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The Use of Ultrasound in Shaping the Properties of Ice Cream with Oleogel Based on Oil Extracted from Tomato Seeds

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Featured Application: The research presents the development of innovative ice cream based on oleogels obtained from oil pressed from tomato seeds using ultrasound as a substitute for traditional pasteurization. Such a product can be put into industrial production.

Abstract: In this study, the possibility of using ultrasound technology as an alternative to traditional pasteurization and homogenization in ice cream production was presented. Three types of ice cream with different proportions of oleogel (5, 6, and 7%) prepared using tomato seed oil were studied. The fatty acid contents of the oil were analyzed. Using chemical analysis, dry matter, fat, protein, dietary fiber, ash, and pH of the ice cream samples were determined. The physical analysis included analysis of the ice cream samples using a differential scanning calorimeter (DSC) and determination of their first drop time, complete melting time, overrun, viscosity, hardness, and adhesiveness. The structure of the samples was evaluated using scanning electron microscopy. Fourier transform infrared spectroscopy spectra were measured using a dedicated QATR-S Single-Reflection ATR ACCESSORY with a diamond prism. With the increase in the proportion of oleogels, the fat and carbohydrate contents, the amount of freezable water, and the overrun of the samples were increased, whereas their viscosity and hardness were decreased. Oleogels were found to be a promising alternative to fat in ice cream rich in unsaturated fatty acids, and the ice cream samples prepared using ultrasound pasteurization showed lower overrun and viscosity and higher hardness.

Keywords: ice cream; ultrasound; tomato seed oil; oleogel; SEM; FTIR spectroscopy

1. Introduction

Ice cream is prepared by freezing a pasteurized and cooled liquid mixture that is made based on milk, cream, or fruit juices with sugar, emulsifiers, stabilizers, and flavorings and is intended for direct consumption [1]. The structure of ice cream is a complex arrangement of dispersed air bubbles in an incompletely frozen continuous phase, which consists of 60–78% water [2]. The ingredients of ice cream are classified into three types: emulsions (stabilizers, fats), suspended solutions (salts, sugars), and colloids (proteins, stabilizers). To the appropriate relationship between the physical states of various components in ice cream, it is important to optimize the production process and understand the individual processes,

such as pasteurization and homogenization of the ice cream mixture, and freezing [3]. The pasteurization process is especially important, which, besides inactivating pathogenic bacteria, transforms the mixture into a product that is ready for freezing. Homogenization of the ice cream mixture is closely related to the pasteurization process. During homogenization, fat particles are broken down, and stable homogeneous emulsions of liquids prone to forming suspensions are created. Maintaining the size of the fat particles at around 1 μm improves the consistency of ice cream, reduces its aeration time, and increases its melting time [4].

Ultrasound is a novel and promising technology in ice cream production that can minimize processing time, increase quality, improve processing effectiveness and efficiency, and ensure food safety while extending the shelf-life of ice cream. Ultrasound technology is generally considered safe, nontoxic, and environmentally friendly, thus having important advantages over other innovative techniques. Ultrasound is defined as sound waves of frequencies beyond the human hearing threshold, typically higher than 20 kHz. Based on the frequency and energy amount or sound intensity, ultrasound technology in the food industry is classified into two types: high- and low-intensity ultrasound. Low-frequency, high-intensity ultrasound shows great potential for a wide range of applications in dairy processing. It provides high power, which is enough to generate cavitation, so it can produce mechanical, chemical, and biochemical effects in liquids. These effects are used to modify the physicochemical properties and enhance the quality of various food systems during processing [5,6]. In ice cream production, ultrasound is used in the homogenization process [7], which reduces the ice crystal size, decreases the freezing time, and prevents incrustation on cold surfaces [6].

The quality of ice cream is determined by the ingredients used, such as solid raw materials (milk and/or vegetable fat, sugar, egg powder, milk powder, stabilizers, emulsifiers, starch hydrolysis products) and liquid raw materials (milk, water, cream). It is important to use the correct proportions of the major ingredients, especially the fat to sucrose ratio and the nonfat dry matter to water ratio [8]. One of the essential ingredients in any type of ice cream is fat. Fat stabilizes ice crystals during the freezing process, facilitates the aeration of the mixture, increases the resistance of ice cream to melting, improves the viscosity and flavor, and imparts a smooth texture. In general, the fat content of ice cream is around 8%. Most often, liquid or powdered cream is used as milk fat, and butter, butter oil, or anhydrous fat is also used. An alternative to fat is oleogels derived from vegetable oils. Oleogels are formed by stabilizing oil in a network created by a gelling medium, and thus, they have a viscous and elastic consistency. Due to oleogelation, oil molecules are trapped in a three-dimensional network, resulting in no change in their chemical composition, and the resulting structure has the properties of a solid [9,10]. In addition, organogels with high gelator concentrations can be used to prepare ice creams with quality characteristics comparable to those of the samples containing milk cream. Organogels are successfully used in artisanal ice cream to prepare low-saturated-fat products (saturated fat < 0.9 g/100 g) with added plant sterols, intended for people who wish to lower their blood cholesterol level [11].

One of the major byproducts of the tomato concentrate industry is tomato seeds. Tomato pomace consists primarily of skin and seeds. Tomato seeds can be separated from the pulp and skin using a sedimentation system, dried, and used in oil extraction. Tomato seed oil has a high content of unsaturated acids—linoleic acid contributing to more than 50% and oleic acid to more than 20%—which prevents floral vasodilation, thrombosis, increase in cholesterol content, and atherosclerosis. It also contains proteins with good functional and nutritional properties. In addition, antioxidant properties of tomato seeds have been reported [12–14].

This study aimed to investigate the potential use of tomato seed oil in the form of oleogels in ice cream production and the possibility of using ultrasound pasteurization as an alternative to traditional pasteurization. Thus, ice cream mixtures with different percentages of oleogels were prepared and subjected to both traditional and ultrasound

pasteurization. The physicochemical properties of the resulting ice cream mixtures were analyzed using differential scanning calorimetry, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM).

2. Materials and Methods

2.1. Ingredients

The following ingredients were used: lactose-free whole milk (Mlekovita, Wysokie Mazowieckie, Poland); lactose-free 30% cream (OSM, Łowicz, Poland); lactose-free skimmed milk powder (Mlekovita, Wysokie Mazowieckie, Poland); commercially cold-pressed tomato seed oil (Turkey) (purchased commercially), fructose (NaturaVena, Piaseczno, Poland); emulsifiers E471 mono- and diglycerides of fatty acids, soy lecithin (Sosa, Spain), and E464 hydroxypropyl methylcellulose (HPMC) (Sigma-Aldrich, St. Louis, MO, USA); and stabilizers E412 guar gum (Agnex, Białystok, India) and E407 carrageenan (Agnex, Białystok, Spain).

2.2. Obtaining and Analyzing Oleogel

An oleogel containing a 60% fat phase and 40% aqueous phase was obtained from cold-pressed tomato seed oil. The aqueous phase was a 2% solution of HPMC. Several stages were involved in the preparation of HPMC oleogel. First, the aqueous phase (2% hypromellose solution) was prepared using a rotary homogenizer (Unidrive X1000D Homogenizer, CAT) at 11,000 rpm for 3 min. Then, the homogenization process was carried out—the aqueous phase was combined with the fat phase and an oil-in-water emulsion was obtained. In the next stage, the emulsion was placed in an aluminum vessel with a diameter of 10 cm and dried at 80 °C for 4 h (laboratory dryer) (Figure 1a). Upon the removal of the water content, an oleogel with a new rigid structure was obtained (Figure 1b). Finally, the dried oleogel was homogenized (11,000 rpm) until a semisolid mousse consistency was obtained (5 min) (Figure 1c).

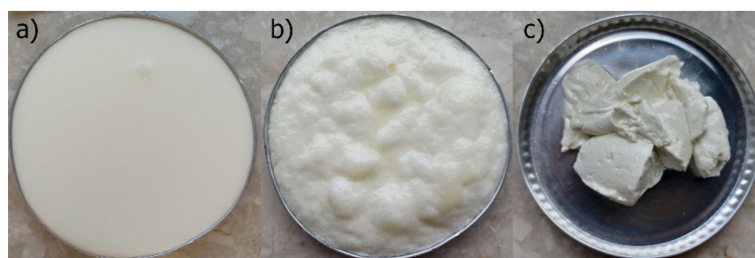


Figure 1. Stages of oleogel formation: (a) emulsion, (b) oleogel after drying, and (c) oleogel after homogenization.

The resulting oleogel was subjected to stability analysis, which was carried out by centrifugation (SOMW) at 21 °C. The oleogel samples were centrifuged at 3000 rpm (Mikro 220 Classic centrifuge, Hettich, Germany) for 20 min. After the removal of the liquid phase, the residue (in a test tube with the gel) was weighed in triplicate. SOMW was calculated from the modified formula [15–17]:

$$SOMW = \frac{(M_{pw} - M_p)}{(M_p - M_p)} \cdot 100\% \quad (1)$$

where:

M_{pz} —is the mass of a test tube including the obtained gel (g);

M_p —is the mass of the empty test tube (g);

M_{pw} —is the mass of the tube and oleogel after centrifugation (g).

2.3. Recipe and Preparation of Ice Cream

Six ice cream mixtures were developed using fixed proportions of ingredients, such as: $55.0 \text{ g} \times (100 \text{ g})^{-1}$ lactose-free whole milk, $15.0 \text{ g} \times (100 \text{ g})^{-1}$ lactose-free 30% cream, $14.4 \text{ g} \times (100 \text{ g})^{-1}$ fructose, $0.1 \text{ g} \times (100 \text{ g})^{-1}$ soy lecithin, $0.125 \text{ g} \times (100 \text{ g})^{-1}$ guar gum, $0.125 \text{ g} \times (100 \text{ g})^{-1}$ carrageenan, and $0.25 \text{ g} \times (100 \text{ g})^{-1}$ mono- and diglycerides of fatty acids. The oleogel and lactose-free skimmed milk powder accounted for 5.0, 6.0, and 7.0 $\text{g} \times (100 \text{ g})^{-1}$; and 10.0, 9.0, and 8.0 $\text{g} \times (100 \text{ g})^{-1}$, respectively. All ingredients were mixed and then subjected to two pasteurization methods: traditional pasteurization at 65.0 °C for 20 min and ultrasound pasteurization at 65.0 °C and 20 kHz for 15 min. After traditional pasteurization, homogenization was carried out at a probe speed of 12,000 rpm for 5 min. The ice cream mixtures were subjected to 18 h of maturation at 6.0 °C. Then, they were aerated for 5 min using a Bosch mixer and frozen. The mixtures were packed in polystyrene containers, tempered for 24 h, and then stored for a week. The ice cream mixtures were labeled as follows: PP5-ice cream with 5% oleogel subjected to traditional pasteurization; PP6-ice cream with 6% oleogel subjected to traditional pasteurization; PP7-ice cream with 7% oleogel subjected to traditional pasteurization; UP5-ice cream with 5% oleogel subjected to ultrasound pasteurization; UP6-ice cream with 6% oleogel subjected to ultrasound pasteurization; and UP7-ice cream with 7% oleogel subjected to ultrasound pasteurization. For physicochemical analyses, the mixtures were freeze-dried.

2.4. Analysis of Oil

The saturated and unsaturated fatty acid contents of commercially cold-pressed tomato seed oil were determined using column chromatography. The fatty acid content of the isolated samples was determined using gas chromatography (Bruker 436GC chromatograph with FID detector) in accordance with the relevant standards [18]. Fatty acid methyl esters were separated on a BPX 70 capillary column (60 m, 0.25 mm, 25 m), using nitrogen as the carrier gas.

2.5. Analysis of Chemical Composition

Dry matter, fat, protein, and ash were determined according to the AOAC [19] standards. The total carbohydrate content was calculated as the difference between 100 and the sum of the percentages of water, protein, total fat, and ash contents. Once the water, fat, protein, ash, and dietary fiber contents were determined, the digestible carbohydrate content was calculated from the difference. The calorific value was calculated using Atwater equivalents: 4.0 kcal for 1.0 g of protein, 9.0 kcal for 1.0 g of fat, 2.0 kcal for 1.0 g of dietary fiber, and 4.0 kcal for 1.0 g of carbohydrates [20]. The pH values of the ice cream mixtures were measured using a pH meter (ELMETRON CP-401) connected to a glass electrode at 20 °C. All measurements were carried out in triplicate.

2.6. Physical Properties of Ice Cream

After the maturation stage, the thermal properties of the ice cream mixtures were analyzed using a Mettler Toledo differential scanning calorimeter (DSC) (model DSC1). A sample of an ice mixture (about 10 mg) was placed in a preweighed aluminum crucible, which was sealed using a clamping device. This test was carried out at temperatures from −40 to +20 °C in a nitrogen atmosphere at a heating rate of 2 °C·min^{−1}. The reference test was conducted using an empty crucible. After the test was completed, the freezing temperatures and heat of fusion were measured. The amount of frozen water (*FW*) was calculated using the following formula [21,22]:

$$FW = \frac{\Delta H_m}{H_f \cdot m_s} \cdot 100\% \quad (2)$$

where:

ΔH_m —is the heat of fusion ($\text{J} \cdot \text{g}^{-1}$),

H_f —is the theoretical heat of ice fusion at 0 °C (333.50 J·g⁻¹),
 m_s —is the sample mass (g).

The overrun was determined according to the relationship of the weight difference between the ice cream mixture and the ice cream itself to the weight of the ice cream [23].

The melting resistance of the ice cream mixtures was determined by measuring the first drop time and the complete melting time of a given volume of ice cream. Frozen samples (28 g) in the shape of a cylinder with a height of 40 mm and a diameter of 30 mm were placed on a funnel with a grid and a beaker (at room temperature of 21 °C) and left to melt completely [24].

Hardness and adhesiveness were determined using an LFRA texture analyzer (Brookfield Engineering Laboratories, Inc., Middleboro, MA, USA). The samples, with a height of 25 mm and a diameter of 30 mm (at -6.0 °C), were subjected to a penetration test with a reverse extrusion chamber. The hardness of the samples was determined by the maximum force [N] required to compress 80% of the sample height, and the adhesiveness was determined based on the negative force recorded during the withdrawal of the probe. The following parameters were used for the analysis: 5 cm probe diameter, 2 mm/s probe velocity during penetration, and 0.2 N pressing force [25].

The viscosity of the ice cream mixtures was determined from the melted samples using a viscometer from IKA (ROTAVISC lo-vi Complete). An SP-4 spindle was used in the measurement of viscosity, which was immersed into a previously prepared sample at 20 °C and a shear rate of 200 rpm. The viscosity value was read after 2 min.

FTIR measurements were performed using a dedicated QATR-S Single Reflection ATR ACCESSORY with a diamond prism. All measurements were taken at room temperature (T = 21 °C). In addition, each spectrum was taken as an average of 40 scans. The spectra were measured using an IR Spirit spectrometer (SHIMADZU). Before each measurement, the crystal was cleaned using ultrapure solvents purchased from Sigma-Aldrich. The spectra were measured with a resolution of 4 cm⁻¹ in the spectral range from 4500 to 250 cm⁻¹. Then, they were subjected to Fourier transformation and averaged. They were analyzed and processed using Grams/AI 8.0 software from Thermo Fisher Scientific, USA. Measurements were taken at the Department of Biophysics of the University of Life Sciences in Lublin. All measurements of infrared spectra were taken in triplicate for each sample.

The structural analysis was performed using scanning electron microscopy SEM. The samples were sputter-coated with a 20-nm layer of gold using a Sputter Coater (Emitech SC7620). The surface of the samples was examined using a Carl Zeiss Ultra Plus scanning electron microscope (Germany), and the samples were imaged using a secondary electron detector at 3 kV.

3. Statistical Analysis

The results were statistically analyzed using StatSoft STATISTICA 13.1 program. The significance of differences between the mean values of the established parameters was verified using the Tukey test. Calculations were performed at a significance level of p value < 0.05.

4. Results and Discussion

The stability of the binding of tomato seed oil was evaluated using a gelling agent. As a result of centrifugation, the oleogel was separated into two phases: liquid and solid. The stability index of the oleogel was 88.54%, which indicates relatively high stability, with only 11.46% of the oil present in the liquid phase. In study [17], there is a higher binding capacity of edible oil using rapeseed oil as an example. They reported a 90.96% stability of the oleogel structure.

Food fat is a concentrated source of energy and contributes to the proper functioning of the body. Tomato seed oil analysis showed that 87.42% of food fats are unsaturated fatty acids, of which 60.68% are polyunsaturated fatty acids [26]. The proportion of C18:2 linoleic acid of the omega-3 group was the highest (60.58%), whereas C18:3 (n-3) linolenic acid and C18:1 oleic acid (n-9) accounted for 10% and 26.74%, respectively (Table 1). The results

obtained were compared with those of high-linoleic oils such as black seed oil, grape seed oil, and wheat germ oil. Tomato seed oil showed a comparable linoleic acid content with black seed oil (60.92%), a higher linoleic acid content than grape seed oil (66.60%), and a lower linoleic acid content than wheat germ oil (57.85%) [27–29].

Table 1. Fatty acid content in tomato seed oil.

	Fatty Acid	Content [%]
Saturated fatty acid	Myristic acid C14:0	0.08
	Palmitic acid C16:0	6.48
	Margaric (heptadecanoic) acid C17:0	0.03
	Stearic acid C18:0	3.30
	Arachidic (eicosanoic) acid C20:0	0.23
Unsaturated fatty acid	Linoleic acid (n-6) C18:2	60.58
	Linolenic acid (n-3) C18:3	0.10
	Palmitoleic acid C16:1	0.10
	Oleic acid (n-9) C18:1	26.74
	cis-5 Eicosenoic acid C20:1	0.14

The dry matter, protein, fat, ash, digestible carbohydrates, dietary fiber, calorific value, and pH of the tested samples are presented in Table 2. The dry matter content showed no statistically significant differences depending on the variable proportion of oleogel in ice cream and ultrasound treatment and ranged from 36.48 to 36.86%. The dry matter content of typical ice cream should be between 28 and 40%, with 7–15% fat content [1]. With an increase in the dry matter content, the diameter of the ice crystals decreases, which is very desirable and improves the consistency of ice cream. In addition, the dry matter content affects the overrun [30]. With an increase in the proportion of oleogel, the protein content increased significantly, which ranged from $12.31 \text{ g} \times (100 \text{ g})^{-1}$ for sample UP7 to $13.80 \text{ g} \times (100 \text{ g})^{-1}$ for sample PP5. Ultrasound pasteurization did not statistically significantly affect the protein content in the ice cream samples.

Table 2. Chemical properties and energy value of the tested ice cream.

Properties	PP5	PP6	PP7	UP5	UP6	UP7
Total solid [%]	36.48 ^a ± 0.11	36.69 ^a ± 0.16	36.50 ^a ± 0.38	36.49 ^a ± 0.28	36.86 ^a ± 0.27	36.53 ^a ± 0.18
pH	6.43 ^a ± 0.01	6.44 ^a ± 0.01	6.46 ^a ± 0.01	6.37 ^b ± 0.01	6.43 ^a ± 0.01	6.45 ^a ± 0.01
Protein [g·(100 g) ⁻¹]	13.80 ^c ± 0.05	12.76 ^b ± 0.08	12.34 ^a ± 0.05	13.77 ^c ± 0.09	12.80 ^b ± 0.07	12.31 ^a ± 0.06
Fat [g·(100 g) ⁻¹]	11.25 ^c ± 0.08	12.65 ^a ± 0.08	13.80 ^e ± 0.07	10.15 ^b ± 0.09	11.93 ^d ± 0.07	12.55 ^a ± 0.05
Ash [g·(100 g) ⁻¹]	3.06 ^c ± 0.03	2.83 ^b ± 0.01	2.70 ^a ± 0.02	3.01 ^c ± 0.02	2.84 ^b ± 0.04	2.70 ^a ± 0.01
Carbohydrate [g·(100 g) ⁻¹]	34.57 ^a ± 0.03	35.51 ^b ± 0.03	40.88 ^c ± 0.04	57.07 ^f ± 0.07	49.78 ^e ± 0.04	45.86 ^d ± 0.05
Dietary Fibre [g·(100 g) ⁻¹]	21.97 ^f ± 0.05	18.62 ^e ± 0.04	13.17 ^a ± 0.04	15.47 ^b ± 0.05	17.74 ^c ± 0.02	18.35 ^d ± 0.04
Caloric value [kcal·(100 g) ⁻¹]	339 ^a ± 0.40	344 ^b ± 0.27	363 ^c ± 0.57	406 ^f ± 0.79	393 ^e ± 0.51	382 ^d ± 0.35

^{a–f} Means in the same line indicated by different letters were significantly different (*p* value < 0.05). The results are expressed as mean ± SD (*n* = 3).

The fat content differed significantly for both the samples with a variable proportion of oleogels and the samples subjected to the ultrasound treatment. It ranged from $10.15 \text{ g} \times (100 \text{ g})^{-1}$ (UP5) to $13.80 \text{ g} \times (100 \text{ g})^{-1}$ (PP7). The ultrasonically treated ice cream samples showed a lower fat content compared with the traditionally pasteurized samples. Fundamental changes in the components of milk (protein and fat) may be attributable to changes in fat globule size, which increase the surface membrane area of the fat globules, the formation of bonds between casein and the fat globules present in the gel network,

denaturation of casein micelles, and the formation of aggregates between k-casein and whey proteins (β -lactoglobulin) [31–33]. Statistically significant differences in the digestible carbohydrate content were observed in the samples. The carbohydrate content ranged from 20.51 to 57.07 $\text{g} \times (100 \text{ g})^{-1}$. The ultrasound-treated ice cream showed a higher content of assimilated carbohydrates. Based on the chemical composition analysis of the tested samples, statistically significant differences in the fiber content were found. Sample PP5 (with 5% oleogel and subjected to traditional pasteurization) showed the highest amount of dietary fiber. Its ash content ranged from 2.70 to 3.06 $\text{g} \times (100 \text{ g})^{-1}$. The higher the proportion of oleogel in the ice cream, the higher the ash content. The highest ash content was observed in sample PP5. The calorific value of ice cream depends on the amount and type of ingredients (sucrose, fat, protein, and other ingredients). Among the tested samples, sample UP5 showed the highest calorific value, whereas sample PP6 showed the lowest. The pH value ranged from 6.37 to 6.46. Neither the variable proportion of oleogel nor the ultrasound as an alternative to traditional pasteurization had a statistically significant influence on the pH change in all samples, except for sample UP5. The pH range was similar to those reported [34,35].

The thermal and physical properties of the ice cream samples after the addition of tomato seed oleogel are shown in Table 3. Neither the variable proportion of oleogel nor the ultrasound treatment of the ice cream mixtures had a statistically significant effect on the freezing point of the samples. The freezing point of the samples ranged from -4.70 to -5.23 °C. It is determined using the concentration and the type of solutes present in the mixture. The presence of dissolved salts and sugars decreases the freezing point of water as the solute molecules interact with the water molecules and inhibit their ability to come together and form an ice crystal lattice (or freeze). The freezing point of the ice cream mixture is an important quality control parameter since it determines the amount of ice that can be formed at a given temperature which, in turn, affects the quality and textural attributes of ice cream. With an increase in the freezing point, the melting rate increases, and firmness decreases (as indicated by osmolality). As the freezing point of the mixture is decreased (osmolality increases), the ice cream contains less ice and more unfrozen water at any given temperature, which results in ice cream that is less firm and melts at a faster rate [36–38]. Significant differences in the amount of freezable water were observed for the ice cream samples with the addition of 5% (PP5) and 6% (PP6) oleogel and subjected to traditional pasteurization, which amounted to 34.70 and 39.15%, respectively. In the samples subjected to traditional pasteurization, the enthalpy values associated with the ice melting transition increased with the percentage of oleogel in ice cream, whereas the samples subjected to ultrasound pasteurization showed the opposite relationship.

Table 3. Thermal and physical properties of the tested samples.

Properties	PP5	PP6	PP7	UP5	UP6	UP7
Freezing point [°C]	$-5.23^a \pm 0.22$	$-5.02^a \pm 0.15$	$-4.82^a \pm 0.13$	$-5.16^a \pm 0.14$	$-4.74^a \pm 0.37$	$-4.70^a \pm 0.24$
Freezable water [%]	$34.70^b \pm 0.74$	$39.15^c \pm 0.38$	$41.81^a \pm 0.36$	$42.19^a \pm 0.79$	$41.93^a \pm 0.47$	$41.60^a \pm 0.47$
Enthalpy of fusion [$\text{J} \cdot \text{g}^{-1}$]	$115.91^b \pm 2.46$	$130.77^c \pm 1.28$	$139.64^a \pm 1.21$	$140.93^a \pm 2.65$	$140.05^a \pm 1.56$	$138.95^a \pm 1.58$
Overrun [%]	$44.62^d \pm 0.25$	$48.32^f \pm 0.33$	$46.75^e \pm 0.20$	$31.29^b \pm 0.11$	$29.52^a \pm 0.05$	$35.17^c \pm 0.11$
First drop time [min]	$9.51^b \pm 0.06$	$9.45^b \pm 0.08$	$8.53^a \pm 0.01$	$8.46^a \pm 0.05$	$8.30^d \pm 0.04$	$8.08^c \pm 0.05$
Complete melting time [min]	$29.34^a \pm 0.05$	$30.01^d \pm 0.08$	$31.36^e \pm 0.02$	$27.56^b \pm 0.05$	$28.50^c \pm 0.04$	$29.39^a \pm 0.04$
Hardness [N]	$3.88^{ab} \pm 1.23$	$3.58^{ab} \pm 0.42$	$1.59^b \pm 0.09$	$9.33^c \pm 1.22$	$6.30^a \pm 1.79$	$4.57^a \pm 0.52$
Adhesiveness [$\text{N} \cdot \text{s}$]	$-6.45^{ab} \pm 1.54$	$-5.77^a \pm 0.74$	$-3.05^a \pm 0.13$	$-19.66^c \pm 2.20$	$-10.38^b \pm 2.44$	$-6.60^{ab} \pm 0.67$
Viscosity [$\text{mPa} \cdot \text{s}$]	$371.67^f \pm 0.58$	$365.67^e \pm 1.53$	$326.97^d \pm 0.95$	$287.63^c \pm 1.10$	$257.63^b \pm 1.48$	$218.97^a \pm 0.06$

^{a–f} Means in the same line indicated by different letters were significantly different (p value < 0.05). The results are expressed as mean \pm SD ($n = 3$).

The overrun of ice cream affects its smoothness, texture, and taste [37]. Significant differences in the aeration (overrun) of ice cream were found between the ultrasound-pasteurized and the traditionally pasteurized samples. The highest overrun (48.32%) was observed in the traditionally pasteurized sample with 6% oleogel (PP6), whereas the lowest overrun (29.52%) was observed in the ultrasound-pasteurized sample with 6% oleogel

(UP6). The aeration capacity of ice cream depends on its viscosity. It has been reported that ice cream with a lower viscosity shows a lower overrun [39]. Statistically significant differences in the first drop time and the complete melting time were observed between the samples. The traditionally pasteurized sample with 7% oleogel (PP7) melted the slowest (the time to melt completely was 31 min). The ultrasound-pasteurized samples showed significantly higher hardness and adhesiveness than the traditionally pasteurized samples. The highest values for these two parameters were observed in sample UP5 (9.33 N and -19.66 Ns), whereas the lowest values were observed in sample PP7 (1.59 N and -3.05 Ns). The highest viscosity was observed in sample PP5 (371.67 mPa·s), whereas the lowest viscosity was observed in sample UP7 (218.97 mPa·s).

Then, the ice cream samples were subjected to Fourier transform infrared spectroscopy analysis. For the clarity of the presentation and to facilitate the analysis of the bands in the spectra, the spectra of the tested samples are presented in Figure 2 for the spectral range of $3600\text{--}500\text{ cm}^{-1}$. The vibrations of the functional groups are assigned to the appropriate bands in Table 4, based on a detailed review of the literature.

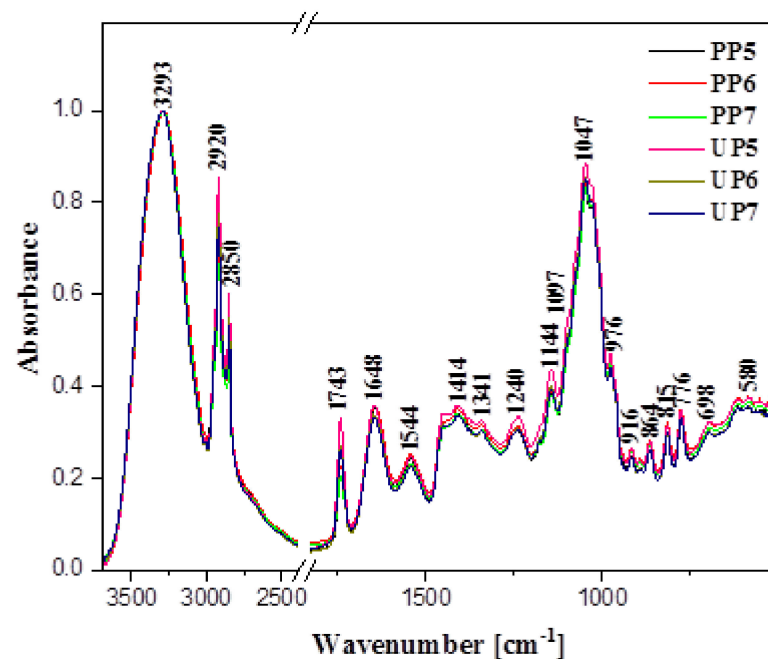


Figure 2. Fourier transform infrared FTIR spectra of ice cream samples subjected to UP ultrasound pasteurization and traditional PP pasteurization, presented in the spectral range from $3600\text{ to }550\text{ cm}^{-1}$ normalized at 3293 cm^{-1} .

According to the previous studies [43–45], the spectral region from $3700\text{ to }3000\text{ cm}^{-1}$ for all samples, with a characteristic maximum of about 3300 cm^{-1} , shows bands characteristic of the stretching vibrations of the --OH group. For this type of food sample, this band is primarily attributable to the vibrations of the --OH groups in fats, carbohydrates, and, especially, water molecules. The region in the range from $3000\text{ to }2800\text{ cm}^{-1}$ is another important area for food products rich in fats and carbohydrates, in which there are stretching vibrations of C--H , alkyl, and aromatic groups resulting from the vibrations of these groups in hydrocarbon molecules. These vibrations are attributable to both symmetric and asymmetric functional groups --CH_2 . Vibrations with a maximum of $\sim 3300\text{ cm}^{-1}$ attributable to the --OH groups, due to their high intensity, significantly underpin the C--H stretching vibrations in the --CH_2 groups.

Table 4. The location of the maxima of absorption bands FTIR with arrangement of appropriate vibration for selected for sampling: PP5, PP6 and PP7 and UP5, UP6 and UP7 [40–47].

FTIR						Type and Origin of Vibrations
Positioning of Band [cm^{-1}]						
PP5	PP6	PP7	UP5	UP6	UP7	
3285	3285	3289	3293	3293	3293	ν_{st} (O-H) in H_2O
2953	2953	2952	2953	2951	2951	$\nu_{\text{s+as}}$ (C-H) in CH_2 and CH_3 groups in both carbohydrates and fatty acids
2918	2918	2918	2920	2920	2920	
2870	2870	2872	2869	2870	2870	
2849	2850	2850	2850	2850	2850	
1742	1742	1742	1743	1743	1743	ν (C=O)
1728	1728	1728	1727	1727	1727	ν (C=O) \cdots OH
1649	1649	1649	1648	1649	1648	δ_{vw} (-OH) and ν_{vw} (-C=C-)
1544	1544	1544	1544	1544	1544	ν_{vw} (-C=C-)
1452	1452	1452	1452	1452	1452	δ (-O-CH) and δ (-C-C-H)
1414	1414	1414	1414	1414	1414	δ_{st} (O-H) in C-OH group + δ (C-H)
1376	1376	1376	1376	1376	1376	δ (-OH) in C-OH group and ν (-C-H, - CH_3) and deformation
1340	1340	1340	1341	1341	1341	
1241	1241	1241	1238	1241	1238	ν_{m} (-C-O) or δ_{m} (- CH_2 -) ν (C-H) in carbohydrates
1144	1144	1144	1144	1144	1144	ν (C-H) in carbohydrates and ν (C-O) in C-O-C group and ν_{st} (C-C) in the carbohydrate structure
1097	1097	1097	1097	1097	1097	
1047	1047	1047	1048	1048	1048	
1028	1028	1028	1030	1030	1030	
976	976	976	976	976	976	ν_{st} (C-C) in the carbohydrate structure, δ (C-H)
963	963	963	964	964	964	
916	916	916	916	916	916	
892	892	892	892	892	892	ν (C-C) in the carbohydrate structure, δ (C-H)
864	864	864	864	864	864	
815	815	815	815	815	815	
776	776	776	776	776	776	
698	698	698	698	698	698	

ν —stretching vibrations, δ —deformation vibrations, s—symmetric, as—asymmetric, st—strong.

A fairly wide band of vibrations attributable to the -OH groups is a result of the formation of strong hydrogen bonds, which—in the case of the tested samples—are primarily found in carboxylic acid dimers. The band that is attributable to the same groups but occurs in a completely different area, i.e., with a maximum of $\sim 1650 \text{ cm}^{-1}$ (Figure 1), is the deformation vibrations of the -OH groups, the origin of which is analogous to those described above. In such samples, this band can also be built up by the asymmetric vibrations stretching COO- groups. Another important area of vibrations with a maximum of about 1743 cm^{-1} is attributable to the stretching vibrations of the functional ketone groups C=O of fructose, one of the components of the tested samples. It is highly reliable in the analysis of the samples because its intensity strongly depends on whether traditional pasteurization or ultrasound pasteurization is used. The band with a maximum of 1743 cm^{-1} is clearly underpinned from the short-wave side by a maximum of $\sim 1720 \text{ cm}^{-1}$. This substructure may result from the hydrogen bond formation between carbonyl groups and -OH groups,

especially after the application of the appropriate treatment. However, a particular characteristic and interesting area is the fingerprint region, primarily from 1500 and 700 cm^{-1} . This region is quite rich in bands that give good information about changes in the samples as a result of the application of the appropriate treatment, such as ultrasound pasteurization or traditional pasteurization. The most important and authoritative vibrations from this area are the stretching vibrations of the C–O, C–C, C=C, and C–H groups and the bending vibrations of the C–H group present in the chemical structure of carbohydrates. These vibrations are also characteristic of the fat contents in these samples, which contribute to a large part of the mass of the samples. The most intense and interesting vibrations from this area are primarily the bands with a maximum of 1544, 1450, 1414, 1341, and 1240 cm^{-1} . These vibrations are primarily attributable to the deformation vibrations of the O–CH and C–C–H groups in the carbohydrate structure, as presented in Table 4. A band with a maximum of 1414 cm^{-1} is also a characteristic of the symmetric vibrations of the COO– group. The areas of these vibrations can be enhanced by the deformation vibrations of the –OH groups belonging to the C–OH junction. A highly significant area of the bands extends from 1230 to about 950 cm^{-1} . The most intense vibrations in the samples are primarily attributable to the C–H C–O groups in the carbohydrate structure. The bands with a maximum of $\sim 1144 \text{ cm}^{-1}$ and most of all with a maximum of 1047 cm^{-1} are attributable to the vibrations of the C–O groups in the characteristic C–O–C connection. They are amplified by the vibrations from fat fractions. The area for this type of sample from about 1050 to 950 cm^{-1} is also the C–O in C–OH group or COO– and C–C stretching vibrations in the carbohydrate structure. The area below 950 cm^{-1} is the vibration characteristic of the anomeric region of carbohydrates or deformation vibrations of the C–H and C–C groups [40–45]. It is worth noting that slight changes in the vibrations from this region of the bands often indicate modifications in the bonds of the carbohydrate fractions. In the tested samples, this region, apart from the change in the intensity, does not differ much for individual spectra. Based on the analysis performed using FTIR, the subtle differences in the spectra are worth emphasizing, which, however, quite strongly confirm the results of previous studies. The differences, primarily in the intensity between the spectra in several areas, seem to be particularly visible. These differences are visible in the region of C–H vibrations in the –CH₂ groups and the carbonyl group C=O, and in the region of C–C, C–H, and C–O vibrations in the area of the fingerprint. Changes in these areas correlate with changes in the treatment used, i.e., traditional pasteurization or ultrasound pasteurization. These factors resulted in a marked decrease in the intensity of vibrations in the regions characteristic of the fat contents and a slight increase in the intensity of these vibrations, which are characteristic of the carbohydrate content. These changes seem to be closely related to the processing factors used in ice cream production. A much higher intensity of these changes can be observed in the spectra after ultrasound pasteurization.

Ice cream has a very complex structure, with many phases affecting its quality, texture, and physical properties, including shape retention and structure collapse during melting. The ingredients are water, fat, fat-free milk dry matter (casein micelles, whey proteins, lactose, and milk salts), sugars (sucrose and partially hydrolyzed starch, including glucose, maltose, and higher saccharides), stabilizers, and emulsifiers, and air is added prior to dynamic freezing. All these elements contribute to the formation of the structural elements of the ice. Fat remains in the form of spherical, emulsified droplets or is converted into a partially crystalline structure. Water is transformed into ice crystals. Air is churned into small bubbles, and sugars and stabilizers are frozen in the nonfrozen phase of the serum [1].

Identification of microstructures of food is important for dimensional regulation of identifiable components that constitute the food. Especially in products such as ice cream, from a technological point of view, the microstructure is important to yield products with the desired textural and sensory properties. Figure 3 shows the microstructure of ice cream subjected to two pasteurization methods: traditional and ultrasound. Larger voids are observed in the images of samples PP5, PP6, and PP7, which were subjected to traditional pasteurization, indicating a higher overrun compared with the samples

subjected to ultrasound pasteurization. A compact structure is observed in the images of samples UP5, UP6, and UP7, which were subjected to ultrasound pasteurization, indicating a lower overrun. The ultrasound treatment is found to promote a reduction in the size of fat globules due to cavitation onto nanoparticles. In addition, finer spaces formed by the formation of the fine crystalline structure of the ice can be observed. The skimmed milk to the ultrasound treatment at 20 kHz for up to 60 min reduced the size of fat globules to about 10 nm [48]. Bermúdez-Aguirre et al. [49] studied the effect of the thermosonic treatment (24 kHz, 400 W, 120 μm amplitude at 63 °C for 30 min) on the microstructure of fat globules in whole milk and found that ultrasound reduced the size of milk fat globules by up to $<1\ \mu\text{m}$, promoting the fusion of casein and serum proteins.

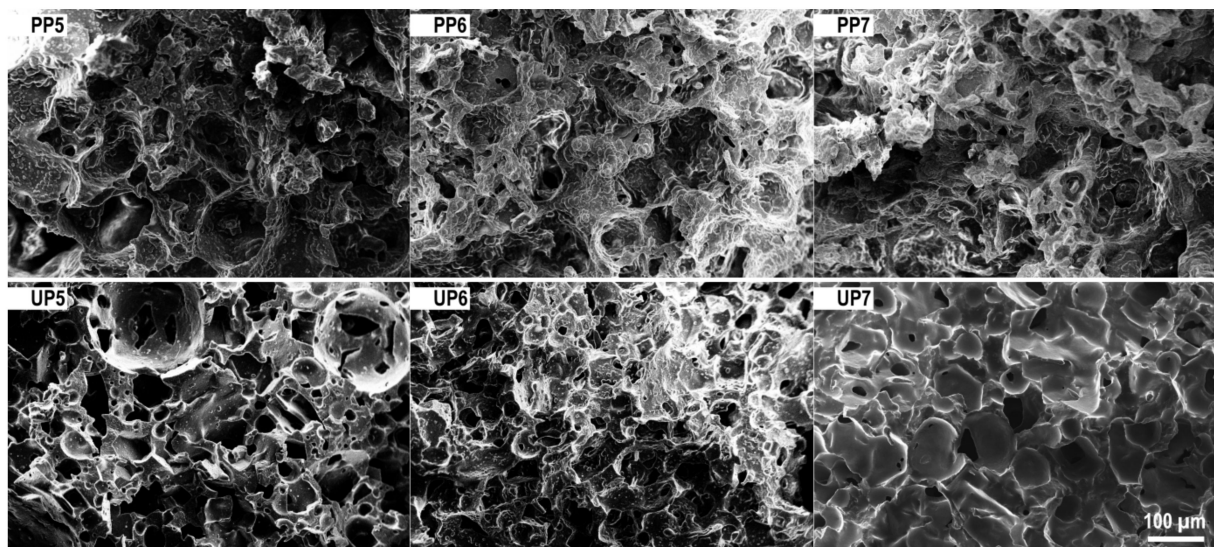


Figure 3. Ice cream structure by scanning electron microscopy. Ice cream samples were pictured with a secondary electron detector at 3 kV on a 100 μm scale. PP5—sample with 5% tomato seed oleogel, traditionally pasteurized; PP6—sample with 6% tomato seed oleogel subjected to traditional pasteurization; PP7—sample with 7% tomato seed oleogel subjected to traditional pasteurization; UP5—sample with 5% tomato seed oleogel subjected to ultrasound pasteurization; UP6—a sample with 6% tomato seed oleogel subjected to ultrasound pasteurization; UP7—sample with 7% tomato seed oleogel subjected to ultrasound pasteurization.

The ultrasound treatment reduced the size of the ice crystals while whipping the emulsion, resulting in a faster formation of the three-dimensional lattice structure of the cream [50]. The application of ultrasound in ice cream production is useful in crystallization processes because it can control crystal growth. In addition, since ice cream contains air up to 50% of its volume, ultrasound can be an effective degasser that can change the texture of ice cream [51].

The thermal treatment used in the ultrasound application results in less organoleptic and nutritional changes in the products compared with the traditional heat treatment, and the reduction in thermal energy consumption lowers the cost of the product [52]. However, long duration, high temperature, and inadequate ultrasound amplitude may lead to a reduction in the physicochemical and textural properties of ice cream; therefore, further research on the selection of appropriate conditions for ultrasound pasteurization of ice cream mixtures (time, temperature, and ultrasound amplitude) is needed to improve the quality of ice cream.

5. Conclusions

This study presented the possibility of using tomato seed oil, which has very good nutritional properties due to the high unsaturated fatty acid content, in ice cream production.

Tomato seed oil forms stable oleogels that determine the properties of ice cream. As the proportion of oleogels increased, the fat and carbohydrate contents, the amount of freezable water, and the enthalpy of fusion, as well as the overrun of the ice cream samples, increased, whereas the viscosity and hardness decreased. Oleogels made with tomato seed oil are a promising alternative to fat in ice cream rich in unsaturated fatty acids. This study proved that ultrasound pasteurization can be used as an alternative to traditional pasteurization with no undesirable changes in physicochemical properties. It also showed that ultrasound pasteurization reduced the fat and carbohydrate contents and thus the energy value of ice cream, as confirmed by FTIR analysis. Furthermore, ultrasound pasteurization increased the amount of freezable water in the ice cream samples and decreased the viscosity and the overrun, thus increasing the hardness. Microstructure analysis showed that ultrasound promotes even distribution of all components of the ice-cream mixture with oleogels, while contributing to its pasteurization. As a result, the size of the fatty globules is reduced, which gives an effect comparable to homogenization. The use of ultrasound pasteurization of ice cream can have benefits as it allows the process to be run at a lower temperature than traditional pasteurization, and the effect is similar. However, research should be continued to elucidate the interaction of time and temperature and the amplitude of ultrasound on the properties of ice cream with oleogels.

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