

Article

The Effectiveness of Combination Stabilizers and Ultrasound Homogenization in Milk Ice Cream Production

Anna Kot , Ewa Jakubczyk  and Anna Kamińska-Dwórznička *

Department of Food Engineering and Process Management, Institute of Food Sciences, Warsaw University of Life Sciences (WULS-SGGW), Nowoursynowska 159C, 02-776 Warsaw, Poland; ewa_jakubczyk@sggw.edu.pl

* Correspondence: anna_kot1@sggw.edu.pl (A.K.); anna_kaminska1@sggw.edu.pl (A.K.-D.)

Abstract: This study aims to contribute knowledge to the area of the ice cream industry by finding an effective way to prevent the recrystallization process in ice cream production. Stabilizers such as ι -carrageenan and its acid and enzymatic hydrolyzates were used with the combination of ultrasound homogenization (20 kHz and exposure time of 5 min) as a method to obtain the deliberate quality of ice cream. In this paper, a comprehensive analysis of the physical characteristic of milk ice creams was made, such as the cryoscopic temperature, osmotic pressure, overrun, and melting time. It was noted that cryoscopic temperature was lower in the samples after ultrasound treatment. Additionally, the osmotic pressure was changed in the case of the stabilizer used. The overrun of ice cream was less than 32% while the longest melting time was at the level of 27 min. The recrystallization process was analysed on the basis of images taken after 24 h, and 1 and 3 months of storage at -18°C . Regarding the results, it was observed that ultrasound homogenization contributed to smaller ice crystals and had a positive influence on the ice crystals' structure.

Keywords: recrystallization; ice crystals; ultrasound; freezing; physical properties



Citation: Kot, A.; Jakubczyk, E.; Kamińska-Dwórznička, A. The Effectiveness of Combination Stabilizers and Ultrasound Homogenization in Milk Ice Cream Production. *Appl. Sci.* **2023**, *13*, 7561. <https://doi.org/10.3390/app13137561>

Academic Editors: Agnieszka Najda, Joanna Maria Klepacka and Marta Czarnowska-Kujawska

Received: 19 May 2023

Revised: 17 June 2023

Accepted: 23 June 2023

Published: 27 June 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Ice cream is defined as a complex colloidal system consisting of air bubbles, ice crystals, and fat droplets dispersed into the serum phase. Considered the category of dairy products, ice cream is one a palatable frozen dessert. Obtaining the desirable texture to meet the expectations of consumers is a requisite for manufacturers [1–4]. Perception of ice cream texture is connected with its creaminess. One of the factors attributed to this property is the crystal structure, especially the number of crystals and their size. Such a factor is inevitably connected with the components of ice cream and the production process [2,5]. The advisable size of ice crystals is between 10 to 20 μm , which guarantees achieving a favourable texture. Because larger crystals of more than 50 μm cause undeniable quality such as coarse or grainy [6–9], it is thus significant to understand the factors that affect it and how they are regulated.

Stabilizers have the ability to modify the water-binding capacity, freezing rates, rheological properties, or ice crystal formation. Polysaccharide stabilizers such as carrageenans, locust bean gum, or xanthan gum are usually used in the formulation of the ice crystal structure [10]. Carrageenan is a commonly used secondary stabilizer and, additionally, it helps prevent the process of wheying off. The fractions of carrageenan such as ι -carrageenan are able to react electrostatically with milk proteins and form a three-dimensional network. Such a structure contributes to the resistance separation of the suspended phase in ice cream mixes [10–12]. Moreover, interesting results were obtained using acid and hydrolyzates of ι -carrageenan in the model sucrose solutions with milk protein. It was proven that hydrolyzates more effectively inhibited the recrystallization process in the ice crystal structure than ι -carrageenan [13].

Ultrasound has been known in the food industry for many years. The mechanism of ultrasound is based on acoustic cavitation, which occurs due to the interaction between

ultrasonic waves, liquid, and dissolved gas. Considering the advantages of this technique, ultrasound is relatively cheap, simple, really fast, non-toxic, environmentally friendly, and also energy saving. Additionally, ultrasound can be used to minimize processing or increase quality and improve processing effectiveness and efficiency, as well as to provide food safety, while extending the shelf life of the product [14–16]. Currently, there is much research on which ultrasound can be used in ice cream production. For instance, during the freezing process, ultrasound may enhance the nucleation rate and the rate of crystal growth, which contribute to decreasing the ice crystal size and freezing time [15,17–19]. On the other hand, the influence of used ultrasound homogenization in milk ice cream and the changes during the creation of the ice crystal structure still require additional research. Moreover, it has been proven that using ultrasound during the pasteurization process can be used interchangeably with traditional pasteurization, with no undesirable changes in ice cream [20]. Furthermore, the study by Tüker and Dogan [21] shows that ultrasound homogenization improved the properties of ice cream such as the melting time. Ultrasound homogenization may generate positive results such as narrowing the particle size, which generates a more stable emulsion in ice cream. Based on this, considering the amount of fat and its structure in ice cream mixes might bring a beneficial effect on the ice crystal size, owing to the fact less space will be created for crystal formation and smaller ice crystals will be obtained [10]. Therefore, ultrasound may be a promising tool for inhibiting the recrystallization phenomena in ice cream, obtaining smaller crystals and improving the quality of the product.

Much of the available literature deals with the problem of recrystallization in ice cream; therefore, the results presented in this paper could be valuable in this field. The following two main points were studied: (1) the effect of using hydrocolloids namely ι -carrageenan and its acid and enzymatic hydrolyzates on the physical properties of milk ice cream, and (2) the effectiveness of ultrasound homogenization in the production of milk ice cream.

2. Materials and Methods

2.1. The Preparation of the Hydrolyzates of ι -Carrageenan

The materials and methods for the hydrolysis of the ι -carrageenan part were described in the paper by Kot et al. [13]. Briefly, ι -carrageenan (obtained from Sigma-Aldrich, St. Louis, MO, USA) was dissolved in distilled water heated up to 40 °C to obtain a 0.4 mg/mL solution. The enzymatic hydrolysis was carried out using β -galactosidase (1000 U/mg, from *Escherichia coli*) (Sigma-Aldrich, St. Louis, MO, USA) for 72 h, at 37 °C or using lactase (min. activity 5200 NLU/g) (Serowar s.c., Szczecin, Poland) for 24 h, at 5 °C. For both hydrolyses, the reaction was stopped by neutralization at 48 °C for 5 min. The acid hydrolysis of ι -carrageenan was performed by dissolving in 0.1 M hydrochloric acid (Chempur, Piekary Śląskie, Poland) solution (pH 3). Then, the solution was heated at 60 °C for 3 h and neutralized using 0.1 M sodium hydroxide (Chempur, Piekary Śląskie, Polska). All of the obtained hydrolyzates were stored frozen at −18° and thawed just before analysis. The molecular mass distribution was estimated through Size-exclusion chromatography analysis (SEC) using the Shimadzu high-performance liquid chromatography system consisting of a RID-10A detector (Shimadzu, Kyoto, Japan), and only hydrolyzates with the highest reduction in molecular mass were used for further analysis.

Moreover, to confirm that the IRI (ice recrystallization inhibition) activity of the obtained hydrolyzates of the ι -carrageenan depends on the functional group's position changes, Fourier transform infrared spectroscopy (FTIR) analysis was performed using a HATR Ge trough (45° cut, yielding 10 internal reflections) crystal plate at 20 °C, and recorded with a 670-IR spectrometer (Agilent, Santa Clara, CA, USA). Based on these results, only the samples with the highest molecular mass reduction and the longest time for hydrolysis were used for further research as a stabilizer in ice cream.

2.2. The Process Production of Ice Cream

The Materials for the Recipe for Ice Cream

The ingredients used to prepare the ice cream mixes were milk 0.5% (Mlekovita, Wysokie Mazowieckie, Poland), inulin (Orafti BENEIO, Tienen, Belgium), skimmed milk in powder (Mlekovita, Wysokie Mazowieckie, Poland), white sugar (Diamant, Poznań, Poland), emulsifier E471 (Fooding Shanghai, Shanghai, China), LBG (Locust Bean Gum) (Fooding Shanghai, Shanghai, China), 0.02% xanthan gum (Fooding Shanghai, Shanghai, China), 0.01% ι -carrageenan (Fluka, Sigma-Aldrich, St. Louis, MO, USA), or 0.005% newly obtained acid hydrolyzates of ι -carrageenan and enzymatic hydrolysis by β -galactosidase and enzymatic hydrolysis by commercial lactase.

Based on the research by [22], an amount of ι -carrageenan of 0.01% is the minimal concentration that could influence the recrystallization process. While the amount of obtained hydrolyzates at 0.005% was connected with the fact that hydrolyzates of ι -carrageenan had the strongest effect compared with the ι -carrageenan. The percentage amount of each ingredient is presented in Table 1.

Table 1. The percentage composition of ice cream milk samples.

Ingredient	C, CH, CU	I, IH, IU	A, AH, AU	B, BH, BU	L, LH, LU
Milk 0.5	76.0	75.49	75.495	75.495	75.495
Inulin	10.0	10.0	10.0	10.0	10.0
Milk powder	7.0	7.0	7.0	7.0	7.0
White sugar	7.0	7.0	7.0	7.0	7.0
Emulsifier E471	0.4	0.4	0.4	0.4	0.4
Locust bean gum	-	0.08	0.08	0.08	0.08
Xanthan gum	-	0.02	0.02	0.02	0.02
ι -Carrageenan	-	0.01	-	-	-
Acid hydrolysate of ι -carrageenan	-	-	0.005	-	-
Enzymatic hydrolyzate of ι -carrageenan obtained by β -galactosidase treatment	-	-	-	0.005	-
Enzymatic hydrolyzate of ι -carrageenan obtained by lactase treatment	-	-	-	-	0.005

The characteristics of the prepared samples of the ice cream mixes are presented in Table 2.

Table 2. The characteristic of the ice cream milk samples.

Sample	Stabilizers	Homogenization Treatment
C	Control sample without stabilizers	-
CH	Control sample without stabilizers	traditional homogenization treatment
CU	Control sample without stabilizers	ultrasound homogenization treatment
I	Sample with the combination of ι -carrageenan, LBG and xanthan gum	-
IH	Sample with the combination of ι -carrageenan, LBG and xanthan gum	traditional homogenization treatment
IU	Sample with the combination of ι -carrageenan, LBG and xanthan gum	ultrasound homogenization treatment

Table 2. Cont.

Sample	Stabilizers	Homogenization Treatment
A	Sample with the combination of acid hydrolyzates of ι -carrageenan, LBG and xanthan gum	-
AH	Sample with the combination of acid hydrolyzates of ι -carrageenan, LBG and xanthan gum	traditional homogenization treatment
AU	Sample with the combination of acid hydrolyzates of ι -carrageenan, LBG and xanthan gum	ultrasound homogenization treatment
B	Sample with the combination of enzymatic β -galactosidase hydrolyzates of ι -carrageenan, LBG and xanthan gum	-
BH	Sample with the combination of enzymatic β -galactosidase hydrolyzates of ι -carrageenan, LBG and xanthan gum	traditional homogenization treatment
BU	Sample with the combination of enzymatic β -galactosidase hydrolyzates of ι -carrageenan, LBG and xanthan gum	ultrasound homogenization treatment
L	Sample with the combination of enzymatic commercial lactase hydrolyzates of ι -carrageenan, LBG and xanthan gum	-
LH	Sample with the combination of enzymatic commercial lactase hydrolyzates of ι -carrageenan, LBG and xanthan gum	traditional homogenization treatment
LU	Sample with the combination of enzymatic commercial lactase hydrolyzates of ι -carrageenan, LBG and xanthan gum	ultrasound homogenization treatment

2.3. The Production of Ice Cream

According to the recipe, the dry and liquid ingredients were weighed separately. After this, all components were mixed using a Bosch MaxoMixx 750W blender (Bosch, Gerlingen, Germany). Then, the pasteurization process was performed using a Vorwerk thermomixer (Vorwerk, Wuppertal, Germany) at a temperature of 85 °C within 1.5 min and then cooled to 25 °C. Two methods of homogenization were used:

- The traditional homogenization using the homogenizer IKA T 25 digital ULTRA-TURRAX 20 rpm (IKA[®]-Werke GmbH & Co. KG, Staufen, Germany) through 2.5 min.
- The ultrasound homogenization by using a homogenizer Ultrasonic Liquid Processor VCX 500 (Sonics & Materials, Inc., Newtown, CT, USA) with a diameter probe (Model CV334). 250 mL of ice cream mixes for each trial. The frequency of 20 kHz and exposure time of 5 min was used. The used frequency of ultrasound was also tested in accordance with other papers such as the paper by O'Sullivan et al. [23] for the ultrasound homogenization on soy and wheat protein isolates and the paper by de Silva et al. [24] during ultrasound homogenization of cupuaçu juice.

After the homogenization step, all of the prepared ice cream mixes were submitted to the maturation process for 24 h at 4 °C (fridge, Whirlpool, Warszawa, Poland).

2.4. The Freezing of Ice Cream

The freezing of ice cream mixes was performed in an ice cream maker, Neumaker Gelato 5K SC (Hemer, Germany), until the ice cream temperature was −7 °C for 15 min. Then, the samples were placed in plastic containers and stored at −18 °C for 24 h, 1 month, and 3 months (freezer, Whirlpool, Warszawa, Poland).

2.5. The Ice Cream's Physical Analysis

2.5.1. Cryoscopic Temperature and Osmolality

The cryoscopic temperature and the osmolality of the ice cream mixes were determined using an osmometer Marcel os3000 (Warszawa, Poland). The accuracy of measurement of the freezing temperature was 0.002 °C and for osmolality it was 1%. According to the instruction of the devices, 100 µL of ice cream mixes after the maturation process were taken into Eppendorf tubes and measured until the device was stabilized. The analysis was performed in duplicate.

2.5.2. Melting Time

The melting behaviour of the ice cream was determined using a cooled metal ring (11 cm in height and 2 cm in diameter, volume 35 mL) that was stored at −25 °C for 24 h before measurement. After this time, the ring was filled with ice cream directly after the freezing process and then stored for 24 h at −25 °C. After storage, the ring was placed on the funnel with two pins located at the ends of the ring at the controlled temperature of 25 °C. The first drop of melted ice cream was recorded as the melting time of the sample [25,26]. The analysis was performed in duplicate.

2.5.3. Determination of the Overrun

The overrun of ice cream was determined according to the following Formula (1) [25,26]:

$$\text{Overrun} = \frac{W_1 - W_2}{W_2} \times 100\% \quad (1)$$

where: W_1 is the mass of the unit volume of the mixture (g) and W_2 is the mass of the unit volume of ice cream (g).

2.5.4. Microstructural Analysis of Ice Crystals

The microstructure of the ice crystals was analysed based on the images taken after 24 h, 1 month, and 3 months of storage at −18 °C. To prepare the samples, a small amount of ice cream was taken from the centre of the plastic box (from at least three different locations, and a minimum of 3 cm away from the surface), then put on the cool slide using a spatula and covered with a cool slip glass on the top of the sample. All of the samples were prepared in a freezing chamber and transferred to a microscope with the cooling system Linkam Scientific PE 94.

The recrystallization process was analysed based on images taken using the Olympus model BX43F (Tokyo, Japan) microscope with the cooling system with liquid nitrogen-Linkam Scientific Instruments LTD model LNP96-S (Tokyo, Japan) and the Olympus model SC50 camera (Tokyo, Japan). The obtained images were analysed using the Olympus cellSens Dimension Desktop program. The around 300 ice crystals were marked for one sample, and then the area, equivalent diameter, and standard deviation were calculated.

2.5.5. The Statistical Analysis

For the melting time, overrun, cryoscopic temperature, and osmolality, a statistical analysis was performed. The data are expressed a mean with standard deviations ($\pm$ SD) in Table 3. The results were analysed using the analysis of variance one-way ANOVA. Tukey's test was used to determine if the differences between the parameters of the ice cream samples were significant. The statistical appraisal was performed using the STATISTICA 13.3 software (Statsoft Polska, Kraków, Poland). The significance of the test was set at $\alpha = 0.05$.

Table 3. The physical analysis of the milk ice cream.

Sample	Cryoscopic Temperature, °C	Osmotic Pressure, mOsm/kg	Overrun, %	Melting Time, min.
C	-2.502 ± 0.006 ^{fg}	1347 ± 4 ^{ab}	15.35 ± 3.52 ^{bcde}	23.19 ± 2.23 ^{cde}
CH	-2.549 ± 0.005 ^{defg}	1372 ± 3 ^d	8.30 ± 0.49 ^a	22.25 ± 3.05 ^{bcde}
CU	-2.510 ± 0.018 ^{efg}	1355 ± 5 ^{ab}	17.53 ± 2.16 ^{de}	22.37 ± 3.01 ^{cde}
I	-2.486 ± 0.013 ^g	1358 ± 0 ^{bc}	24.40 ± 0.00 ^f	25.14 ± 0.97 ^{de}
IH	-2.531 ± 0.033 ^{defg}	1346 ± 7 ^a	10.90 ± 1.94 ^{abcd}	27.46 ± 1.34 ^{de}
IU	-2.519 ± 0.013 ^{defg}	1348 ± 4 ^{ab}	20.59 ± 0 ^{ef}	26.23 ± 2.09 ^{de}
A	-2.611 ± 0.044 ^{de}	1422 ± 0 ^f	18.86 ± 3.77 ^{ef}	28.26 ± 2.36 ^{de}
AH	-2.921 ± 0.008 ^a	1572 ± 4 ⁱ	9.05 ± 0.00 ^{ab}	27.33 ± 0.00 ^{de}
AU	-2.512 ± 0.066 ^{efg}	1377 ± 0 ^{de}	15.72 ± 0.05 ^{bcde}	30.34 ± 0.00 ^e
B	-2.721 ± 0.022 ^{bc}	1456 ± 0 ^g	31.79 ± 0.86 ^g	12.34 ± 0.00 ^{abc}
BH	-2.608 ± 0.025 ^{def}	1413 ± 0 ^f	16.06 ± 0.37 ^{cde}	12.17 ± 5.06 ^{ab}
BU	-2.754 ± 0.020 ^b	1490 ± 0 ^h	18.27 ± 0.00 ^{ef}	11.23 ± 0.45 ^a
L	-2.578 ± 0.000 ^{defg}	1388 ± 0 ^e	14.32 ± 0.99 ^{abcde}	18.15 ± 1.85 ^{abcd}
LH	-2.622 ± 0.026 ^{cd}	1421 ± 0 ^f	10.63 ± 2.33 ^{abc}	20.13 ± 1.85 ^{abcd}
LU	-2.535 ± 0.017 ^{defg}	1368 ± 4 ^{cd}	20.43 ± 1.10 ^{ef}	23.19 ± 5.89 ^{de}

Different superscript letters in columns represent significant differences in the means of the same parameter ($p < 0.05$). Values represent means $\pm$ standard deviations.

The frequency distribution of ice crystal size was calculated using Microsoft Excel 2011 macro data analysis. The relative frequency of any class interval was calculated as the number of crystals in that class (class frequency) divided by the total number of crystals, and expressed as a percentage (Figures 1–3). According to the method described by Regand and Goff [27], the parameter X_{50} was analysed as the mean diameter (D_A) for 50% of the crystals in the sample. The mean diameter (D_A) and standard deviations (SD) of each class were also calculated (Table 4). The method has been described previously [7,13].

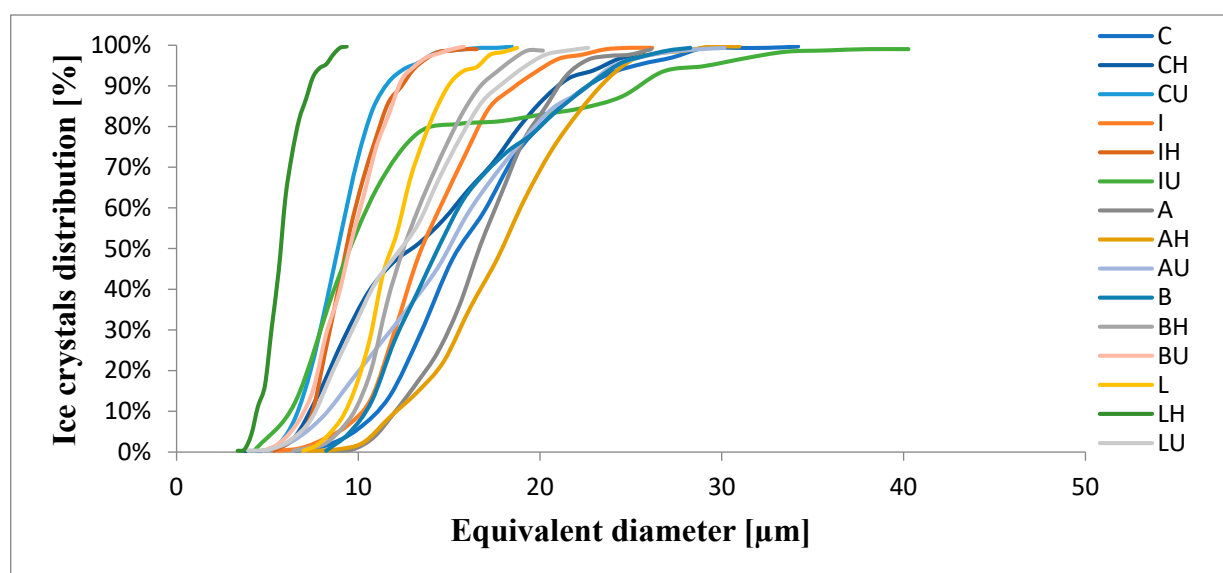
**Figure 1.** Ice crystal size distribution in ice cream after 24 h of storage at -18 °C.

Table 4. Ice crystals size distribution in ice cream after 24 h, 1 and 3 months of storage at -18°C .

Time of Storage and Variant of Ice Cream		Average Diameter D_A in the Class with the Highest Frequency [μm] $\pm$ SD	The Minimal Size of Ice Crystals [μm]	The Maximal Size of Ice Crystals [μm]
C	24 h	16.37 ± 4.71	8.12	32.61
	1 month	17.87 ± 13.16	3.91	27.26
	3 months	24.64 ± 7.45	15.18	30.91
CH	24 h	14.64 ± 5.45	7.10	24.82
	1 month	16.13 ± 4.00	7.58	28.57
	3 months	16.28 ± 3.34	9.01	25.47
CU	24 h	9.05 ± 2.08	5.37	18.53
	1 month	9.86 ± 2.07	5.79	16.40
	3 months	14.74 ± 3.37	8.33	22.69
I	24 h	13.9 ± 3.47	6.98	27.14
	1 month	15.57 ± 4.23	6.90	23.49
	3 months	18.52 ± 4.21	9.32	28.24
IH	24 h	9.64 ± 2.00	5.49	16.29
	1 month	19.9 ± 4.39	11.79	35.50
	3 months	21.74 ± 4.89	12.37	33.92
IU	24 h	13.23 ± 7.54	7.85	21.21
	1 month	18.00 ± 5.45	10.98	27.07
	3 months	18.40 ± 4.57	9.82	26.68
A	24 h	16.70 ± 3.46	9.44	27.52
	1 month	21.27 ± 6.93	8.33	33.86
	3 months	22.03 ± 5.27	11.58	32.33
AH	24 h	17.85 ± 4.20	9.46	28.51
	1 month	17.06 ± 6.93	9.20	26.16
	3 month	19.51 ± 5.27	11.92	29.13
AU	24 h	15.14 ± 5.35	5.00	28.69
	1 month	15.50 ± 3.93	8.01	25.53
	3 months	17.50 ± 8.48	6.30	34.27
B	24 h	16.40 ± 4.65	8.97	28.84
	1 month	16.09 ± 3.72	8.08	27.65
	3 months	16.87 ± 2.90	10.07	25.33
BH	24 h	12.92 ± 2.76	6.43	21.01
	1 month	15.91 ± 4.47	6.53	25.90
	3 months	15.14 ± 3.12	9.84	23.42
BU	24 h	9.45 ± 2.11	5.83	15.14
	1 month	14.28 ± 4.37	6.27	24.24
	3 months	15.13 ± 5.28	7.88	26.94
L	24 h	12.03 ± 2.31	6.97	19.46
	1 month	18.22 ± 4.29	8.59	33.45
	3 months	18.30 ± 4.25	10.64	28.57
LH	24 h	5.84 ± 1.12	4.48	9.17
	1 month	16.11 ± 4.14	8.24	33.21
	3 months	17.10 ± 4.94	9.14	26.38
LU	24 h	12.44 ± 3.92	6.58	20.95
	1 month	14.47 ± 4.83	7.32	22.80
	3 months	14.86 ± 3.38	9.17	24.68

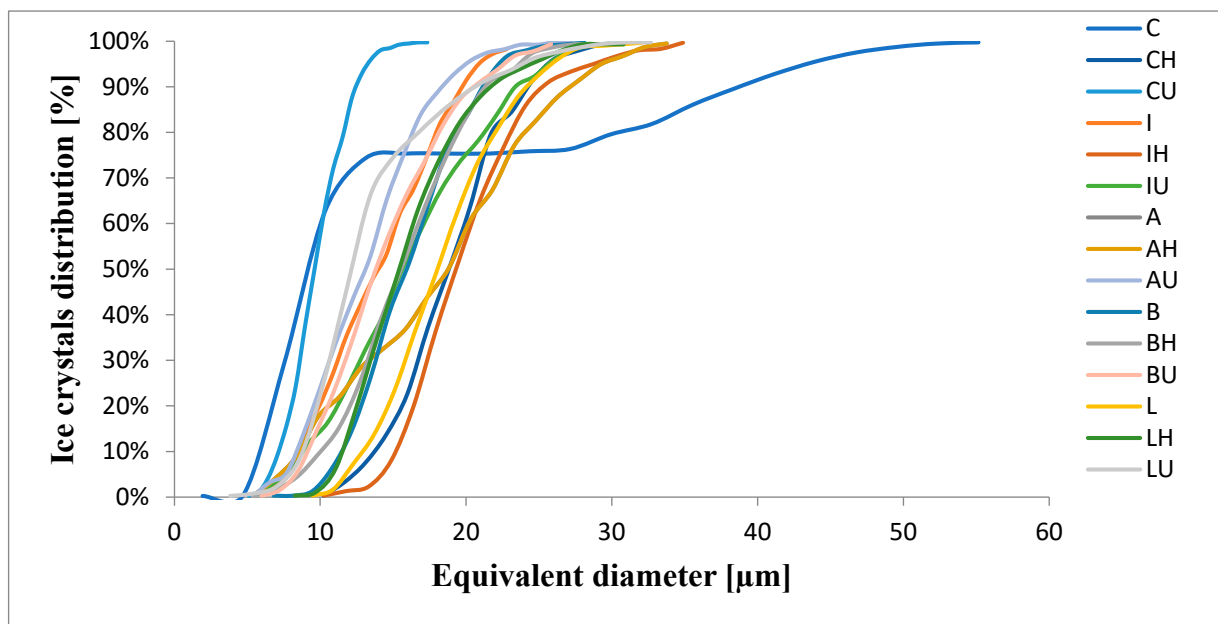


Figure 2. Ice crystal size distribution in ice cream after 1 month of storage at -18°C .

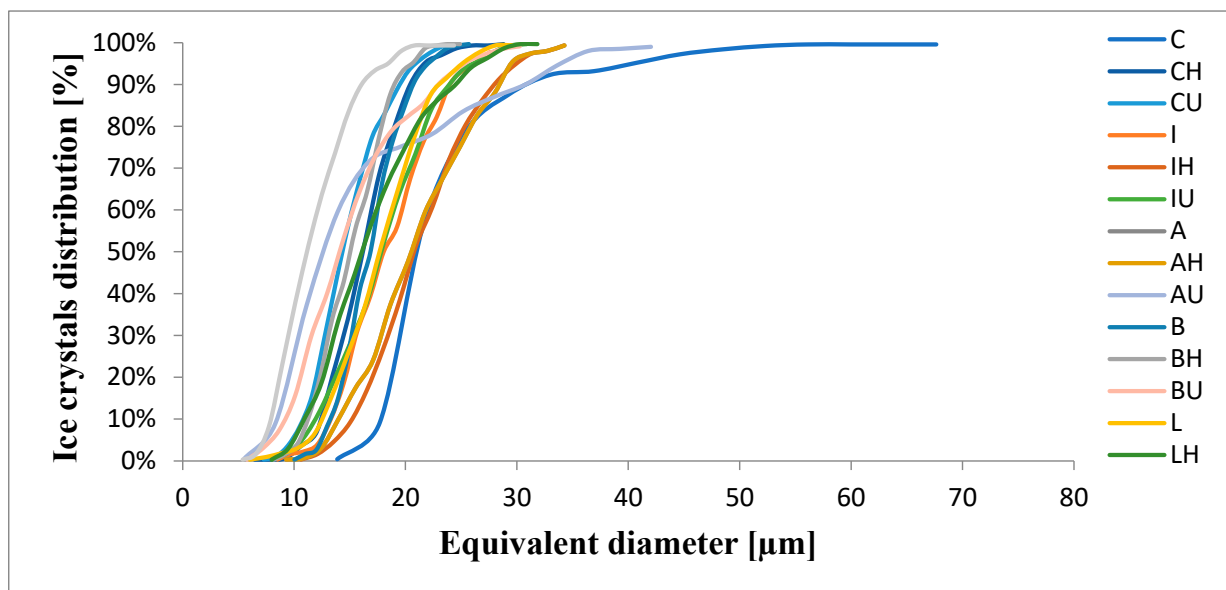


Figure 3. Ice crystal size distribution in ice cream after 3 months of storage at -18°C .

3. Results and Discussion

3.1. The Physical Characteristic of Milk Ice Cream

The physical characteristic of milk ice cream was conducted based on the values of the cryoscopic temperature, osmotic pressure, overrun, and melting time. All of results are collected in Table 3.

The cryoscopic temperature of milk ice cream ranged from -2.921 to -2.486°C (Tables 1–3). Based on the statistical analysis ($p < 0.05$), the differences between the stabilizers and homogenization types used were statistically significant. The lowest temperature was observed for sample AH (samples with the addition of acid hydrolyzates of ι -carrageenan and after traditional homogenization), while the higher one was for sample I (with the addition of ι -carrageenan). Overall, it can be seen that traditional homogenization contributed to the lower temperature, for instance, in samples CH (with the addition of ι -carrageenan), AH (with the addition of acid hydrolyzates of ι -carrageenan), or LH

(with the addition of enzymatic commercial lactase hydrolyzates of ι -carrageenan). While ultrasound homogenization influenced the growth of the temperature. Moreover, it was observed that in the control sample, the differences between the sort of homogenization used were not significant. Otherwise, the temperature for the samples after the ultrasound treatment was slightly lower. The reason for that may be improving in heat transfer during freezing [14]. Ultrasound homogenization might contribute to accelerating the freezing process, which resulted in higher temperatures. Nonetheless, the addition of stabilizers afforded changes in this parameter, with or without homogenization treatment. Hagiwara and Hartel [11] proved that the lower molecular mass of the sweeteners used in ice cream production resulted in greater depression of the freezing point. To follow these observations in our research, the ι -carrageenan and its hydrolyzates differed from each other by molecular mass and structure [13]. Therefore, it may be concluded that hydrolyzates of ι -carrageenan with a lower molecular weight and more flexible structure redounded on the higher cryoscopic temperature.

In the case of osmotic pressure, the lowest value was noted for sample IH (with the addition of ι -carrageenan and after traditional homogenization treatment) at the level of 1346 mOsm/kg and the highest for sample AH (samples with the addition of acid hydrolyzates of ι -carrageenan and after traditional homogenization), at a level of 1572 mOsm/kg. Additionally, the most striking observation was the dependence between cryoscopic temperature and osmotic pressure. In the control sample, the higher temperature of ice cream mixes displayed a lower pressure. For comparison, in samples with the addition of stabilizers, the lower temperature achieved a higher pressure. Moreover, Buniowska-Olejniak et al. [28] also reported that in low-fat milky ice cream with oat β -glucan, the higher osmolality of ice cream mixes resulted in a lower cryoscopic temperature. Such observations were connected with the fact that cryoscopic temperature depends on the type and concentration of dissolved substances. Consequently, the moisture-binding capacity of the stabilizers used may influence the method of creating the ice crystal structure during freezing. Additionally, the variations in the freezing point of ice cream mixes may alter the recrystallization rate during storage [29].

Overrun is defined as the increase in the volume of ice cream, affecting some properties such as melting time or hardness. Moreover, the properties of ice cream mixes (e.g., instance composition) or freezing (e.g., freezing time) may influence the overrun of ice cream [30]. In the present study, the overrun of the milk ice cream that was obtained was less than 32%. The lowest overrun was noted at the level of 8.3% for sample CH (the control sample after the traditional homogenization), while the highest value was in sample B (the sample with the addition of the enzymatic hydrolyzates by β -galactosidase of ι -carrageenan) (Table 3). Alvarez et al. [31] proved that the overrun of milk ice cream ranged from 65.04% to 72.54%. In our research, the reason for the lower overrun of prepared ice cream was presumably the small amount of fat in the recipe. The discrete and partially coalesced fat, as it is hydrophobic, is able to absorb at the air bubble surface. Otherwise, in ice cream with a low fat content, there is not enough fat to cover the whole surface of the air bubbles. Consequently, the air bubble structure may not be stabilized and the final product will have a lower overrun. As found by Sofijan and Hartel [30], the higher overrun would be expected to decrease the thermal diffusivity, providing the insulation effect and thus the ability to retard the melting time. Therefore, in our study, the lower overrun of ice cream may contribute to the lower value of melting time (Table 3). Moreover, the addition of fat replacers such as inulin may decrease the overrun values. In research by Mahdian and Karazhian [32] or Ismail et al. [33], the samples with the addition of inulin had a lower overrun than the samples without this ingredient. Inulin is able to absorb water, which may increase the viscosity of ice cream mixes. Therefore, the higher viscosity might be a primary reason for the decreased whipping abilities of ice cream [10]. According to the statistical appraisal ($p < 0.05$), the differences between samples could be explained by both the stabilizers and the homogenization process. Looking at the influence of stabilizers on the overrun of the obtained ice cream, it can be observed that ι -carrageenan, (I), acid

(A), and enzymatic hydrolyzates (B) of ι -carrageenan improved the air structure in ice cream (Table 3), and the obtained overrun for these samples was from 18.86 to 31.79%. However, the connection of stabilizers and traditional homogenization contributed to the lower overrun, for samples IH, BH, and AH, in which the value was from 9.05 to 16.06%. While the ultrasound homogenization increased this parameter, simultaneously rising the content of air bubbles in the structure of ice cream, the value of the overrun was from 15.72 to 20.43% (samples CU, BU, AU, and LU). Tüker and Dogan [21] also showed that the ultrasound homogenization resulted in increasing the value of the overrun of ice cream. Moreover, these findings proved that differences in such parameters may be a reason for the composition and the same for the ultrasound.

Melting time is a pivotal physical property of ice cream that may be affected by several factors such as ingredients, type and level of stabilizers or emulsifiers, or overrun [34]. Table 3 illustrates the measurement melting time in the presented research, which ranges from 11.23 to 30.34 min. The composition of ice cream and homogenization processes influenced the melting time of the ice cream, which is statistically significant at $p < 0.05$. However, the difference in samples such as I (with ι -carrageenan) (25.14 min), IH (with ι -carrageenan after traditional homogenization) (27.46 min), and IU (with ι -carrageenan after ultrasound treatment) (26.23 min) was not exactly visible to access the effect of ultrasound homogenization compared with the traditional one. The differences in all milk ice cream were connected with the sort of stabilizers used. For instance, it is advisable to use ι -carrageenan or its acid hydrolyzates with the combination of homogenization to prolong the melting time and at the same to raise the consumer acceptability. The addition of enzymatic hydrolyzates of ι -carrageenan, especially after β -galactosidase treatment (B), obtained the lowest value (11.23–12.34 min), even less than for the control sample (C), at 23.19 min. As mentioned in the discussion about overrun, fat may play a key role in properties such as overrun or melting time. The fact that during the freezing process, the clump of de-emulsified fat globules is able to form a protective layer around the air cells and tends to impart foam stability and yield better overrun. However, the samples with less fat clumps had a lower foam stability and overrun. Therefore, ice cream samples with less overrun with fewer air cells resulted in higher heat transfer and hence the faster meltdown of ice cream [34]. Additionally, the use of ultrasound homogenization contributed to the higher heat transfer by acoustic cavitation, hence the melting of ice cream was becoming faster. The next explanation for the short melting time in the prepared milk ice cream could be the addition of inulin. Mahdian and Karazhian [32] reported that with the increasing content of inulin in low-fat ice cream, the melting resistance decreased. Moreover, Góral et al. [26], in ice cream based on coconut milk, proved that the melting resistance was weaker in samples with a higher addition of inulin. Zambrano-Mayorga et al. [35] reported that the melting time in milk ice cream ranged from 19.5 to 31.5, with changes in the amount of whey powder and sweeteners.

3.2. The Microscopy Structure Analysis

Not only are the small ice crystals pivotal, but also the smooth and creamy mouthfeel of ice cream. Hence, controlling the crystallization process and the size or distribution of ice crystals are factors that determine the desired product and prolong the shelf life [15,32,36]. First and foremost, in the presented research, the size of the ice crystals was analysed based on the value of the average diameter (D_A) in Table 4. Moreover, the distribution of the obtained ice crystal structure was studied in accordance with distributions obtained from the image analysis, which were characterised by the values for the ice crystal equivalent diameter at 50% of the cumulative distribution (X_{50}) (Figures 1–3). To estimate the progress of recrystallization, the samples of milk ice cream were examined after 24 h, 1 month, and 3 months of storage at -18°C .

After 24 h of storage, it was noted that the average diameter (D_A) ranged from 9.05 to 17.85 μm (Table 4). The lowest diameter of crystals was observed for sample CU (without the addition of stabilizers and after the ultrasound treatment), while the highest one was

for sample AH (with the acid hydrolyzates of ι -carrageenan and after the traditional homogenization treatment). Overall, the size of the ice crystals in the obtained milk ice cream did not exceed 18 μm . The ultrasound homogenization used contributed to the smaller ice crystals in milk ice cream, except for samples IU (sample with the ι -carrageenan, and after the ultrasound homogenization treatment) and LU (sample with the enzymatic commercial lactase hydrolyzates of ι -carrageenan and after the ultrasound homogenization treatment), in comparison with the traditional homogenization. The effect of ultrasound homogenization may be explained by the violent collapse of bubbles, which initiate the ice nucleation by creating local zones of high pressure in a very short time. Moreover, the force generated by the collapse of cavitation bubbles is able to fragment bigger ice crystals into smaller ones [15,18,37]. Therefore, ultrasound homogenization might be more effective in comparison with traditional homogenization. Based on the value of the ice crystal diameter at 50% of the cumulative distribution of the sample, the X_{50} diameter ranged from 6 to 17 μm (Figure 1). Kamińska-Dwórznička et al. [38], in whey ice cream with the addition of ι -carrageenan as the main stabilizer, showed that the X_{50} was around 15 μm , which was similar to the presented research.

The analysis of the structure of ice crystals after 1 month of storage, for milk ice cream, concludes that the effect of the recrystallization process was visible (Table 4). The average diameter of ice crystals ranged from 9.86 to 21.27 μm . As mentioned in the description of results after 24 h of storage, sample CU (without the addition of stabilizers and after the ultrasound treatment) was characterized the smallest size of ice crystals. The highest progress of recrystallization was noticed for sample LH (sample with the enzymatic commercial lactase hydrolyzates of ι -carrageenan, and after the ultrasound treatment), owing to the fact that the crystals grew almost 11 μm . The most striking observation after this time in storage was the fact that the ultrasound treatment significantly contributed to the inhibition of the recrystallization process. In all of the prepared ice cream samples, the average diameter was smaller (from 9.86 to 18 μm) than in the samples after mechanical homogenization (from 16.11 to 19.90) or only with the addition of stabilizers (15.57 to 21.27 μm). Moreover, the value of ice crystal diameter at 50% of the cumulative distribution of the sample (X_{50} diameter) was observed, extending from 9 to 20 μm (Figure 2). In comparison with the results recorded after 24 h of storage, the recrystallization process could be proved based on parameter X_{50} . The research by Kamińska-Dwórznička et al. [19] also showed that using ultrasound (the frequency of 21.5 Hz) during freezing also contributed to the lower diameter of ice crystals for mango sorbet, of less than 10 μm . Moreover, in research by Mortazavi and Tabatabaie [18], the effect of ultrasound shortened the freezing time and at the same increased the overrun of ice cream, which improved the sensory of the final product. Additionally, Islam et al. [39] proved that ultrasound effectively triggered ice nucleation and minimized the size of the ice crystals in mushrooms. On the other hand, Dai et al. [40] indicated that the size of ice crystals was larger at higher nucleation temperatures and the sublimation time was reduced by almost 22 % compared with the control sample.

Looking at the results after 3 months of storage (Table 4 and Figure 3), it is seen that the average diameter of ice crystals was less than 25 μm . The highest ice crystals were noted for control sample C, while the smallest result was 14.74 μm for the control sample after ultrasound treatment (CU). For all samples with the addition of stabilizers but without homogenization treatment, the size of the ice crystals ranged from 16.87 to 22.03 μm . However, samples with the addition of stabilizers and after the traditional homogenization ranged from 15.14 to 21.74 μm , while after the ultrasound treatment, they ranged from 14.86 to 18.4 μm . Consequently, it was seen that the implementation of the homogenization process was necessary to obtain smaller ice crystals. However, the ultrasound was more beneficial than the traditional method. What should be highlighted is that the control sample (C) without stabilizers but after the ultrasound treatment achieved the smallest value for the average diameter at a level of 14.74 μm . Additionally, the range of obtained ice crystals was close to 20 or less than this value, which may guarantee the desirable texture of ice cream, especially for consumers. Based on the value of the X_{50} diameter, ranging

from 12 to 21 μm (Figure 3). Notable, to compare to results after 1 month of storage, the recrystallization process could be observed based on the parameter X_{50} .

Chow et al. [41] proved that existing big crystals might be broken up into smaller ice crystals by high-intensity ultrasound. Maybe, in this case, such an ability of ultrasound was observed. Therefore, this increases the chances of effectiveness to inhibit the recrystallization process in milk ice cream. Taking into consideration the influence of the stabilizers used on the recrystallization process, it may be seen that in samples with ι -carrageenan (I) and its enzymatic hydrolyzates (B and L), the size of ice crystals was similar (around 18 μm). For sample A, with the acid hydrolyzates of ι -carrageenan, the average diameter of ice crystals was higher (22.03 μm). The reason may be the difference in the molecular structure of these stabilizers. In our previous study [13], it was proven that the mentioned enzymatic hydrolyzates of ι -carrageenan were more flexible based on FTIR (Fourier Transform Infrared Spectroscopy) analysis, and the vibration intensity of the $-\text{OH}$ groups in the model solutions was seen in comparison with acid hydrolyzates. Overall, for the obtained hydrolyzates of ι -carrageenan, smaller vibrations were observed at 1213 and 914 cm^{-1} , which was connected with the decreasing number of sulphate groups and determined the better flexibility of hydrolyzates. Hence, the distinctive influence of stabilizers on the ice crystal structure was noted. Moreover, the average diameter in samples B and L had a similar value in comparison with sample A after 3 months of storage. It is mentioned here because based on the FTIR analysis, it was confirmed that samples B and L after the enzymatic hydrolysis had a similar course on the FTIR spectra. It can be evidence that the enzymatic hydrolyzates of ι -carrageenan were more favourable in milk ice cream than ι -carrageenan and its acid hydrolyzates. In the research by Tecson et al. [42], the ultrasound-assisted depolymerization of κ -carrageenan was investigated. It was proven that the raw carrageenan average molecular mass was reduced by 89.32% while in the irradiated κ -carrageenan it was only 41.98%. Such observations were connected with the fact that larger molecules present more resistance to flow and the same accumulate greater shear force, which leads to more frequent ruptures as the cavitation bubbles collapse than in the shorter polymer. Therefore, in our research, presumably, the effect of ι -carrageenan was similar even if the initial molecular mass of this polymer was higher than the obtained hydrolyzates [13]. Moreover, in the same research, Tecson et al. [42] noticed that after ultrasound treatment, κ -carrageenan with a lower molecular mass revealed the retention of absorbance peaks with the decrease in the sulphate functional group and most of the peaks were retained, with one exception of a methylene peak. In our study, the chain of ι -carrageenan underwent changes by ultrasonic cavitations, which led to a similar effect on the IRI activity in ice cream as the hydrolyzates. Moreover, it generated information that hydrolyzates will be more stable and less vulnerable on the ultrasound treatment than pure ι -carrageenan. Nonetheless, further studies will be recommended to investigate the possible changes in the molecular structure of ι -carrageenan and its hydrolyzates after ultrasound treatment. Finally, the mechanism of ultrasound, such as inducing ice nucleation as well as increasing heat and mass transfer, is connected with the initial step of ice cream production. In our research, it was proven that ultrasound forces were so strong that they influenced the structure of ice cream even after a few months of storage and prevented excessive growth of ice crystals.

The observation of the progress of the recrystallization process was also analysed based on images (Figures 4 and 5). To access this we used the overview of the appearance of ice crystals during storage after 24 h and 3 months of storage at -18°C (only representative photos were chosen to show in the paper, not including the photos after 1 month). Looking at the images, it may be noticed that the shape of the ice crystals was round and regular (Figure 4). The research by Sanchez-Garcia et al. [43] showed that in lactose solutions with the presence of whey proteins and κ -carrageenan, after 48 h of ultrasound-assisted crystallization, the shape of crystals was mostly irregular, even with the tomahawk-like shape. Nonetheless, it is known that high ultrasound energies may be responsible for breaking crystals already formed and generating a disruption of agglomerates or even

nuclei [44]. After 24 h of storage of ice cream, the changes between the samples treated with different sorts of homogenization were not visible. For example, in samples AH_24h (sample with acid hydrolyzates of ι -carrageenan and after traditional homogenization) and AU_24h (sample with acid hydrolyzates of ι -carrageenan and after ultrasound), the changes were connected with the size of ice crystals (which was confirmed in Table 4), but not in the appearance of crystals. A similar effect on the shape of the ice structure was noticed in frozen pork tissue [45] or in frozen mushrooms [39], that ultrasound treatment unified the shape and sizes of the ice crystals. Moreover, Xu et al. [46] indicated that differences in the structure of ice crystals may depend on the ultrasonic power intensities.

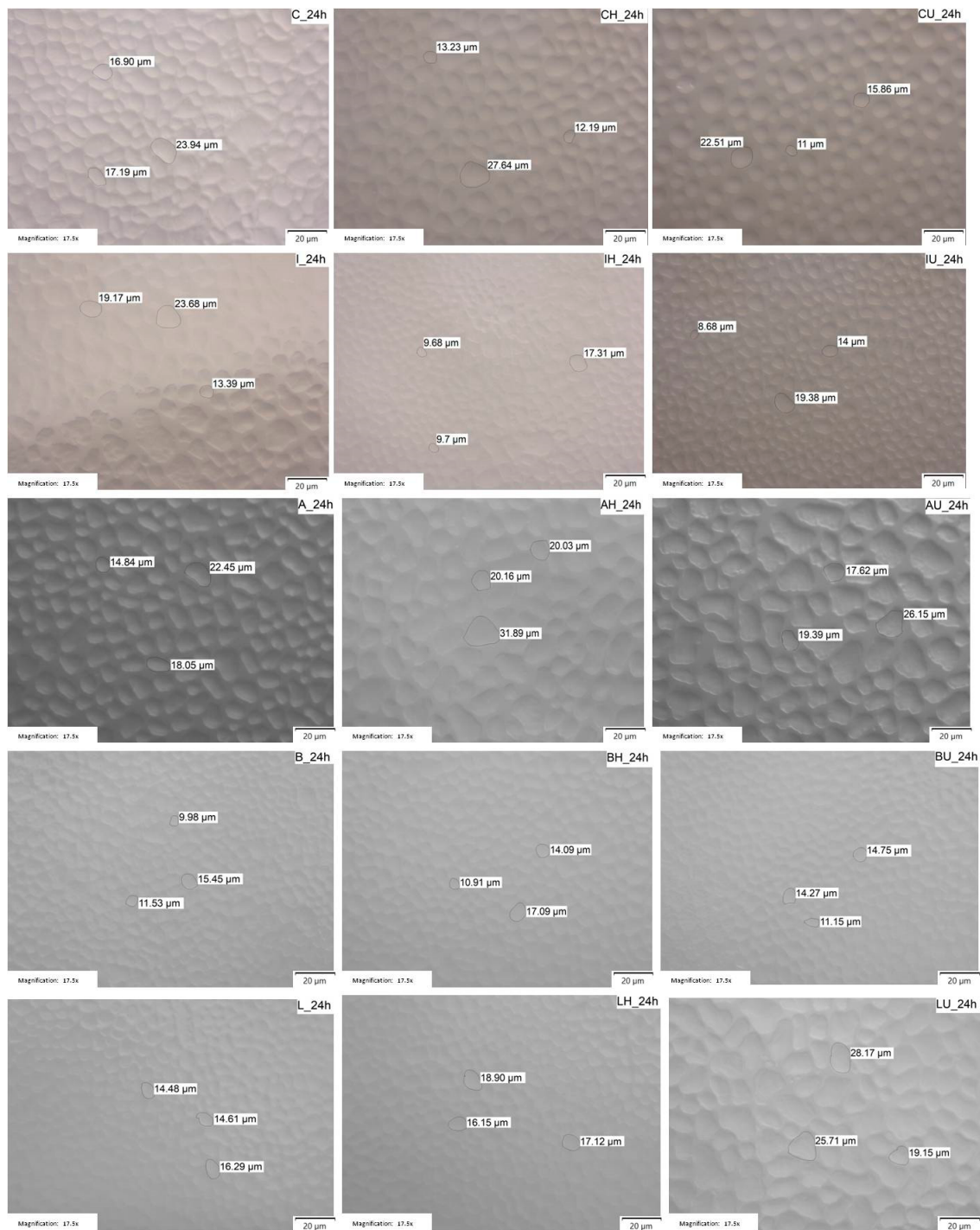


Figure 4. Ice crystal size distribution in ice cream after 24 h of storage at -18°C .

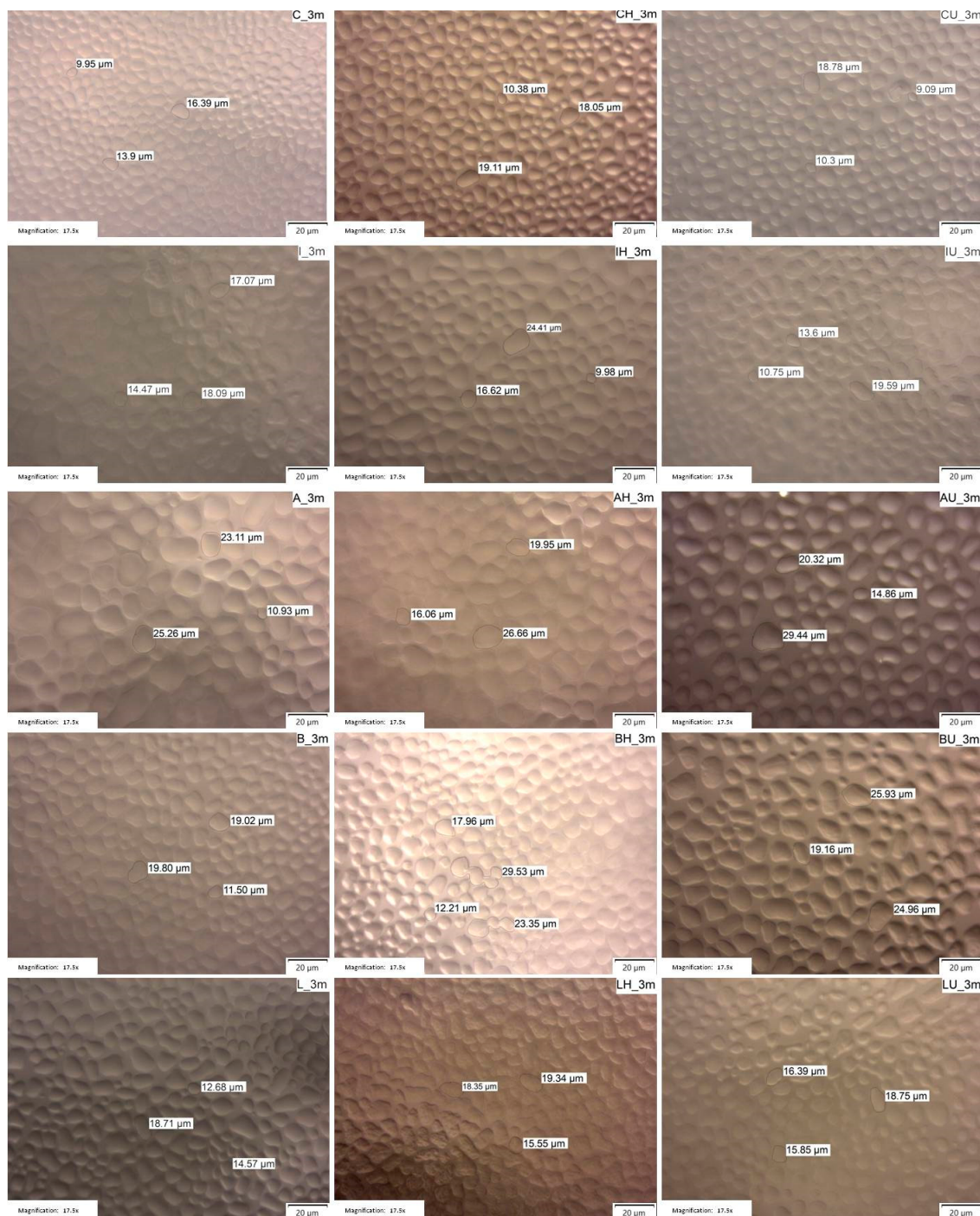


Figure 5. Ice crystal size distribution in ice cream after 3 months of storage at -18°C .

After 3 months of storage, milk ice cream was characterised by different sizes as a result of the progress of recrystallization (Figure 5 and Table 4). In addition, as mentioned earlier, the size of the ice crystals was regular and round. The changes were strictly connected with the size of the ice crystals, especially in samples after ultrasound treatment, where the average diameter was the smallest in comparison with the other samples of ice cream. However, in the image, BH_3m (the sample with enzymatic β -galactosidase hydrolyzates of ι -carrageenan and after the traditional homogenization); the coalescence between the crystals was marked, which proved the recrystallization phenomena during storage (Figure 5).

Exploratory notes: C, control sample (without stabilizers); I, sample with ι -carrageenan, locust bean gum (LBG), and xanthan gum; A, B, and L, sample with hydrolyzates obtained by acid, β -galactosidase, and commercial lactase treatment of ι -carrageenan, respectively, LBG, and xanthan gum; H, samples after traditional homogenization treatment; U, samples after ultrasound homogenization treatment.

Exploratory notes: C, control sample (without stabilizers); I, sample with ι -carrageenan, locust bean gum (LBG), and xanthan gum; A, B, and L, samples with hydrolyzates obtained by acid, β -galactosidase, and commercial lactase treatment of ι -carrageenan, respectively, LBG and xanthan gum; H, samples after traditional homogenization treatment; U, samples after ultrasound homogenization treatment.

4. Conclusions

Ultrasound treatment of ice cream resulted in the reduction in their sizes during storage and simultaneously increased the IRI (ice recrystallization inhibition) activity of the prepared samples. After 3 months of storage, in the control samples, after ultrasound treatment, the average diameter did not exceed 15 μm . In comparison, among the samples with the addition of stabilizers, this value was less than 19 μm . The beneficial influence of ultrasound is connected to acoustic cavitation and accelerating heat and mass transfer. This occurred to initiate the ice nucleation of ice crystals and improve the condition to build the ice structure during freezing. As a result, in cryoscopic temperature, osmotic pressure, overrun, and melting time such changes were observed. It was found that ultrasound influenced the increase in cryoscopic temperature in comparison with traditional homogenization. Additionally, the overrun of ice cream ranged from 8.3 to 31.79%. The lower overrun was created by the small amount of fat in ice cream and presumably the addition of inulin. Therefore, the influence of sort of homogenization was not significantly visible. The melting time of the prepared milk ice cream was less than 29 min. According to the results of the melting time, it was concluded that ultrasound homogenization in comparison with the traditional one did not differ, so they can be used interchangeably.

Detailed studies on the physical and presumably sensory properties, as well as the shelf stability of the ice cream from ultrasound mixes, will be recommended. Studies on the improvement in ice cream properties through the inclusion of stabilizers with the connection of ultrasound treatment can yield further optimization of the efficiency of the parameter. Finally, we investigated the possibilities of producing a milk ice cream with HIU (high-intensity ultrasound) that can confer desirable properties, which could possibly yield economically and environmentally attractive possibilities.

Author Contributions: Conceptualization, A.K. and A.K.-D.; methodology, A.K. and E.J.; software, A.K.; validation, A.K., A.K.-D. and E.J.; formal analysis, A.K. and A.K.-D.; investigation, A.K., A.K.-D. and E.J.; resources, E.J.; data curation, A.K., A.K.-D. and E.J.; writing—original draft preparation, A.K., A.K.-D. and E.J.; writing—review and editing, A.K., A.K.-D. and E.J.; visualization, A.K.; supervision, E.J.; project administration, A.K.-D. All authors have read and agreed to the published version of the manuscript.

Funding: Research equipment was purchased as part of the “Food and Nutrition Centre-modernisation of the WULS campus to create a Food and Nutrition Research and Development Centre (CŻiŻ)” co-financed by the European Union from the European Regional Development Fund under the Regional Operational Programme of the Mazowieckie Voivodeship for 2014-2020 (Project No. RPMA.01.01.00-14-8276/17).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Goff, H.D. Colloidal aspects of ice cream—A review. *Int. Dairy J.* **1997**, *7*, 363–373. [\[CrossRef\]](#)
- Soukoulis, C.; Chandrinos, I.; Tzia, C. Study of the functionality of selected hydrocolloids and their blends with κ -carrageenan on storage quality of vanilla ice cream. *LWT-Food Sci. Technol.* **2008**, *41*, 1816–1827. [\[CrossRef\]](#)
- Inoue, K.; Ochi, H.; Habara, K.; Taketsuka, M.; Saito, H.; Ichihashi, N.; Iwatsuki, K. Modeling of the effect of freezer conditions on the hardness of ice cream using response surface methodology. *J. Dairy Sci.* **2009**, *92*, 5834–5842. [\[CrossRef\]](#) [\[PubMed\]](#)
- Clarke, C. *The Science of Ice Cream*; The Royal Society of Chemistry: Cambridge, UK, 2004.
- Wildmoser, H.; Scheiwiller, J.; Windhab, E.J. Impact of disperse microstructure on rheology and quality aspects of ice cream. *LWT-Food Sci. Technol.* **2004**, *37*, 881–891. [\[CrossRef\]](#)
- Gaukel, V.; Leiter, A.; Spiess, W.E.L. Synergism of different fish antifreeze proteins and hydrocolloids on recrystallization inhibition of ice in sucrose solutions. *J. Food Eng.* **2014**, *141*, 44–50. [\[CrossRef\]](#)
- Kamińska-Dwórznička, A.; Gondek, E.; Łaba, S.; Jakubczyk, E.; Samborska, K. Characteristics of instrumental methods to describe and assess the recrystallization process in ice cream systems. *Foods* **2019**, *8*, 117. [\[CrossRef\]](#)
- Smith, A.K.; Goff, H.D.; Kakuda, Y. Microstructure and rheological properties of whipped cream as affected by heat treatment and addition of stabilizer. *Int. Dairy J.* **2000**, *10*, 295–301. [\[CrossRef\]](#)
- Fiol, C.; Prado, D.; Romero, C.; Laburu, N.; Mora, M.; Alava, I.J. Introduction of a new family of ice creams. *Int. J. Gastron. Food Sci.* **2017**, *7*, 5–10. [\[CrossRef\]](#)
- Adapa, S.; Schmidt, K.A.; Jeon, I.J.; Herald, T.J.; Flores, R.A. Mechanisms of ice crystallization and recrystallization in ice cream: A review. *Food Rev. Int.* **2000**, *16*, 259–271. [\[CrossRef\]](#)
- Hagiwara, T.; Hartel, R.W. Effect of sweetener, stabilizer, and storage temperature on ice recrystallization in ice cream. *J. Dairy Sci.* **1996**, *79*, 735–744. [\[CrossRef\]](#)
- Lomolino, G.; Zannoni, S.; Zabara, A.; Da Lio, M.; De Iseppi, A. Ice recrystallisation and melting in ice cream with different proteins levels and subjected to thermal fluctuation. *Int. Dairy J.* **2020**, *100*, 104557. [\[CrossRef\]](#)
- Kot, A.; Kamińska-Dwórznička, A.; Antczak, A.; Jakubczyk, E.; Matwijczuk, A. Effect of ι -carrageenan and its acidic and enzymatic hydrolysates on ice crystal structure changes in model sucrose solution. *Colloids Surf. A Physicochem. Eng. Asp.* **2022**, *643*, 128744. [\[CrossRef\]](#)
- Chemat, F.; Zill-e-Huma; Khan, M.K. Application of ultrasound in food technology: Processing, preservation and extraction. *Ultrason. Sonochem.* **2011**, *18*, 813–835.
- Akdeniz, V.; Akalin Sibel, A. New approach for yoghurt and ice cream production: High-intensity ultrasound. *Trends Food Sci. Technol.* **2019**, *86*, 392–398. [\[CrossRef\]](#)
- Carrillo-Lopez, L.M.; Garcia-Galicia, I.A.; Tirado-Gallegos, J.M.; Sanchez-Vega, R.; Huerta-Jimenez, M.; Ashokkumar, M.; Alarcon-Rojo, A.D. Recent advances in the application of ultrasound in dairy products: Effect on functional, physical, chemical, microbiological and sensory properties. *Ultrason. Sonochem.* **2021**, *73*, 105467. [\[CrossRef\]](#) [\[PubMed\]](#)
- Firouz, M.S. Application of high-intensity ultrasound in food processing for improvement of food quality. In *Design and Optimization of Innovative Food Processing Techniques Assisted by Ultrasound; Developing Healthier and Sustainable Food Products*; Barba, F., Cravotto, G., Chemat, F., Lorenzo Rodriguez, J.M., Sichert Munekata, P.E., Eds.; Elsevier Inc.: Amsterdam, The Netherlands, 2021; pp. 155–159.
- Mortazavi, A.; Tabatabaie, F. Study of ice cream freezing process after treatment with ultrasound. *World Appl. Sci. J.* **2008**, *4*, 188–190.
- Kamińska-Dwórznička, A.; Kot, A.; Jakubczyk, E.; Buniowska-Olejek, M.; Nowacka, M. Effect of ultrasound-assisted freezing on the crystal structure of mango sorbet. *Crystals* **2023**, *13*, 396. [\[CrossRef\]](#)
- Nazarewicz, S.; Kozłowicz, K.; Kobus, Z.; Gładyszewska, B.; Matwijczuk, A.; Slusarczyk, L.; Skrzypek, T.; Sujka, M.; Kozłowicz, N. The Use of Ultrasound in Shaping the Properties of Ice Cream with Oleogel Based on Oil Extracted from Tomato Seeds. *Appl. Sci.* **2022**, *12*, 9165. [\[CrossRef\]](#)
- Tüker, D.A.; Dogan, M. Effect of ultrasound homogenization on the structural and sensorial attributes of ice cream: Optimization with Taguchi and data envelopment analysis. *J. Food Meas. Charact.* **2021**, *15*, 4888–4898.
- Kamińska-Dwórznička, A.; Skrzypczak, P.; Gondek, E. Modification of kappa carrageenan by β -galactosidase as a new method to inhibit recrystallization of ice. *Food Hydrocoll.* **2016**, *61*, 31–35. [\[CrossRef\]](#)
- O'Sullivan, J.; Park, M.; Beevers, J. The effect of ultrasound upon the physicochemical and emulsifying properties of wheat and soy protein isolates. *J. Cereal Sci.* **2016**, *69*, 77–84. [\[CrossRef\]](#)
- da Silva, L.F.R.; da Silva Gomes, A.; Castro, D.R.G.; das Chagas do Amaral Souza, F.; Mar, J.M.; Souza da Silva, L.; Sanches, E.A.; de Araújo Bezerra, J.; Bakry, A.M.; Campelo, P.H. Ultrasound-assisted homogenisation and gum Arabic combined to physicochemical quality of cupuaçu juice. *J. Food Process. Preserv.* **2019**, *43*, e14072. [\[CrossRef\]](#)
- Dłużewska, E.; Gazda, B.; Leszczyński, K. Wpływ wybranych hydrokoloidów polisacharydowych na jakość koncentratów lodów owocowych. *Acta Sci. Pol. Technol. Aliment.* **2003**, *2*, 97–107.
- Góral, M.; Kozłowicz, K.; Pankiewicz, U.; Góral, D.; Kluza, F.; Wójtowicz, A. Impact of stabilizers on the freezing process, and physicochemical and organoleptic properties of coconut milk-based ice cream. *LWT-Food Sci. Technol.* **2018**, *92*, 516–522. [\[CrossRef\]](#)

27. Regand, A.; Goff, H.D. Structure and ice recrystallization in frozen stabilized ice cream model systems. *Food Hydrocoll.* **2003**, *17*, 95–102. [\[CrossRef\]](#)
28. Buniowska-Olejnik, M.; Mykhalevych, A.; Polishchuk, G.; Sapiga, V.; Znamirska-Piotrowska, A.; Kot, A.; Kamińska-Dwórznińska, A. Study of Water Freezing in Low-Fat Milky Ice Cream with Oat β -Glucan and Its Influence on Quality Indicators. *Molecules* **2023**, *28*, 2924. [\[CrossRef\]](#)
29. Patel, M.R.; Baer, R.J.; Acharaya, M.R. Increasing the protein content of ice cream. *J. Dairy Sci.* **2006**, *89*, 1400–1406. [\[CrossRef\]](#)
30. Sofijan, P.S.; Hartel, R.W. Effect of overrun on structural and physical characteristic of ice cream. *Int. Dairy J.* **2004**, *14*, 255–262. [\[CrossRef\]](#)
31. Alvarez, V.B.; Wolters, C.L.; Vodovotz, Y.; Ji, T. Physical properties of ice cream containing milk protein concentrates. *J. Dairy Sci.* **2005**, *88*, 862–871. [\[CrossRef\]](#)
32. Mahdian, E.; Karazhian, R. Effect of fat replacers and stabilizers on rheological, physicochemical and sensory properties of reduced-fat ice cream. *J. Agric. Sci. Technol.* **2013**, *15*, 1163–1174.
33. Ismail, E.A.; Al-Saleh, A.A.; Metwalli, A.A.M. Effect of Inulin Supplementation on Rheological Properties of Low-Fat Ice Cream. *Life Sci.* **2013**, *10*, 1742–1746.
34. Roy, S.; Hussain, S.A.; Prasad, W.G.; Khetra, Y. Quality attributes of high protein ice cream prepared by incorporation of whey protein isolate. *Appl. Food Res.* **2022**, *2*, 100029. [\[CrossRef\]](#)
35. Zambrano-Mayorga, L.; Ramirez-Navas, J.; Ochoa-Martinez, C.I. Influence of the formulation on the thermophysical properties and the quality parameters of dairy ice cream. *Dyna* **2019**, *86*, 117–125. [\[CrossRef\]](#)
36. Regand, A.; Goff, H.D. Effect of biopolymers on structure and ice recrystallization in dynamically frozen ice cream model systems. *J. Dairy Sci.* **2002**, *85*, 2722–2732. [\[CrossRef\]](#)
37. Cheng, L.N.; Sun, D.W.; Zhu, Z.W.; Zhang, Z. Emerging techniques for assisting and accelerating food freezing processes: A of recent research progresses. *Crit. Rev. Food Sci. Nutr.* **2017**, *57*, 769–781. [\[CrossRef\]](#)
38. Kamińska-Dwórznińska, A.; Łaba, S.; Jakubczyk, E. The effects of selected stabilizers addition on physical properties and change in crystal structure of whey ice cream. *LWT-Food Sci. Technol.* **2022**, *154*, 112841. [\[CrossRef\]](#)
39. Islam, M.N.; Zhang, M.; Fang, Z.; Sun, J. Direct contact ultrasound assisted freezing of mushroom (*Agaricus bisporus*): Growth and size distribution of ice crystals. *Int. J. Refrig.* **2015**, *57*, 46–53. [\[CrossRef\]](#)
40. Dai, C.; Zhou, X.; Zhang, S.; Zhou, N. Influence of ultrasound-assisted nucleation on freeze-drying of carrots. *Dry Technol.* **2016**, *34*, 1196–1203. [\[CrossRef\]](#)
41. Chow, R.; Blindt, R.; Chivers, R.; Povey, M. A study on the primary and secondary nucleation of ice by power ultrasound. *Ultrasonics* **2005**, *43*, 227–230. [\[CrossRef\]](#)
42. Tecson, M.G.; Abad, L.V.; Ebajo, V.D., Jr.; Camacho, D.H. Ultrasound-assisted depolymerization of kappa-carrageenan and characterization of a degradation product. *Ultrason. Sonochem.* **2021**, *73*, 105540. [\[CrossRef\]](#)
43. Sánchez-García, Y.; García-Vega, K.S.; Leal-Ramos, M.Y.; Salmeron, I.; Gutiérrez-Méndez, N. Ultrasound-assisted crystallization of lactose in the presence of whey proteins and κ -carrageenan. *Ultrason. Sonochem.* **2018**, *42*, 714–722. [\[CrossRef\]](#)
44. de Castro, M.D.; Priego-Capote, F. Ultrasound-assisted crystallization (sonocrystallization). *Ultrason. Sonochem.* **2007**, *14*, 717–724. [\[CrossRef\]](#) [\[PubMed\]](#)
45. Zhang, P.Z.; Zhu, Z.W.; Sun, D.W. Using power ultrasound to accelerate food freezing processes: Effects on freezing efficiency and food microstructure. *Crit. Rev. Food Sci. Nutr.* **2018**, *58*, 2842–2853. [\[CrossRef\]](#) [\[PubMed\]](#)
46. Xu, B.-G.; Zhang, M.; Bhandari, B.; Cheng, X.-F.; Islam, M.N. Effect of ultrasound-assisted freezing on the physic-chemical properties and volatile compounds of red radish. *Ultrason. Sonochem.* **2015**, *27*, 316–324. [\[CrossRef\]](#) [\[PubMed\]](#)

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.