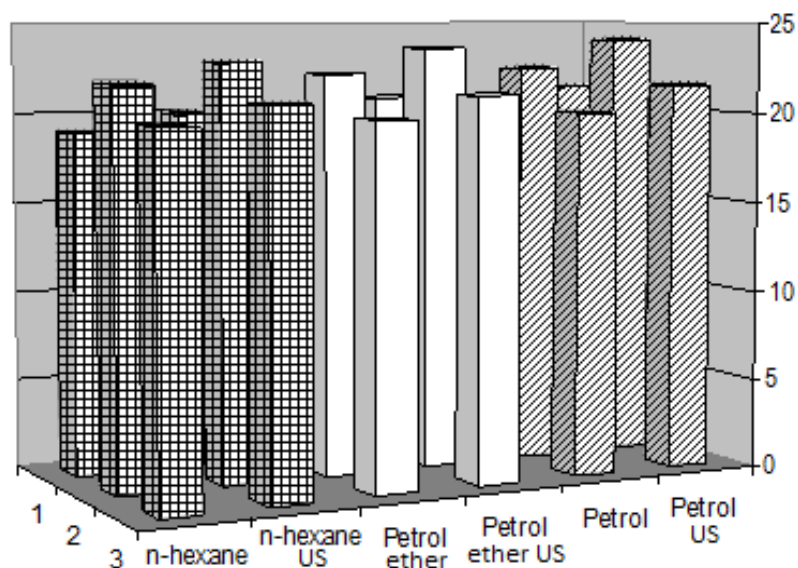


Figure 2. A graphic presentation of ultrasound application in sunflower oil extraction of the mechanically pressed sunflower seeds in various solvents.

Extraction in the batch reactor (BE)

The results of the sunflower oil extraction kinetics, with and without the use of ultrasound, are shown graphically in Figure 3. In both cases, the shape of the curve is as in the extraction of tobacco oil (Stanisavljević et al., 2009), the extraction of sage (Veličković et al., 2006) and St. John’s wort (*Hypericum perforatum*)(Milenović et al., 2002). The kinetic curve had two distinctly separate periods. The first part (<5 min), flushing, was characterized by a rapid change in the concentration of oil in the solvent, and the second part, the period of slow extraction (diffusion phase), in which the slow progress in the yield was observed. The largest part of the oil (> 95%) was extracted for up to 20 minutes and complete extraction was achieved in 60 minutes.

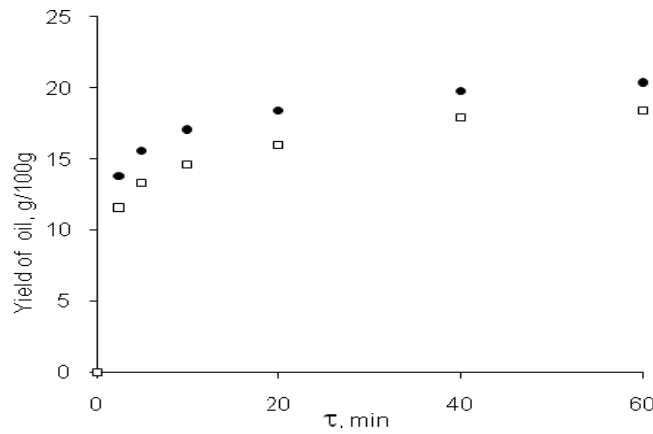


Figure 3. Kinetics of sunflower oil extraction from the cake after sunflower seed pressing with (\*) and without (□) the use of ultrasound.

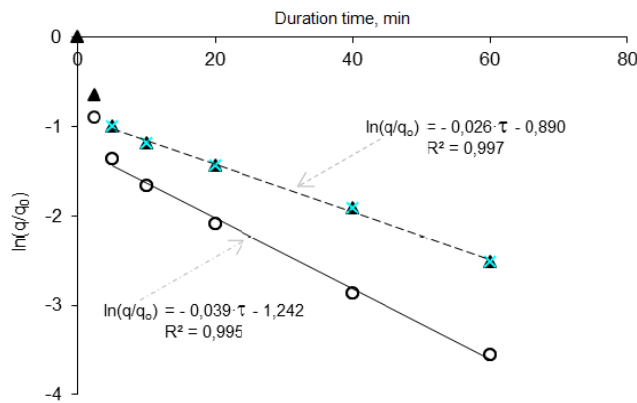


Figure 4. The linear shape of the kinetic curve of the extraction of sunflower oil with (o) and without (Δ) ultrasound.

Table 3. Parameters of the unsteady diffusion model without and with ultrasound.

Without ultrasound			With ultrasound		
$k', \text{min}^{-1}$	$b'$	R	$k', \text{min}^{-1}$	$b'$	R
0.026	0.589	0.997	0.039	0.713	0.995

A large number of models can be found in the literature describing solid-liquid extraction. However, the kinetics of the extraction process of substances from raw materials has most often been modeled using the unsteady diffusion through plant material (Stanković et al., 1994), the film theory (Pekić et al., 1988) and the empirical equation of Ponomaryov (1976). Some of them are shown in Table 4.

Table 4. Some mathematical models describing solid-liquid extraction.

Model	Equation	Linearized form	References
Ponomaryov	$(q_0 - q)/q_0 = b + kt$		Ponomaryov (1976)
Film theory	$(1 - c/c_0) = (1 - b') \cdot e^{-k't}$	$\ln(1 - c/c_0) = \ln(1 - b') - k't$	Pekić et al. (1988)
Non-stationary diffusion model	$q/q_0 = (1 - b') \cdot e^{-k't}$	$\ln(q/q_0) = \ln(1 - b') - k't$	Stanković et al. (1994)
Parabolic diffusion model	$\bar{q} = A_0 + A_1 \tau^{1/2} + A_2 \tau$		Kitanović et al. (2008)
Power law model	$\bar{q} = B \tau^n$	$\ln \bar{q} = \ln B + n \ln \tau$	Kitanović et al. (2008)
Hyperbolic model	$\bar{q} = \frac{C_1 \tau}{1 + C_2 \tau}$	$\frac{1}{\bar{q}} = \frac{1}{C_1} \cdot \frac{1}{\tau} + \frac{C_2}{C_1}$	Kitanović et al. (2008)
Weibull's equation	$\bar{q} = 1 - \exp\left[-\left(\frac{\tau}{\delta}\right)^m\right]$	$\ln[-\ln(1 - \bar{q})] = -\ln D + m \ln \tau$	Kitanović et al. (2008)
Elovich's equation	$\bar{q} = E_0 + E_1 \ln \tau$		Kitanović et al. (2008)

In this paper, a model based on the unsteady diffusion (non-stationary diffusion model) is used, which has the following form:

$$q/q_0 = (1 - b') \cdot \exp(-k't) \quad (1)$$

or a linearized form:

$$\ln(q/q_0) = \ln(1 - b') - k't \quad (2)$$

where:  $b'$  is the washing coefficient, 1;  $k'$  is the slow diffusion coefficient,  $\text{min}^{-1}$ ,  $q$  is the oil content in the seeds during the extraction, g/100 g;  $q_0$  is the oil

content initially present in the seeds,  $g/100\text{ g}$ , and  $\tau$  is time in minutes. The  $b'$  and  $k'$  were determined experimentally from the slope and segment by plotting  $\ln(q/q_0)$  against  $\tau$ . The linear regression analysis, in this work, was done using Microsoft Office Excel 2007.

The model is based on the two-stage extraction mechanism and two parameters included. The first parameter ( $b'$ ) characterizes the washing stage (the so-called washing coefficient) and the second parameter ( $k'$ ) characterizes the slow extraction (the so-called slow extraction coefficient). A graphic presentation of a change of the  $\ln(1-q/q_0)$  during the time is shown in Figure 4. Significant deviations from the model equation could only be observed in the initial period. The values of both kinetic parameters are presented in Table 3.

On the basis of linear forms of the kinetic curves (shown in Figure 4) and the linear correlation coefficient, it can be concluded that the kinetic models applied fitted well the experimental data. The coefficients of linear correlation were higher than 0.995 based on values in Table 3. In the second stage of extraction, a high level of coefficients of linear correlation ( $R$ ) illustrates a very good agreement between the theory model and the experiment.

Based on the values of the rapid ( $b'$ ) and slow ( $k'$ ) parameters of the extractions shown in Table 3, the positive effect of ultrasound is seen. The coefficient of slow extraction without ultrasonic application was  $k' = 0.026\text{ min}^{-1}$ , whereas with the use of ultrasound  $k' = 0.039\text{ min}^{-1}$ . The coefficient of slow extraction was found to be 50% higher with the use of ultrasound. The effect of ultrasound is probably due to cavitation. The cavitation phenomenon is likely to partially destroy the structure of the solid phase (Toma et al., 2001). In addition, the cavitation phenomenon is likely to partially increase the temperature of oil in the solid phase. This affects the viscosity of the oil as well. In this way, the diffusion of the oil through the solid phase is accelerated.

The coefficient of washing without the application of ultrasound was  $b' = 0.589$ , whereas with the use of ultrasound  $b' = 0.713$ . The use of ultrasound increased the rate of extraction in the initial period (washing) by 21%. Obviously, a greater contribution of the ultrasound to the extraction of the oil was recorded in a slower phase, in the diffusion, through the solid material.

### Conclusion

The obtained results show that the ultrasound had a positive effect on extraction in the Soxhlet apparatus. For the same number of cycles in the Soxhlet apparatus, the yield with ultrasound was about 1.5% higher. In relation to n-hexane and petroleum, the extraction with petrol showed the best results (yield) in extraction, both without and with ultrasound.

A two-parameter mathematic model was used for the extraction kinetics modeling; a model based on unsteady diffusion through solid material. In the second stage, the applied mathematical model best followed experimental results. The linear correlation coefficient was  $R = 0.995$  without ultrasound and  $R = 0.997$  by using of ultrasound. A positive effect of ultrasound, both on the extraction rate and on the oil yield, was confirmed by the applied model ( $b'$ ,  $k'$ ). As is known, in addition to ultrasound, many other parameters (grain size, temperature, hydrodynamic conditions, etc.) also affect the extraction efficiency. Therefore, the obtained results provide a good base for further research.

### Acknowledgments

This work was financed by the Serbian Ministry of Education, Science, and Technological Development, project TR 34008.

### References

- Amirante, R., Distaso, E., Tamburrano, P., Paduano, A., & Clodoveo, L.M. (2017). Acoustic cavitation by means ultrasounds in the extra virgin olive oil extraction process. *Energy Procedia*, 126, 82-90.
- Erica, R.B., María, E.C., & Amalia, A.C. (2016). Extraction of sunflower oil using ethanol as solvent. *Journal of Food Engineering*, 178, 190-197.
- Ivanor, Z., Andressa, E.S., Ligia, D.F.M., & Julia, S. (2017). Optimization of ultrasound assisted extraction of phenolic compounds from sunflower seed cake using response surface methodology. *Waste Biomass Valor*, 8, 1-12.
- Kitanović, S., Milenović, D., & Veljković, B.V. (2008). Empirical kinetic models for the resinoid extraction from aerial parts of St. John's wort (*Hypericum perforatum* L.). *Biochemical Engineering Journal*, 41, 1-11.
- Lei, Z., Cunshan, Z., Bei, W., Abu, E.G.A.Y., Haile, M., Xiao, Z., & Mian, W. (2017). Study of ultrasonic cavitation during extraction of the peanut oil at varying frequencies. *Ultrasonics Sonochemistry*, 37, 106-113.
- Luque-Garcia, L.J., & Luque, C.D.M. (2004). Ultrasound-assisted Soxhlet extraction: an expeditive approach for solid sample treatment. Application to the extraction of total fat from oleaginous seeds. *Journal of Chromatography A*, 1034 (1-2), 237-42.
- Milenović, D., Veljković, B.V., Todorović, B., & Stanković, R.M. (2002). Analiza ekstrakcije rezinoida kantariona (*Hypericum perforatum* L.) I. Efikasnost i optimizacija ekstrakcije. *Hemijaska Industrija*, 56, 54-59.
- Moubarik, A., El-Belghiti, K., & Vorobiev, E. (2011). Kinetic model of solute aqueous extraction from Fennel (*Foeniculum vulgare*) treated by pulsed electric field, electrical discharges and ultrasonic irradiations. *Food and Bioproducts Processing*, 89 (4), 356-361.
- Pekic, B., Stojanovic, D., Lepojevic, Z., & Tolic, A. (1988). Investigation of extraction kinetics of glycoside from leaves of *Digitalis lanata* Ehrh. *Pharmazeutische Industrie*, 50 (8), 984-986.
- Ponomaryov, V.D. (1976). *Medicinal Herbs Extraction*. Medicina, Moscow.
- Rai, A., Mohanty, B., & Bhargava, R. (2016). Supercritical extraction of sunflower oil: A central composite design for extraction variables. *Food Chemistry*, 192, 647-659.
- Stanisavljević, T. I., Lazić L.M., & Veljković, B.V. (2007). Ultrasonic extraction of oil from tobacco (*Nicotiana tabacum* L.) seeds. *Ultrasonics Sonochemistry*, 14, 646-652.



- Stankovic, M.Z., Cakic, M.D., Cvetkovic, D.M., & Veljkovic, V.B. (1994). Kinetics of extraction of resinoids from over ground parts of sweet clover (*Melilotus officinalis* L.). *Journal Serbian Chemical Society*, 10 (39), 735-741.
- Szentmihaályi, K., Vinkler, P., Lakatos, B., Illeés, V., & Then, M. (2002). Rose hip (*Rosa canina* L.) oil obtained from waste hip seeds by different extraction methods. *Bioresour Technology*, 82, 195-201.
- Toma, M., Vinatoru, M., Paniwnyk, L., & Mason, T.J. (2001). Investigation of the effects of ultrasound on vegetal tissues during solvent extraction. *Ultrasonics Sonochemistry*, 8, 137-142.
- Veličković, T.D., Milenović, M.D., Ristić, S.M., & Veljković, B.V. (2006). Kinetics of ultrasonic extraction of extractive substances from garden (*Salvia officinalis* L.) and glutinous (*Salvia glutinosa* L.) sage. *Ultrasonics Sonochemistry*, 13, 150-156.
- Yaqin, M., Xingqian, Y., Yunbin, H., Guoneng, X., Guihua, X., & Donghong, L. (2008). Ultrasound-assisted extraction of hesperidin from Penggan (*Citrus reticulata*) peel. *Ultrasonics Sonochemistry*, 15 (3), 227-32.

Received: February 28, 2018

Accepted: March 29, 2018

## ULTRAZVUČNO POTPOMOGNUTA EKSTRAKCIJA POGAČE NAKON PRESOVANJA LJUŠTENIH ZRNA SUNCOKRETA

**Dragan D. Milenković<sup>1\*</sup>, Milutin M. Milosavljević<sup>2</sup> i Aleksandar Lj. Bojić<sup>3</sup>**

<sup>1</sup>Visoka tehnička tehnološka škola strukovnih studija,  
Kosančićeva 36, 37000 Kruševac, Srbija

<sup>2</sup>Tehnički fakultet, Univerzitet u Prištini,  
Kneza Miloša 7, 38220 Kosovska Mitrovica, Srbija

<sup>3</sup>Prirodno-matematički fakultet, Univerzitet u Nišu,  
Višegradska 33, 18000 Niš, Srbija

### R e z i m e

U radu je istražen uticaj primene ultrazvuka na ekstrakciju pogače nakon presovanja oljuštenih zrna suncokreta. Za ekstrakciju u Soxlet aparatu su primenjena tri rastvarača: n-heksan, petrol etar i ekstrakcioni benzin. Kao najefikasniji rastvarač pokazao se ekstrakcioni benzin. Doprinos primene ultrazvuka na ekstrakciju u Soxlet aparatu, kod sva tri rastvarača je 1% do 1,5% pri istom broju ciklusa, 24.

Kinetika procesa, bez i sa primenom ultrazvuka, praćena je u šaržnom reaktoru uz primenu ekstrakcionog benzina. Koeficijent linearne korelacije ( $R=0,995$ ) potvrđuje dobro slaganje eksperimentalnih rezultata i primenjenog modela nestacionarne difuzije kroz čvrst materijal. Primena ultrazvuka pri ekstrakciji u šaržnom reaktoru pokazala je pozitivan efekat celim tokom procesa, kako u početnom periodu (ispiranje) tako i u drugom delu ☐ spora ekstrakcija. Pozitivan efekat primene ultrazvuka, kako na brzinu tako i na prinos, potvrđuje se vrednostima parametara primenjenog modela nestacionarne difuzije ( $b'$ ,  $k'$ ). Parametri primenjenog modela sa i bez ultrazvuka su  $k' = 0,039 \text{ min}^{-1}$  i  $k' = 0,026 \text{ min}^{-1}$  odnosno  $b' = 0,713$  i  $b' = 0,589$ . Na osnovu ovih parametara, doprinos primene ultrazvuka u istraživanom slučaju veći je u fazi spore ekstrakcije – difuzije za 50% u odnosu na fazu ispiranja, gde je doprinos 21%.

Dobijeni rezultati ukazuju na opravdanost daljih istraživanja uticaja i drugih relevantnih parametara (temperatura, hidromodul, veličina čestica itd.) na efikasnost primene ultrazvuka pri ekstrakciji ulja iz pogače nakon mehaničkog presovanja zrna suncokreta.

**Ključne reči:** ultrazvuk, suncokret, ekstrakcija.

Primljeno: 28. februara 2018.  
Odobreno: 29. marta 2018.

---

\* Autor za kontakt: e-mail: dragan956@gmail.com