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INFLUENCE OF THE ULTRASONIC TREATMENT ON THE PROCESS OF OBTAINING ESSENTIAL OIL FROM CARAWAY SEEDS

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ABSTRACT

The paper presents research results concerning the influence of ultrasonic pre-treatment on the process of distillation of essential oils from caraway seeds. Tests were carried out in two variants. In the first one, the whole seeds and in the second one ground seeds were subjected to a distillation process. An ultrasonic processor (Sonic VC 750) with a head of 19 mm diameter operating with frequency 20 kHz was used for generating ultrasounds. The following parameters of initial sonication of seeds were applied: treatment time from 20 to 60 minutes, intensity of ultrasounds: 28 and 42 W·cm^{-2}. It was found out that the initial ultrasonic treatment of caraway considerably speeds up the rate of obtaining essential oils and increases the final efficiency of the process. Depending on the time and intensity of ultrasounds the increase of efficiency from 9 to 40% was reported. Moreover, a significant impact of seeds fragmentation on the kinetics of the distillation process was reported.

Introduction

Caraway (*Carum carvi L.*) is one of the most important herbs. It is grown on the area of about 8.000 hectares in Poland (Seidler-Łożykowska et al., 2010). Caraway seeds constitute medical raw material and also commonly known dietetic spice. They are used as a supplement for bread and other baker's goods, meat dishes, soups, vegetables, salads, some brands of cheese and alcoholic drinks, such as *vodka*, liqueurs, etc (Peter, 2006). The medical properties of caraway seeds are important due to a high content of essential oils (from 2% to 8%) in which carvone and limonene are the main active compounds (Bailer et al., 2001; Chemat et al., 2004). Essential oil from caraway seeds is given by mouth as a cholagogue, choleretic and eccoprotic. It is also used as a microbicide, fungicide and parasticide. Essential oils are applied as an additive for soaps, decontaminators, cosmetic creams, toothpastes and perfumes (Ożarowski and Jaroniewski, 1987). It is also used as a mean that prevents potatoes germination (Oosterhaven et al., 1996).

The most common method applied for obtaining essential oils is steam distillation. Due to low contain of essential oils in raw material new methods for increase of the yield of distillation are being searched for (Kowalski and Wawrzykowski, 2009). One of them can be applied of preliminary ultrasonic treatment. Ultrasound causes many physical phenomena that influence mass transfer in a solid-liquid system. The most important are cavitation and microstreamings. These phenomena provide a greater penetration of solvent into cellular materials, destroy plant tissues and improve the release of cell contents into the bulk medium.

The use of an ultrasonic treatment for the extraction of essential oil proved to diminish the risk of thermal degradation and aid extraction by significantly reducing extraction times (Kimbaris et al., 2006).

Objective of the study

The objective of the study was to examine the influence of preliminary ultrasonic treatment on the kinetic and final yield of distillation of essential oils from caraway seeds.

Research methodology

The research material was in the form of caraway seeds (*Carum carvi L.*) Kończewicki variety bought in the local chemist's shop. The research was conducted in two stages, one involving a distillation process of the whole seeds and the second of comminuted seeds. The raw material was ground in colloid mill WŻ-11 in the time of 12 seconds. Then, the material was divided into fractions using a laboratory screeing device with round meshes. One fraction with particle size of 150-250 μm was taken for testing. The ultrasonic treatment was carried out with the use of an ultrasonic processor (Sonic VC 750) with a horn of 19 mm diameter and working at a frequency of 20 kHz. The following parameters of ultrasonic treatment were applied: sonication time – 20, 40 and 60 minutes and intensity of ultrasound – 28 and 42 $W \cdot cm^{-2}$.

The preliminary ultrasonic treatment was conducted in a distilling flask of 1000 ml capacity. For this purpose, samples of 25 g were placed in the flask and filled with 600 ml of water. After ultrasonic treatment the flask was connected to Deryng apparatus and essential oil was distillated for 3 hours. The readings were taken for every 10 minutes during the distillation process.

The percent content of essential oil was calculated from the following formula:

$$X = \frac{a \cdot 100}{m} (\%) \tag{1}$$

where:

m – weight of sample, (g)

a – volume of essential oil, (cm^3)

Figure 1. Ultrasonic processor Sonic VC 750

For comparative purposes the distillation process of essentials oil according to the standard procedure (Farmakopea Polska, 2002) was conducted. The experiments were carried out in 3 replications. Achieved results were subjected to statistical processing applying variance analysis. Significance of differences between means was tested by Tukey's test, with a significant level of 5%. All computations were made using Statistica 6.0 software.

Results and discussion

The influence of preliminary ultrasonic treatment on the yield of essential oil obtained from the whole caraway seeds is shown in figure 2-3.

The preliminary ultrasonic treatment had significant effect on distillation curves of essential oil. The sonication of caraway seeds increased the velocity and final yield of distillation of essential oils for all the examined cases.

The obtained results showed that preliminary sonication at intensity of 28 $W \cdot cm^{-2}$ (fig. 2) allows achieving the same yield of process after 70 minutes like 180-minutes distillation of untreated seeds and improves final yield of distillation by 12%.

The distillation yield depended on time and intensity of preliminary ultrasonic treatment. The extension of sonication to 60 minutes resulted in increasing the processing yield by 40%. The increase in intensity of ultrasound resulted in the speed acceleration and final distillation yield. Sonication of seeds at intensity of 42 $W \cdot cm^{-2}$ allows achieving after 40 minutes the same results like 60 minutes treatment at intensity of 28 $W \cdot cm^{-2}$.

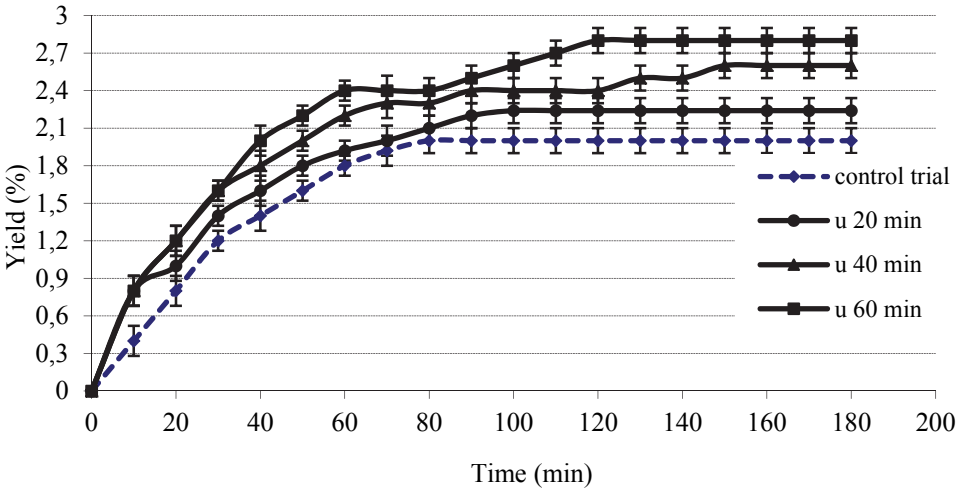

Figure 2. Influence of ultrasound pre-treatment of intensity 28 W·cm^{-2} on the process of obtaining essential oil from whole caraway seeds

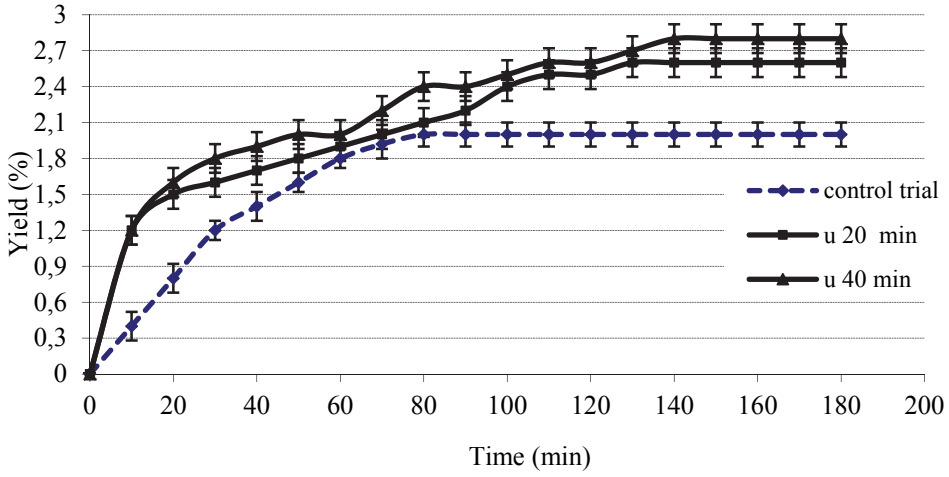

Figure 3. Influence of ultrasound pre-treatment of intensity 42 W·cm^{-2} on the process of obtaining essential oil from whole caraway seeds

The influence of the preliminary ultrasonic treatment on the yield of essential oils obtained from the ground caraway seeds is shown in fig. 4-5.

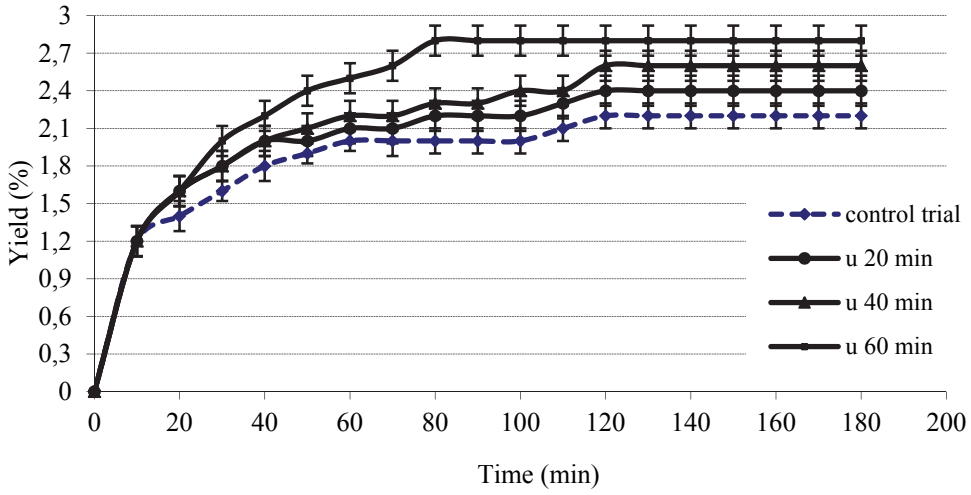

Figure 4. Influence of ultrasound pre-treatment of intensity 28 W·cm^{-2} on the process of obtaining essential oil from fragmented caraway seeds

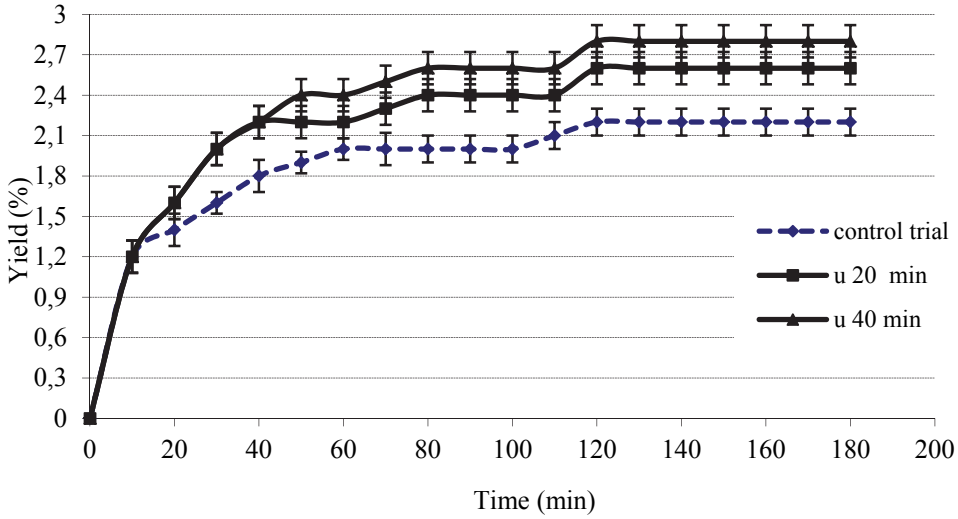

Figure 5. Influence of ultrasound pre-treatment of intensity 42 W·cm^{-2} on the process of obtaining essential oil from fragmented caraway seeds

The grinding of caraway seeds had an important effect on the shape of distillation curves. In the case of the classical distillation the grinding of seeds resulted in increasing the speed and the final efficiency of the process. The yield of essential oils from the ground seeds was 10% higher in comparison to the yield from the whole seeds. In the case of distillation which was preceded by the preliminary ultrasonic treatment only the increase of the distillation speed was observed. There was no influence of the seeds grinding on the final efficiency of the process.

The list of the final efficiencies of distillation of essential oils from caraway depending on the degree of fragmentation and the type of ultrasonic pre-treatment was shown in Table 1.

Table 1
The list of the final efficiencies of distillation of essential oil from caraway depending on the degree of fragmentation and the type of pre-treatment

Raw material/ Parameters of processing	Intensity of ultrasound ($W \cdot cm^{-2}$)	Time of treatment (min)	Yield (%)	Standard devitation (%)
Whole caraway seeds	0 (control trial)	0	2	0.05
	28	20	2.24	0.05
		40	2.6	0.05
		60	2.8	0.05
	42	20	2.6	0.06
		40	2.8	0.06
Ground caraway seeds	0 (control trial)	0	2.2	0.05
	28	20	2.4	0.06
		40	2.6	0.06
		60	2.8	0.06
	42	20	2.6	0.06
		40	2.8	0.06

The mechanism of intensification of essential oils distillation from caraway seeds in the presence of ultrasound is strictly connected to their physical effects. Sonication of seeds breaks cell walls and releases contents of cells into the extraction solvent. Ultrasonic treatment increases the speed of distillation process and also can improve the final efficiency of the whole process. The evidence for the mechanical impact of ultrasounds are structural changes in caraway seeds after ultrasonic treatment (Chemat et al., 2004). The kinetics of steam distillation is depended on intensity of ultrasounds. Assami et al. (2012) examining the influence of ultrasonic treatment at intensity of 1 $W \cdot cm^{-2}$ obtained significant acceleration of process distillation, but they didn't observe increase in the final efficiency of the process.

Application of higher intensity of ultrasounds results in the increase of the yield of process distillation (Kowalski and Wawrzykowski, 2009).

Conclusions

1. Preliminary ultrasonic treatment accelerates distillation of essential oils from caraway seeds and increases the final yield of process. The increase of the distillation yield depends on the time and intensity of ultrasound and ranges from 9% to 40%.
2. Grinding of caraway seeds increases the speed and final efficiency of essential oils only in case of the classical distillation. In case of distillation which was preceded by the preliminary ultrasonic treatment only the increase of the distillation speed was observed. There was no influence of the seeds grinding on the final efficiency of the process.

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WPŁYW OBRÓBKI ULTRADŹWIĘKOWEJ NA PROCES POZYSKIWANIA OLEJKÓW ETERYCZNYCH Z NASION KMINKU ZWYCZAJNEGO

Streszczenie. W pracy przedstawiono wyniki badań dotyczące wpływu wstępnej obróbki ultradźwiękowej na proces destylacji olejków eterycznych z nasion kminku zwyczajnego. Badania przeprowadzono w dwóch wariantach. W pierwszym procesowi destylacji poddano całe nasiona, zaś w drugim nasiona rozdrobnione. Do generowania ultradźwięków wykorzystano procesor ultradźwiękowy (Sonic VC 750) z głowicą o średnicy 19 mm, pracujący z częstotliwością 20 kHz. Zastosowano następujące parametry wstępnej sonifikacji nasion: czas obróbki od 20 do 60 minut, natężenie ultradźwięków: 28 i 42 $W \cdot cm^{-2}$. Stwierdzono, że wstępna obróbka ultradźwiękowa nasion kminku znacząco przyśpiesza tempo pozyskiwania olejków oraz zwiększa końcową wydajność procesu. W zależności od czasu i natężenia ultradźwięków zanotowano wzrost wydajności od 9 do 40%. Zaobserwowano także istotny wpływ rozdrobnienia nasion na kinetykę procesu destylacji.

Słowa kluczowe: kminek zwyczajny, olejki eteryczne, destylacja, ultradźwięki