Ultrasound degumming of sunflower oil

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Abstract

Introduction. The degumming of vegetable oils is a crucial stage of oil refining aimed at phospholipid removal. The objectives of this study were to investigate the possibility of ultrasound acceleration of the sunflower oil degumming.

Materials and methods. The ultrasound degumming of vegetable oil was carried out on the mixer MEDITON (Ukraine) and ultrasound thermostat Bandelin Sonorex (Austria) under addition of 5 % water from 5 to 15 minutes. The volume of degummed oil was measured after gum separation by centrifugation. Phosphorus content in oil ash was detected photometrically. Acid and peroxide values were determined by standard methods. Antioxidant properties of the oil were estimated as 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging capacity.

Results and discussion. The ultrasound degumming leads to an increase in the yield of degummed oil by 2-2.5 % compared to water degumming with maximal yield at ultrasonic treatment duration from 5 to 11 min. The ultrasonic degumming as well as water degumming decreased the phosphorus content from 98 ppm in crude oil to 28-32 ppm in the degummed sunflower oil. The oil acidity was reduced substantially due to ultrasound degumming, the peroxide value demonstrated some increase, and the antioxidant capacity of the sunflower oil varied from 18.6 to 26.9 % of DPPH radical scavenging as a result of degumming.

Ultrasound degumming at fixed temperatures of 30 and 60 °C resulted in close phospholipids content in degummed oil. The minimal phospholipid content in the range from 24 to 35 ppm of phosphorus was observed after ultrasound degumming during 6-9 min at both temperatures. Ultrasound degumming at 60 °C resulted in an acid value increase from 0.64 mg KOH/g at the beginning to 1.1 mg KOH/g after treatment for 15 min. The acid value of oil after ultrasound degumming at 30 °C was not higher than in the control at any duration of treatment and was in the range from 0.5 to 0.7 mg KOH/g. The peroxide values of oil after ultrasound degumming at 60 °C were higher compared to the control oil and samples after ultrasound degumming at 30 °C.

Conclusion. The ultrasound degumming accelerated the removal of phospholipids from sunflower oil and increased the yield of degummed oil. Ultrasound degumming can be recommended for the oil industry and it is important to carry out ultrasound treatment of the oil at low temperatures.

Introduction

The degumming of vegetable oils removes various hydrophilic substances from oils, such as phospholipids and other gums. The phospholipids of vegetable oils have different levels of hydrophilicity, one part is more hydrophilic (more polar) and another, which is less hydrophilic. More hydrophilic phospholipids are called hydratable, and they could be removed by water degumming. The other part of the phospholipids are nonhydratable, and acid degumming is required to remove them from oil. Water degumming is a sustainable method of oil processing (Hamm et al., 2013) and it is possible to obtain food-grade lecithin. However, water degumming does not provide a high level of phospholipid removal and oil yield as well as requires a long exposition time. Thus, improving oil degumming is important for the oil industry.

One of the methods for phospholipids removal from oil is enzymatic degumming with various types of phospholipases (Dijkstra, 2010; Gofferjé et al., 2014; Guerrand, 2017). The effectiveness of new types of phospholipase preparations with phospholipase A and C activity for improving sunflower oil degumming was demonstrated (Nosenko and Zhupanova, 2023). However, phospholipase preparations are sensitive to degumming conditions and are still expensive for the industry.

Various methods of physical treatment are used for improving oil degumming as well as other processes of oil processing. In particular, ultrasonic treatment is widely used in food technology to accelerate chemical reactions and technological processes, such as extraction (Sun et al., 2023; Tiwari, 2015) and emulsion obtaining (Bernardi, et al, 2021; Soria and Villamiel, 2010). Ultrasound treatment is relatively cheap, simple, and energy saving and has become a promising technology to be used for food processing (Chew and Ali, 2021).

Ultrasound is a sound wave with a frequency greater than the upper limit of the human hearing range. An ultrasonic wave is a mechanical wave with a frequency range of $10^4 \sim 10^{12}$ Hz. When the ultrasonic wave spreads in the medium, it produces a pressure wave, which forms a high-pressure region (compression region) and a low-pressure region in the medium. In the region of high pressure and low pressure, the medium molecules contract and expand respectively. In the process of expansion, the bounds between molecules are pulled apart to produce tiny cavities or microbubbles. When the critical radius is reached, a very high local energy density is released due to the explosion, which is called the cavitation effect. At the moment of collapse of ultrasonic cavities, local high temperature, high pressure, and accompanying strong shock waves are generated around it (Ashokkumar and Mason, 2007; Legay, et al., 2011; Chemat et al., 2016). The shock wave, shear, and vibration enhance the movement of macromolecules, and particles and significantly increase the mass transfer rate, which is very important for chemical reactions in heterogeneous systems.

Several studies were devoted to the ultrasound-assisted enzymatic degumming of crude oil. Jiang et al (2014) detected that ultrasonic treatment had increased the rate of enzymatic degumming reactions of rape oil resulting in phosphorus content of less than 10 ppm during 2 h. At the same time, 4 h is needed to achieve the same phosphorus content under mechanical mixing of the reaction mixture. However, it was shown that the use of ultrasonic treatment could reduce the oxidative stability of rapeseed oil.

The authors (More and Gogate, 2018) studied the ultrasound-assisted enzymatic degumming of crude soybean oil, quantifying the extent of degumming. The influence of different operating parameters such as enzyme content, pH, presence of water, temperature, and ultrasonic power on the enzymatic degumming of crude oil has been investigated. Ultrasound combined with enzyme at a dose of 2.0 ml/L resulted in an extent of degumming of 92.2% under ambient conditions. The addition of water (5%) in combination with ultrasound and enzyme at 2.0 ml/l and pH of 5 resulted in maximum extent of degumming (98.4%) during 120 min of treatment. Scale-up studies were performed at 500 ml and 1 l operating volume under optimized conditions of 2.0 ml/l as the enzyme loading, pH of 5, 5% water addition, and ultrasonic power of 100 W where 93.63% and 91.15% phospholipid separation respectively was obtained. The effects of ultrasonic treatment on the acid value reduction and oxidative stability for the processed oil were estimated too. It was demonstrated the reduction in acid value (final value less than 1) and oxidative stability (TOTOX less than 4) were obtained using enzymatic degumming. Overall, the approach of enzymatic degumming was established to show much higher efficacy for soybean oil processing as compared to only ultrasound or only enzymatic treatment.

Ultrasound-assisted enzymatic degumming was applied also to the arachidonic acid oil produced by *Mortierella alpina* (Guo et al., 2021). The conditions of the degumming process were optimized by response surface methodology. The efficiency of degumming 98.82% was achieved under optimum conditions of 500 U/kg of phospholipase A_1 (PLA_1) dosage, 2.8 mL/100 g of water volume, 120 min of ultrasonic time, and 135 W of ultrasonic power. The phosphorus content of ultrasonic-assisted enzymatic degumming oil was 4.79 mg/kg, which was significantly lower than that of enzymatic degumming oil (17.98 mg/kg). The oxidation stability of the same oil after enzymatic degumming of crude oil was equivalent to that after enzymatic degumming and weaker than crude oil.

As Ukraine is a main sunflower oil producer, improvement of its processing is important for oil industry. In our previous work, the influence of the different types of phospholipases on the efficacy of sunflower oil degumming was shown (Nosenko and Zhupanova, 2023). However, industrial use of phospholipases is still complicated by their high sensitivity to degumming conditions and oil composition. Thus, it is important to improve the oil degumming efficacy by simple and accessible methods. The objectives of this study were to investigate the influence of the ultrasound itself on the sunflower oil degumming.

Materials and methods

Crude sunflower oil was purchased from the local market.

Ultrasound degumming

Crude sunflower oil, 100 g, with 5 % of distilled water was placed into the ultrasound mixer MEDITON (Ukraine) and treated for 5 to 15 minutes at ultrasonic treatment frequency 44 kHz. Every 3 min the samples were taken for analysis. Mixture was kept for 30 min at room temperature, 10 ml of the reaction mixture was put into a graduated centrifuge tube and the gums were separated from the oil by centrifugation at 2000 rpm for 10 min.

For thermostated degumming ultrasound thermostat Bandelin Sonorex (Austria) was used and a mixture of 100 g crude sunflower oil with 5 % of distilled water was treated at 30 or 60 °C at ultrasonic treatment frequency 28 kHz. Every 3 min the samples were taken for analysis.

The water degumming (control sample) was carried out according to the next procedure. Crude sunflower oil, 100 g, was placed into a 250-ml conical flask. The oil was heated to about 60 °C followed by the addition of 5 % of distilled water. The mixture was thoroughly mixed for 1 min and stirred at 60 °C for 1 h. After that, reaction mixture was kept for 30 min at room temperature. The 10 ml of the reaction mixture was put into a graduated centrifuge tube and the gums were separated from the oil by centrifugation at 2000 rpm for 10 min.

Oil yield determination

The volume of degummed oil was measured after gums separation and oil yield (X, % of crude oil) was calculated as

$$X = (b/9.52)\cdot 100,$$

where 9.52 was the volume of the crude oil in 10 ml degumming mixture before centrifugation in ml, and b was the volume of separated oil in ml.

Phosphorus content analysis

The oil samples were ashed for phosphorus content determination according to (Yang et al., 2006). 0.7 g of MgO and 0.6 to 0.7 g of oil were weighed and heated in the oven at 110 °C for 10 min. After that, the samples were carbonized by heating on the hot plate and then ashed in an electric muffle furnace at 600 °C until a constant mass was achieved.

The phosphorus content of the ash was determined according to the AOCS method Ca 12–55 (1997). Cold ash was transferred to a 100 ml flask, 10 to 12 ml water, and 20 ml 2N H_2SO_4 were added. The solution was heated to dissolve the ash. After that 20 ml molybdenum reagent was added and the solution was heated in boiling water bass for 30 min. The volume of the cold solution was adjusted to 100 ml. Photometric analysis was carried out on UV/VIS spectrometer PerkinElmer Lambda 35 at 750 nm.

A standard solution of KH_2PO_4 (concentration 10 μg/ml) was used for the calibration curve obtaining. The calibration solution set was prepared in the same condition as the ash samples. The concentration of KH_2PO_4 in the calibration solution set was adjusted from 0.05 to 2.0 μg/ml.

Phosphorus content (P, % of oil mass) was calculated as follows:

$$P = 0.01\ d/m,$$

where d was the phosphorus content in ash solution according to a calibration curve, μg/ml, m was oil mass, g.

The content of phospholipids as stearoiloleil phosphatidylcholine (PC, % of oil mass) was calculated as follows:

$$PC = 25.4\ P,$$

where P was the phosphorus content, % of oil mass, and 25.4 was a coefficient for calculation of the stearoiloleil phosphatidylcholine mass on the base of phosphorus content.

Chemical parameters of oils

The acid value of oil samples was determined by the titrimetric method according to ISO 660:2020.

The oil's peroxide value was determined using the iodometric method according to ISO3960:2017.

Antioxidant activity

The radical scavenging capacity was determined by the 2,2-diphenyl-1-picrylhydrazyl (DPPH) method. For the DPPH test, DPPH was dissolved in a small volume of ethyl acetate and diluted with ethyl acetate by adjusting the absorbance to 0.700 ± 0.020 at 520 nm. 100 mg of oil was weighed in a test tube and 15 ml DPPH$^{\bullet}$ free radical solution was added (Vovk et al., 2023). The sample was agitated and absorbance was measured at 520 nm against ethyl acetate. After 30 min of incubation in darkness, absorbance was measured against ethyl acetate on UV/VIS spectrometer PerkinElmer Lambda 35 at 520 nm.

The results of the DPPH test were expressed as % of DPPH$^{\bullet}$ free radicals that were scavenged by antioxidants in 100 mg oil (A, %):

$$A = (1 - D_1/D_0) \cdot 100,$$

where D_0- was the absorbance of the reaction solution at 520 nm before incubation, and D_1- was the absorbance of the reaction solution at 520 nm after incubation.

Statistical analysis

Each sample was analyzed in triplicate, and the results were reported as mean ± standard deviation. Differences were considered to be significant at validity α=0.95.

Results and discussion

Effect of the ultrasound degumming on the sunflower oil yield and properties

To evaluate the effect of ultrasonic treatment on the effectiveness of sunflower oil degumming, we determined the yield of oil after ultrasound degumming. The duration of ultrasonic treatment was from 5 to 15 minutes in the presence of 5 % water. The control oil sample was treated with the same amount of water with constant stirring for 1 h at 60 ºC.

The obtained results demonstrated that using ultrasound degumming leads to an increase in the yield of degummed oil by 2–2.5% (Figure 1). The maximal yield was observed under ultrasonic treatment duration from 5 to 11 min followed by a slight decrease in oil yield. The last phenomenon may be due to the enhancement of the oil emulsifying under the continued ultrasonic treatment.

The most important parameter of the oil degumming is the phospholipid content in the oil. It is known that water degumming itself can remove only part of phospholipids from vegetable oils, which are called hydratable phospholipids. The ratio of hydratable phospholipids to nonhydratable phospholipids in vegetable oils depends on different factors - oil nature, method of obtaining, condition, and duration of storage. For entrance, in our previous work about half of sunflower oil phospholipids were hydratable phospholipids (Nosenko and Zhupanova, 2023). In the present study, content of phospholipids had dropped sharply after water degumming of sunflower oil (Figure 2). About 68 % of phospholipids were removed by water degumming.

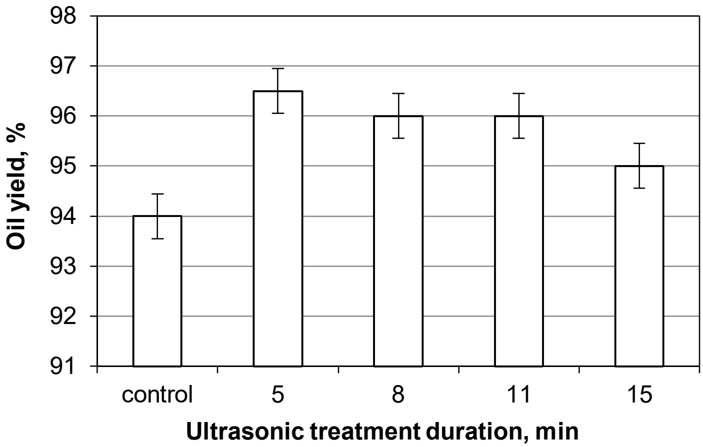

Figure 1. Influence of the ultrasound degumming on the sunflower oil yield.

Other researchers had obtained that about 80 % of the initial phospholipids content in sunflower oil were hydratable phospholipids (Zufarov et al., 2008; Zufarov and Serkayev, 2023) and the residual amount of phosphorus after water degumming at 80 °C during 15 min was in the range from 50.1 to 56.9 ppm. At the same time, phosphorus content in water-degummed soybean oil was 127 ppm (Dijkstra and Opstal, 1989).

The ultrasonic treatment did not sufficiently affect the oil phospholipid content compared with water degumming. The phospholipid content in the degummed sunflower oil was in the range of 0.07-0.08 % of stearoiloleil phosphatidylcholine, which corresponds to 28-32 ppm phosphorus (Figure 2).

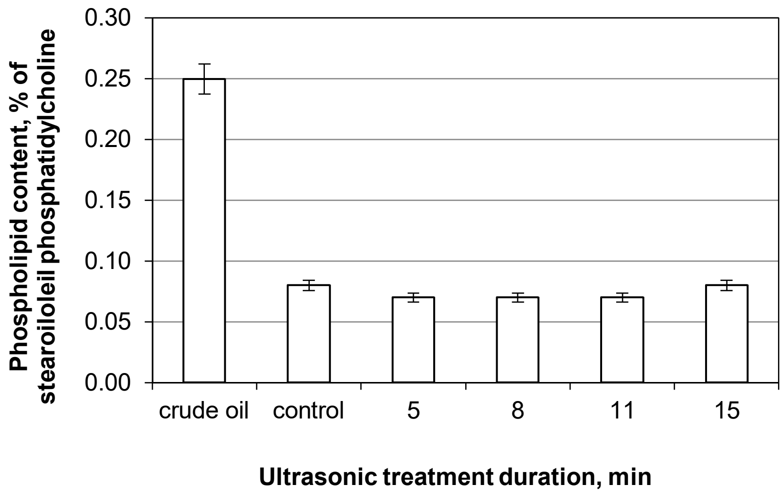

Figure 2. Influence of degumming on the phospholipid content of the sunflower oil (ultrasonic treatment frequency 44 kHz)

Simultaneously with the phospholipid content decrease, we observed the reduction of oil acidity (Figure 3). The acid value of crude oil was high 5.6 mg KOH/g and declined substantially due to ultrasound degumming. Thus, it was 3.1 mg KOH/g in oil after 11 min ultrasound treatment. Such as there was a correlation between phospholipid content and the acid value of the oil, we can suppose, that the oil acidity decrease was mainly due to acid phospholipids removing, first, phosphatidic acid.

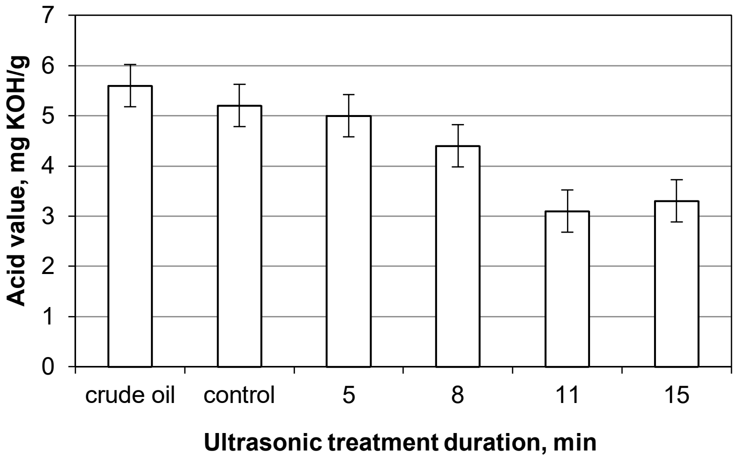

Figure 3. Influence of ultrasound degumming on the acid value of the sunflower oil (ultrasonic treatment frequency 44 kHz)

The peroxide value of crude sunflower oil in this experiment was 4.2 meq O/kg. There was a slight rise in peroxide value because of degumming (Figure 4). The influence of ultrasound treatment was unimportant; the peroxide value was in the range from 5.1 to 5.9 meq O/kg.

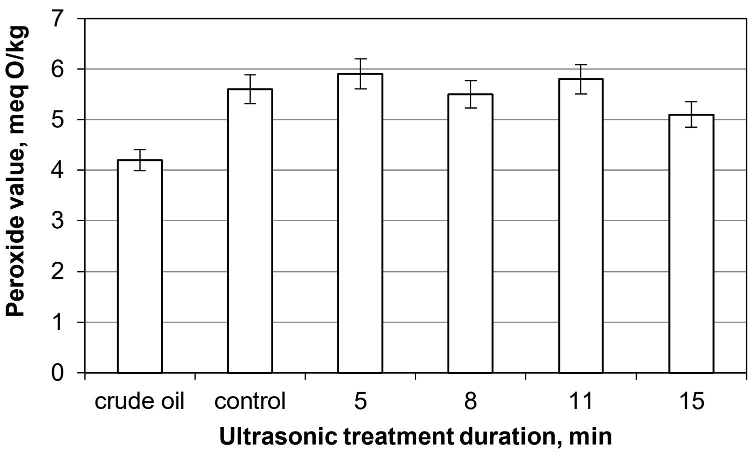

Figure 4. Influence of ultrasound degumming on the peroxide value of the sunflower oil (ultrasonic treatment frequency 44 kHz).

The water and ultrasound degumming had no substantial effect on the antioxidant capacity of the sunflower oil (Figure 5). The antioxidant capacity of the degummed sunflower oil varied from 18.6 to 26.9 % of DPPH scavenging, the crude oil antioxidant capacity was 20.4 % of DPPH. A slight rise in the antioxidant property after degumming could be caused by metal removal, which is known as prooxidants (Zufarov and Serkayev, 2023). On the other hand, long ultrasonic treatment could result in antioxidant inactivation.

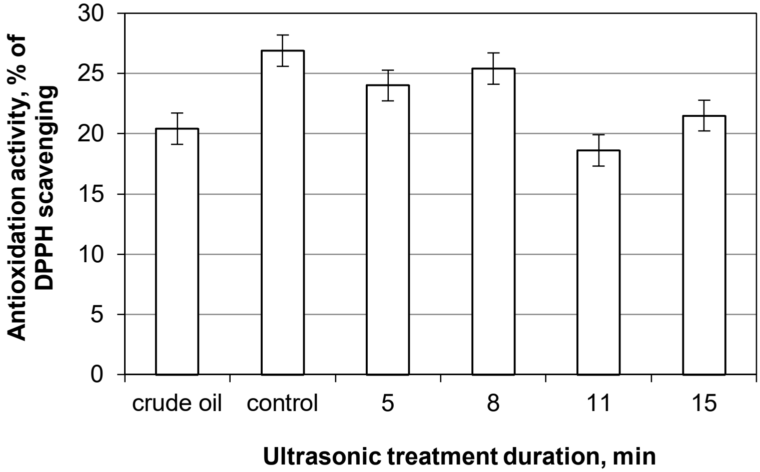

Figure 5. Influence of ultrasound degumming on the antioxidant capacity of the sunflower oil (ultrasonic treatment frequency 44 kHz).

Effect of the ultrasound degumming at different temperatures on the sunflower oil quality

Such as ultrasonic treatment results in an increase in the oil temperature to 55- 60 °C it is important to separate the influence the ultrasound and heating on the effectivity of the oil degumming. For this, we carried out ultrasound degumming at the constant temperature and ultrasonic treatment frequency of 28 kHz. Ultrasound degumming at fixed temperatures of 30 and 60 °C results in close phospholipids content in degummed oil (Figure 6). The minimal phospholipids content in the range of 0.06 to 0.09 % of stearoiloleil phosphatidylcholine was observed after ultrasound degumming during 6-9 min. There was a drop of phospholipids content in degummed oil only after ultrasonic treatment for 9 min at 60 °C. It was the lowest phospholipids content, which corresponds to 24 ppm phosphorus in sunflower oil. Prolongation of ultrasound treatment was accompanied by a phospholipid content increase, which could be due to phospholipids stabilization in oil because of emulsifying.

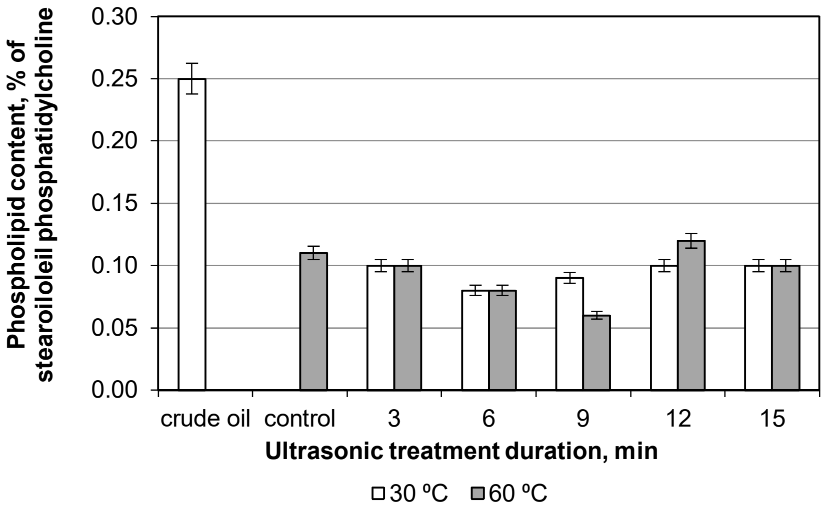

Figure 6. Influence of the ultrasound degumming at different temperatures on the phospholipid content in the sunflower oil (ultrasonic treatment frequency 28 kHz)

It is important also to study the effect of ultrasound degumming on the other parameters of oil quality, first, the acidity of the oil. Increasing the free fatty acid content in the oil could result in the loose of oxidation stability followed by a shelf life decrease. Ultrasound degumming at 60 °C was accompanied by acid value increase from 0.64 mg KOH/g at the beginning to 1.1 mg KOH/g after 15 min treatment (Figure 7).

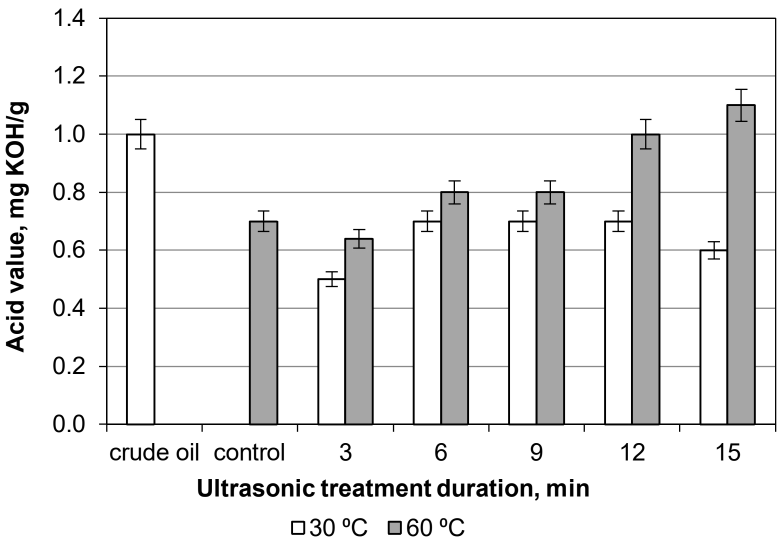

Figure 7. Influence of the ultrasound degumming at different temperatures on the acid value of the sunflower oil (ultrasonic treatment frequency 28 kHz)

It is supposed that higher temperature together with ultrasound could accelerate the hydrolysis of acylglycerols followed by free fatty acids release. It is obvious, that under ultrasound degumming at low temperature acid value of oil was not higher than in the control at any duration of treatment and was in the range from 0.5 to 0.7 mg KOH/g.

It was shown, that water degumming, acid degumming, and TOP (total degumming process) degumming result in removing of metals from rapeseed and sunflower oils enhancing the oxidative stability of these oils (Zufarov and Serkayev, 2023). In our previous study, we detected that water and enzymatic degumming were accompanied by a decrease in peroxide content in the sunflower oil (Nosenko and Zhupanova, 2023).

The data obtained in the present study have demonstrated that ultrasound degumming results in a decrease in the peroxide value of sunflower oil in every sample compared with crude oil (Figure 8). However, oil samples after ultrasound degumming at 60 °C had higher peroxide values than control oil and samples after ultrasound degumming at 30 °C except the samples of long ultrasound degumming. The long ultrasound treatment usually was accompanied by a reduction of quality parameters of oil. Thus, we observed higher phospholipids content, and acid value of oil after ultrasound degumming at 60 °C during 12-15 min compared with 6-9 min treatment.

The content of oxidation products and oxidation stability in vegetable oil depends on numerous internal and external factors, such as fatty acid composition, antioxidant capacity, quality of oil seeds, processing technology, and conditions of storage. Thus, the content of oxidation products in the same oil can be very different.

Commonly, the ultrasound degumming did not have a prominent negative effect on the peroxides content in the oil. We are suggesting that the removal of phospholipids because of degumming can be accompanied by the removal of peroxides and prooxidants, mainly metals from the oil. The increase of peroxide content in some samples could be the result of stable emulsion creation after long ultrasound treatment.

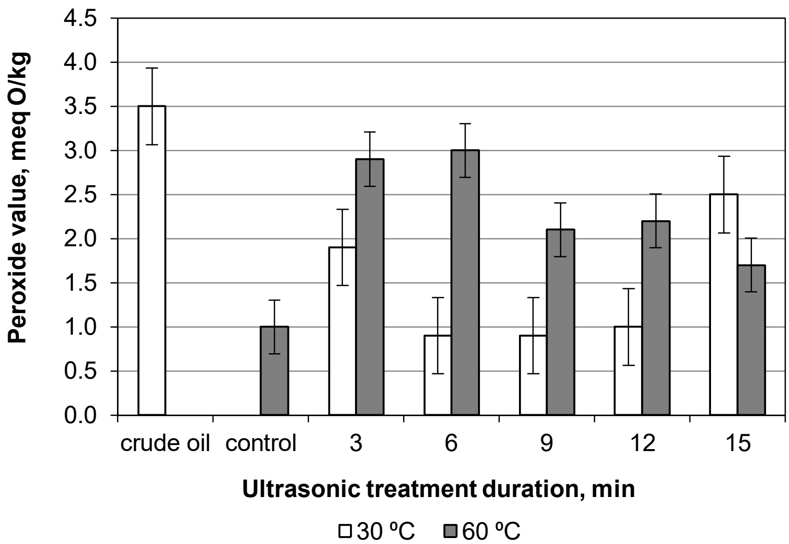

Figure 8. Influence of the ultrasound degumming at different temperatures on the peroxide value of the sunflower oil (ultrasonic treatment frequency 28 kHz)

Conclusions

1. The ultrasound degumming is a prominent tool to reduce the duration of this process and increase the oil yield. The maximal degummed oil yield was 96–96.5 % at ultrasonic treatment duration from 5 to 11 min which was higher by 2–2.5% compared to water degumming during 60 min.
2. The water degumming for 60 min and ultrasound degumming for 5 min had the same effect on the phospholipid content in the oil. Because of degumming, the phosphorus content decreased from 98 ppm in crude oil to 28–32 ppm in degummed sunflower oil.
3. Ultrasound degumming at fixed temperatures of 30 and 60 °C resulted in close phospholipids content in degummed oil. The minimal phospholipids content in the range from 24 to 35 ppm of phosphorus was observed after ultrasound degumming during 6-9 min.
4. It is worth carrying out ultrasound treatment of the oil at low temperatures such as the simultaneous effect of ultrasound and high temperature can stimulate the hydrolytic and oxidative reactions in the oil.

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