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RESEARCH ARTICLE

The Effect of Alpha-Tocopheryl Acetate Addition on the Quality of Dairy Ice Cream

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Abstract

Ice cream is a popular frozen dessert eagerly consumed regardless of the season. The production and storage of ice cream is done under frozen conditions due to the thermal instability of this product. Thermal fluctuations disrupt the structure of the ice cream, causing melting, shrinkage and recrystallisation of the ice, which ultimately leads to deterioration of the product. The addition of substances with protective properties can improve the quality of ice cream during storage and its acceptability to the consumers. In this study, the effect of alpha-tocopheryl acetate on the physicochemical properties of ice cream and its quality attributes (melting rate, overrun, texture) was investigated. In addition, the ice cream was subjected to temperature fluctuations to assess changes in its quality parameters. Fortification of the ice cream mixes with ATA increase the glass transition temperatures, while the freezing temperatures were maintained at a similar level. The overrun of fresh ice cream samples ranged from 48.55±1.08% to 51.99±0.20% and decreased with the storage time. Ice cream subjected to heat shocks characterized with similar overrun values. A slight shift in the melting curves was observed in ice cream stored for 90 days, but the amount of drip-off material appeared to be similar (in the range 56.50±3.64% to 77.32±0.52%) in all the samples tested. The thermal treatment caused significant changes in the melting process - less intense melting and a lower melting rate were observed. ATA addition reduced the hardness of the ice cream, which could be positively perceived by the consumers.

KEYWORDS
ice cream, meltdown, overrun, texture analysis, alphatocopheryl acetate

Introduction

Ice cream is a specific kind of frozen dessert which is widely-consumed by the people in all ages. Ice cream can be described as a frozen foam which, in fact, is a multiphase complex system consisting of ice crystals, partially coalesced fat globules and air cells, all embedded in a freeze-concentrated unfrozen phase [Goff 2013]. Due to its specific composition and characteristics, ice cream should be served and consumed at low temperatures. The typical base for the dairy ice cream mixture consists of milk, milk fat, stabilizers and emulsifiers [Goff 2013]. Several components are added to ice cream formulation to improve its sensory, olfactory and even physicochemical features. These include flavorings, colorants, vegetables oils, fruits and fruit pulps, artificial sweeteners, dietary fibers, vitamins, and probiotic bacteria [Da Silva Junior and Lannes 2011; Kowalczyk et al. 2021; Senanayake et al. 2013; Shamshad et al. 2023]. Therefore, ice cream can show a high nutritional value. In recent years, the food industry additionally develops the alternative plant-based ice cream sector which arouse as a response to vegan nutrition trends and the increase of lactose intolerance of the consumers [Ghaderi et al. 2021]. The traditional base ingredients are replaced with plant-based components of which the most mentioned are vegetable oils, inulin, plant based-milks and plant proteins (soy protein, pea protein, chickpea protein, rice protein) [Erem et al. 2024; Hasan et al. 2023; Narala et al. 2022; Ng et al. 2023].

The components used for ice cream blends determine the final ice cream structure together with crucial ice cream features, especially the melting behavior, overrun, the texture and the sensory characteristics [Muse and Hartel 2004]. Ice crystal size is crucial for the textural quality perceived by the consumers. Typical desired ice crystal sizes vary between 10 and 20 μm which is recognized as a smooth texture that provides a pleasant mouthfeel. Larger crystals, especially the significant number crystals with size exceeding diameter of 75 μm, enhance the sandiness and iciness, and is often perceived as a grainy texture of the product [Buyck et al. 2011; Kamińska-Dwórznicka et al. 2022; Russell 1999]. The creamy and greasy sensation is rather associated with partially-coalesced fat in dairy desserts [Dresselhuis et al. 2008]. In addition, proteins act as emulsifying agents and show water-holding capacity what plays an important role in formation of ice cream structure [Roy et al. 2022]. Ice cream is thermodynamically unstable product and any changes in the external temperature during storage or transportation affect its quality. The most visible changes include ice cream shrinkage, ice crystals melting and consequent recrystallization into crystals of greater average size [Regand and Goff 2003]. However, this phenomenon is associated with the refreezing of melted water onto the existing ice crystals rather than the formation of new small ice crystals. This can be however monitored by supplementing ice cream blends with functional substance which show cryoprotective effect due to water-binding capacity or improving the resistance to thermal stress and melting (Jeremiah 2019; Marshall et al., 2003]. Typically, various hydrocolloids are used as these functional substances as they characterize various modes of action. Components commonly added to ice cream blends which stabilize ice cream structure and inhibit recrystallisation are predominately agar, κ-carrageenan, and locust bean gum [Miller-Livney and Hartel 1997]. On the contrary, the control of ice cream melting, ice cream shrinkage limitation or syneresis prevention are reached after alginate, gelatin, or guar gum addition.

In this research the use of alpha-tocopheryl acetate as an additive to dairy ice cream was evaluated. The study provides new information on the use of substances commonly used in dietary supplements for food design and the verification of structuring properties, which are an important factor in both the production and cold storage of ice cream. The aim of the study was to assess the effect of adding selected concentrations of ATA on various parameters related to ice cream stability. Any changes in physicochemical properties, overrun, melting behavior (melting rate, degree of ice cream melting) and texture of ice cream were investigated for ice cream samples subjected to thermal fluctuations and frozen storage.

MATERIALS AND METHODS

Ice cream formulations and ice cream preparation

The material for the study was ice cream prepared from formulations with the composition shown in Table 1. The ice cream mixes were prepared from skimmed milk powder (Mlekovita, Wysokie Mazowieckie, Poland), sucrose (Diamant, Poland), milk fat (SM Mlekpol, Grajewo, Poland), emulsifying-stabilizing preparation (Palsgaard A/S, Juelsminde, Denmark) and water and then pasteurized (80.0±1.0°C, 15 min) with continuous stirring (80 rpm) using Achiever 5000 Stirrer (OHAUS Europe GmbH, Greifensee, Switzerland). The pasteurized formulations were cooled to 50.0±1.0°C and homogenized (CAT homogenizer, model Unidrive X 1000MPW-120, Germany) at 14,000 rpm for 15 min, after which α-tocopheryl acetate (ATA; Merck KGaA, Germany, Darmstadt) was added at the appropriate concentration. The ice cream mixes were aged at 4.0±1.0°C for 20 hours.

Table 1. The composition of ice cream formulations models

Component [%]	Ice cream formulation model			
	T0	T1	T2	T3
sucrose	10.00	10.00	10.00	10.00
skim milk powder	12.00	12.00	12.00	12.00
milk fat	8.00	8.00	8.00	8.00
Palsgaard 260	0.50	0.50	0.50	0.50
α-tocopheryl acetate (ATA)	-	0.01	0.02	0.03
water	69.50	69.50	69.50	69.50

After aging, the ice cream formulations were transferred to a freezer (Resto Quality, model RQ18T, Italy) and frozen in the automatic mode to maintain the temperature of-5.0±0.1°C of the extruded ice cream mass. The ice cream mass was packaged in 250 ml plastic containers and stored under freezing conditions at -35.0°C for a period of 7 and 90 days.

Physicochemical analysis

Selected physicochemical parameters were determined in aged ice cream formulations. The dry matter content was measured according to technical methods PN-67/A-86430 and PN-A-86431. The pH of the ice cream mixes was measured using a pH meter model CP 505 (Elmetron, Zabrze, Poland). The water activity (aw) of the ice cream mixes was measured using the AquaLab4 water activity meter TEV model (Decagon Devices Inc., Pullman, WA, USA) according to the manufacturer's instructions. The protein content was evaluated by the Kjeldahl method [Varelis 2016].

Ice cream formulations viscosity

The rheological properties of ice cream mixes were studied using a Brookfield RST CC stress rheometer (AMETEK Brookfield, Middleborough, MA, USA) in a cylindrical system (measuring block with a rotational ramp, using a CSR (Controlled Shear Rate) profile, with the following set parameters: shear rate 150÷600 s-1, time 300 seconds, temperature 5.0°C) and the data obtained were presented in a linear distribution and described using Ostwald de Waele and Herschel-Bulkley models. The viscosity of ice cream mixes at a shear rate of 350 s-1 was also determined. Analyses were carried out in triplicate.

Glass transition determination

Differential scanning calorimetry (DSC) was used to determine the glass transition temperature (Tg) of ice cream. The temperature was measured using a Linseis Chip-DSC 100 differential scanning calorimeter (Linseis Inc., Selb, Germany). Samples weighing 16 mg were placed in the 20 μL aluminum crucibles and stored at -35.0°C. Next, they were transferred to a liquid nitrogen cooled calorimeter and kept until the temperature of the samples reached approximately -70.0°C. After starting the measurement, the thermogram was recorded in the range of -70°C to 15°C so that the glass transition and melting process could be observed. The heating rate was 10 degrees per minute.

Texture analysis

In the texture analysis, a CT3 TA texture analyzer (AMETEK Brookfield, MA, USA) was used to determine the hardness [N] and adhesiveness [mJ] of the ice cream according to the procedure described by Tiwari et al. (2015) with modifications. Ice cream samples (250 ml) stored for 7 days at -25.0°C were transferred to a freezer (-10.0°C) and stored overnight. Texture profile analysis (TPA) was then carried out using a 4 mm cylindrical TA44 stainless steel probe under the following conditions: penetration distance 15 mm, force 0.5 N, probe velocity during penetration 3.3 mms-1, probe velocity after penetration 3.3 mms-1. The data obtained were analyzed using TexturePro CT V 1.2 Build 9 software.

Overrun

The ice cream overrun (OR) was evaluated as the ratio of the fixed volumes of ice cream mix and frozen ice cream and calculated using the following equation:

$$OR\ (\%) = \frac{mix\ (g) - ice\ cream\ (g)}{ice\ cream\ (g)} \times 100\%$$

Meltdown test

The ice cream melting test was performed based on the methodology described by Karaca et al. (2009) and Muse and Hartel (2004) with modifications. Ice cream samples stored for 7 days under freezing conditions (-25.0°C) were taken to the experiment. Approximately 20g of ice cream was cut using a cylinder and placed on a metal wire mesh suspended over a glass beaker on a scale and kept at ambient temperature (22.0±1.0°C). For a total of 90 minutes, the melting process of the ice cream was monitored and the amount of melted ice mass was weighed every 10 minutes. The time of the first drop was also recorded and considered as an induction time. The obtained results were presented as the percentage value of the weight of the ice cream drip-through divided by the weight of the ice cream sample. Next the drip-through was plotted against the time (minutes) and the melting rate was calculated from the slope of the linear fitting of the fast-melting phase range.

Ice cream stability and storage

The ice cream produced was stored in a freezer at -35.0°C for 90 days. After this time, the ice cream was analyzed to see if any changes in color, overrun, melting behavior, and texture occurred. The experiment was performed with untreated ice cream and ice cream exposed to temperature variations.

Temperature fluctuations

The effect of temperature fluctuations during frozen storage was investigated by subjecting ice cream to a temporary heat shock procedure described by Markowska et al. (2023). Ice cream samples were taken from frozen storage and allowed to stand at the temperature of 22.0±1.0°C for 60 minutes. Then, the samples were placed back in the freezing conditions (-25.0°C) and stored overnight. This procedure was done twice both for aged ice cream and ice cream stored for 90 days and all the measurements were done in triplicate. Next, the ice cream samples were studied whether any changes in the color, overrun, melting and texture occurred.

Statistical analysis

The experiments were assayed in triplicates and the results were presented as the mean with standard deviation (SD). The statistical analysis was carried out using Statistica® 10.0 PL software (StatSoft Poland Sp. z o.o., Kraków) using a one-way Analysis of Variance (ANOVA) and Tukey HSD Test (α ≤ 0.05).

RESULTS AND DISCUSSION

The present study investigated the effect of the addition of α-tocopheryl acetate (ATA) on the changes in the parameters of ice cream mixes and ice cream after storage in freezing conditions for 90 days, as well as the effect of temperature fluctuations on the quality of ice cream.

The chemical composition of T0, T1, T2 and T3 ice cream formulations differed in the amount of added α-tocopheryl acetate. It was found that the presence of ATA in concentrations ranging from 0.01 to 0.03% did not significantly alter the dry matter content, water activity and energy value (about 659.30 kJ; 157.61 kcal). The pH of the reference ice cream mix (T0 model) was 6.58±0.04, which was significantly lower than that of the α-tocopheryl acetate-supplemented ice cream mix (pH=6.69 approximately) (Table 2).

Table 2. Ice cream formulations' physical parameters

Parameter		Ice cream formulation model			
		T0	T1	T2	T3
dry matter	[%]	32.56±0.17[a]	32.66±0.27[a]	32.51±0.18[a]	32.52±0.19[a]
a_w	[-]	0.9851±0.0004[a]	0.9850±0.0002[a]	0.9846±0.0001[a]	0.9852±0.0004[a]
pH	[-]	6.58±0.04[a]	6.71±0.02[b]	6.68±0.03[b]	6.69±0.04[b]
EV	[kJ]	659.30	659.30	659.30	659.30
	[kcal]	157.61	157.61	157.61	157.61

where: a_w – water activity, EV – energy value; at the value α ≤ 0.05, the differences are statistically significant

Table 3. The results of DSC analysis of the ice cream samples studied

Parameter			Ice cream formulation model			
			T0	T1	T2	T3
Glass transition [°C]		Tg	- 47.28±0.1[a]	- 45.85±0.1[b]	- 45.39±0.2[c]	- 45.00±0.2[c]
		Onset	- 49.36±0.1[a]	- 47.26±0.2[c]	- 46.75±0.1[b]	- 46.94±0.2[bc]
		Off set	- 46.71±0.2[a]	- 44.47±0.1[b]	- 44.34±0.2[b]	- 44.23±0.3[b]
Freezing temperature [°C]		Tf	- 1.74±0.14[a]	- 1.73±0.01[a]	- 1.72±0.09[a]	- 1.73±0.16[a]
Melting reaction	Temperature [°C]	onset	- 8.11±0.20[a]	- 7.53±0.31[ab]	- 7.09±0.12[b]	- 4.15±0.22[c]
		offset	3.49±0.30[ab]	3.11±0.10[b]	3.20±0.21[b]	3.92±0.21[a]
		ΔT	11.60[a]	10.64[b]	10.29[c]	8.07[d]
	Enthalpy [J/g]	ΔH	221.80±6.41[a]	218.70±3.65[a]	217.65±2.89[a]	206.27±2.21[b]

Tg – glass transition temperature; Tf – freezing temperature; ΔH – endothermic enthalpy of the melting; at the value α ≤ 0.05, the differences are statistically significant

As shown in Table 3, the addition of α-tocopheryl acetate slightly changed the studied parameters of the freezing and melting process, although the freezing temperatures did not differ significantly among the tested models. The glass transition range of the reference sample was found to be between -49.36±0.1 and -46.71±0.2°C, with the glass transition temperature (Tg) of -47.28±0.1°C. The addition of the vitamin component slightly increased both the onset and offset temperature ranges, which were observed from -47.26±0.2 to -46.75±0.1°C and from -44.47±0.1 to -44.23±0.3°C respectively. Furthermore, a statistically significant increase in Tg was noted in the fortified samples compared to the reference (Table 3). A similar property, i.e. an increase in Tg, was observed in ice cream with dietary fibre supplementation and in reduced-fat ice cream with inulin [Junyusen et al. 2017; Soukoulis et al. 2009]. Both polysaccharides and proteins can modify the transition from the high viscous-rubbery to the glassy state while the freezing extent is primarily dependent on small solutes as dissociated salts and sugars [Roos 2021]. A well-known glass formers are sugars, including sucrose and lactose which have been intensively studied recently. In presented experiment the content of major ingredients remained unchanged but it did differ with the amount of ATA added. It can, therefore, be concluded that the addition of alpha-tocopheryl acetate affects the transition from the rubbery to the glassy state and also has moderate cryoscopic properties, which improves the storage stability of the ice cream. Still, the typical Tg range of the ice cream, known as the glassy state, varies between -43.0°C and -23.0°C. Below Tg, the samples are stable against recrystallization, molecular movement and unfavorable reactions due to the high viscosity [Goff et al. 1993]. The higher glass transition temperature of the ice cream could therefore be a modelling factor influencing the technological process of ice cream production as well as the quality of the product during frozen storage. Nevertheless, the changes in the glass transition might vary depending on the type of components used in the preparation of ice cream mixes and the interactions between them [Soukoulis et al. 2009; Whelan et al. 2008; Zhang et al. 2016].

Increasing the concentration of ATA in the ice cream formulation resulted in a significant change in the melting reaction parameters, particularly the melting temperature. The onset values for melting temperature ranged from -8.11±0.20 °C (model T0) to -4.15±0.22 °C (model T3), while the offset values remained at relatively similar levels (Table 3). The values for the melting temperature ranges were 10.64, 10.29, and 8.07 for the models T1, T2, and T3, respectively, which were lower than for the control sample (ΔTT0=11.60). It is claimed that the narrow range of ΔT values reflects greater homogeneity of ice crystals distribution, which requires a narrower temperature range for melting [Alvarez et al. 2005; Soukoulis et al. 2009]. Hence, α-tocopheryl acetate supplementation of ice cream improves ice crystal distribution compared to the reference ice cream model.

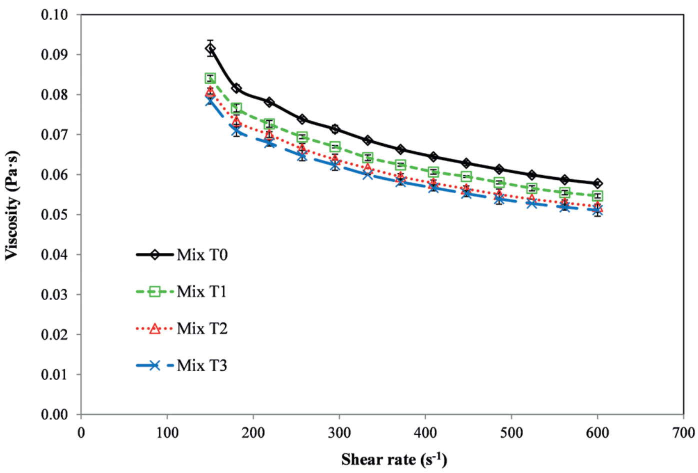

Figure 1. Flow behavior of ice cream formulations fortified with α-tocopheryl acetate at 0% (mix T0), 0.01% (mix T1), 0.02% (mix T2) and 0.03% (mix T3)

Figure 1 presents the results of the rheological analysis of the ice cream formulations studied. The negative correlation between the viscosity and the shear rate, thus the decrease in viscosity under the shear rate, indicated a typical shear thinning behavior of a pseudoplastic non-Newtonian fluid. Our findings are in agreement with literature data on the rheological studies of ice cream formulations [Arellano et al. 2013; De La Cruz Martínez et al. 2020; Goff and Hartel 2013b; Karaman and Kayacier 2012].

Ice cream mixes are colloidal emulsions of several components which undergo rearrangement and exhibit different apparent viscosities and flow resistances when subjected to the shear stress. It was found, that the addition of α-tocopheryl acetate to ice cream mixes changes the rheological parameters of ice cream formulations. According to the data shown in Table 4, the flow behavior of ice cream is better characterized by the Herschel-Bulkley model than by the Ostwald de Waele model, as the stability indices (R2) were higher. The scientific data claim, that the pseudoplastic behavior of ice cream mixes have a flow index (n) below unity. The flow indices in both the Ostwald de Waele and Herschel-Bulkley models were in range of 0.5118-0.5414 and 0.7549-0.7636, respectively, and are in agreement with literature data on different additives [Dertli et al. 2016; Lozano et al. 2022; Zarzycki et al. 2019].

Table 4. Rheological parameters of ice cream mixes with α-tocopheryl acetate

Ice cream mix	Apparent viscosity*	Model						
		Ostwald de Waele			Herschel-Bulkley			
		k	n	R^2	τ_0	k	n	R^2
T0	0.0678 ± 0.0006 a	1.0000 a	0.5414 a	0.9971 a	1.7842 a	0.2619 a	0.7549 a	0.9997 a
T1	0.0636 ± 0.0004 b	1.0000 a	0.5312 b	0.9958 b	1.2596 ab	0.2405 ab	0.7626 a	0.9997 a
T2	0.0606 ± 0.0002 c	1.0000 a	0.5224 c	0.9961 b	1.2457 ab	0.2496 ab	0.7472 a	0.9996 a
T3	0.0593 ± 0.0003 c	1.0000 a	0.5118 d	0.9952 b	1.2142 b	0.2221 b	0.7636 a	0.9996 a

* apparent viscosity measure at 350.00 s^{-1} shear rate; at the value $\alpha \leq 0.05$, the differences are statistically significant

The ice cream mixes with added vitamin component showed a gradual decrease in both yield stress (τ0) and the apparent viscosity when compared to the reference. In general, the yield stress is a parameter that reflects the shear stress required to initiate the flow of the material [Sun and Gunasekaran 2009]. In colloidal systems, the increase in apparent viscosity and yield stress values is associated with the increase in both particle volume fraction and interparticle forces and the decrease in particle size [Genovese et al. 2007]. This is of particular interest in food technology, especially in terms of material flow within the industrial installation systems. In our study, the addition of α-tocopheryl acetate in an oily form seemed to significantly affect the values of both τ0 and apparent viscosity. The gradual decrease of these parameters can be related to improved flowability of ice cream formulations and as a consequence could lead to better scoopability of ice cream [Sun and Gunasekaran 2009; Trivana et al. 2023].

The structure and texture of ice cream is defined by several parameters including the viscosity, size and amount of ice crystals present and the overrun which is a quantity of air entrapped in the frozen ice cream mass. The overrun is reported to affect the consistency of ice cream and its melting properties [Wu et al. 2019]. In the presented study, ice cream overrun was studied after frozen storage and after the thermal treatment applied. The obtained results are presented in Figure 2.

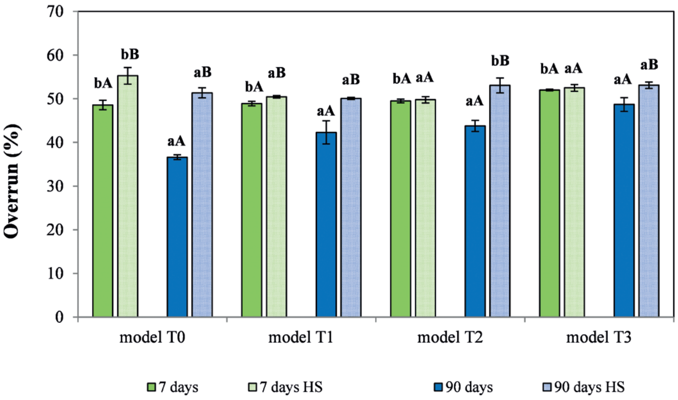

Figure 2. The results for ice cream overrun after production, frozen storage, and temperature fluctuations; HS – heat shock

* A, B – the average values with different letters (within the same model) of different storage times are significantly different at α ≤ 0.05)

a, b – the average values with different capital letters (within the same model) of different temperature conditions applied are significantly different at α ≤ 0.05

After seven days of storage, all the samples tested, both the reference and those supplemented with the vitamin component, showed similar average overrun values ranging between 48.55±1.08% to 51.99±0.20%. The highest overrun was observed in ice cream with 0.03% α-tocopheryl acetate (T3), which could indicate the formation of smaller bubbles during the freezing process. The 90-day frozen storage resulted in the significant overrun decrease in ice cream samples, especially those with no added vitamin source (T0). According to the literature, a phenomenon of overrun decrease is often observed in several types of ice cream which are kept under different storage times [Güzeler et al. 2012; Murtaza et al. 2004; Singh et al. 2014]. This could suggest that the storage time is more relevant factor than, for example, the ratio of stabilizers and emulsifiers [Güzeler et al. 2012]. The results of this research may indicate that the addition of α-tocopheryl acetate may act as an agent to facilitate the overrun stability during frozen storage.

The study of thermal fluctuations on ice cream overrun showed ambiguous results. The overrun of all the samples showed a similar level, in the range of 49.76±0.74 - 55.25±1.91%, regardless of the time of storage or the application of temperature shocks (Figure 2). The reference ice cream and ice cream with 0.01% α-tocopheryl acetate stored for 7 days showed higher overrun values by approximately 13.80 and 3.20% respectively, after the heat shock was applied when compared to untreated samples. The overrun of ice cream with 0.02 and 0.03% ATA added (T2, T3) stored for one week seemed affected by the thermal treatment in a negligible extent. Ice cream samples subjected to 90 days of frozen storage were characterized by the overrun ranging from 36.60±0.54.76 to 48.68±1.52%, which was lower than in samples subjected to thermal stress. Such phenomenon, i.e. the increase in overrun, could probably be due to the physical changes in the ice cream structure combined with measurement difficulties. Increasing the storage temperature causes the ice crystals to melt and consequently intensifies the mobility of unfrozen water. In addition, the clustering of air cells and their migration to the outer layers of the ice cream are observed [Sofjan and Hartel 2004]. The significant effect of the temperature variations is often observed as changes in the appearance of the ice cream, particularly at the edges and on the surface as well as ice cream shrinkage [Dubey and White 1997; Lomolino et al. 2020]. The visible changes include both a denser structure and the effect of recrystallisation - the presence of larger ice crystals. Therefore, the area of the sample to be taken for overrun analysis is critical, otherwise it will impede the final results. Nevertheless, substances added to ice cream formulations that have stabilizing or cryoprotective properties can facilitate the storage of ice cream [Leducq et al. 2015; Lomolino et al. 2020; Regand and Goff 2003]. In the present work, the addition of α-tocopheryl acetate appeared to have a moderate preservative effect on the shelf life of ice cream in terms of changes of the overrun during frozen storage. In samples with ATA in 0.03% added, the overrun after 90 days of storage was only 3.31% lower than in samples stored shortly after the production. On the contrary, the overrun of the reference ice cream was almost 12.00% lower after 3 months of frozen storage. Unfortunately, due to the inconclusive results obtained for ice cream exposed to thermal fluctuations, additional studies are required to verify whether ATA could have at least a weak cryoprotective effect.

The melting behavior of ice cream studied is presented as melting curves in **Figure 3**. After 7 days of storage, the reference sample (T0) showed the most intense melting. The first drop was observed after 13-15 minutes of the analysis and, after 90 minutes, the amount of drip-off material reached approximately 80.00%. The samples with added α-tocopheryl acetate (T11, T2, T3) started to melt after 15-16 minutes and the average amount of drip-off was in the range of 71.88 to 72.97%. The noted shift in melting curves probably results from thermal properties of ice cream as the melting temperatures of ice cream with ATA added were higher than in the reference, according to DSC analysis. The melting curves of samples T1, T2, and T3 did not show any significant differences (Figure 3). Similar results were obtained after 90 days of storage. The amount of drip-off collected was in the range of 66.55 - 77.32% for samples T0, T1 and T2. In the ice cream with 0.03% ATA, only 56.60% drip-off was collected. In addition, all the ice cream samples started to melt earlier, i.e. after 13-14 minutes, which could be seen as a shift in their melting curves.

The application of thermal fluctuations had a significant effect on the melting behavior of ice cream, independent of the storage time and the concentration of vitamin component. The majority of the samples were characterized by a slightly shorter time for the appearance of the first drop but, unfortunately, no clear trend was observed. The amount of the drip off material was smaller than in the untreated samples. The ice cream mass of freshly prepared samples was observed to melt less dynamically and drip off at approximately 40.00 – 46.50%. More visible differences were observed in samples stored for 90 days and subjected to thermal stress. The average drip off of the reference sample reached 53.27%. In ice cream supplemented with ATA, the average amount of dripped material was equal to 52.95, 44.55 and 44.34% in ice cream T1, T2 and T3 respectively. The results showed that the higher concentration of α-tocopheryl acetate added, the less ice cream mass melts and drips off. This suggests that ATA addition might exhibit slightly stabilizing effect on ice cream structure

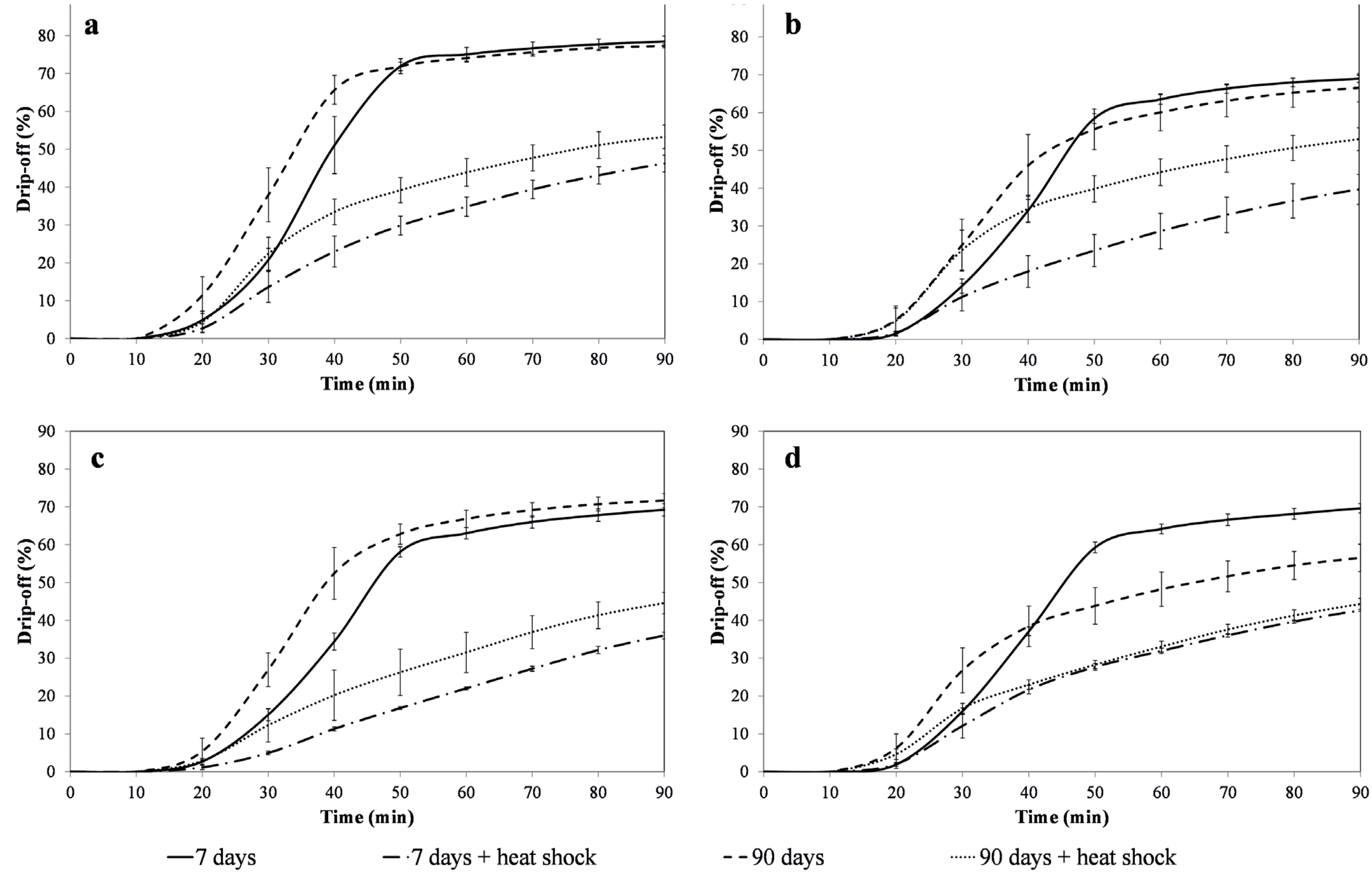

Figure 3. Melting curves of ice cream models studied before and after the thermal treatment; a – model T0 (reference), b – model T1 (0.01% ATA), c – model T2 (0.02% ATA), d – model T3 (0.03% ATA)

in terms of storage and temperature fluctuations. In general, the melting process was observed to be less dynamic after the frozen storage whereas the thermal shocks applied resulted in decrease of ice cream melting rates (Table 5).

Table 5. The comparison of melting rate and chosen texture parameters in ice cream (fresh, stored and subjected to thermal stress)

		Melting rate		Hardness, T [N]		Adhesiveness, A [mJ]	
Ice cream model*	Days	before heat shock	after heat shock	before heat shock	after heat shock	before heat shock	after heat shock
T0	7	2.31 ± 0.18^{yX}	0.91 ± 0.10^{xX}	2.42 ± 0.37^{aA}	2.01 ± 0.39^{aA}	2.58 ± 0.33^{gG}	2.38 ± 0.25^{gG}
T1		1.91 ± 0.04^{yY}	0.72 ± 0.13^{xX}	3.13 ± 0.40^{bB}	2.07 ± 0.53^{aA}	2.77 ± 0.21^{gG}	2.83 ± 0.41^{hG}
T2		1.86 ± 0.56^{yX}	0.53 ± 0.02^{xX}	2.31 ± 0.42^{aA}	2.24 ± 0.46^{aB}	2.85 ± 0.45^{gG}	2.70 ± 0.24^{gH}
T3		1.93 ± 0.86^{yY}	0.86 ± 0.05^{xX}	2.18 ± 0.46^{aA}	1.91 ± 0.56^{aA}	2.72 ± 0.42^{gG}	2.58 ± 0.62^{gH}
T0	90	2.09 ± 0.15^{yX}	1.15 ± 0.13^{xX}	2.41 ± 0.46^{aA}	2.00 ± 0.18^{aA}	3.20 ± 0.33^{gG}	2.52 ± 0.32^{gG}
T1		1.72 ± 0.08^{yX}	1.16 ± 0.12^{xY}	2.33 ± 0.16^{aA}	1.59 ± 0.20^{aA}	2.43 ± 0.27^{gG}	2.13 ± 0.48^{gG}
T2		1.98 ± 0.10^{yX}	0.78 ± 0.21^{xX}	2.22 ± 0.22^{bA}	1.50 ± 0.20^{aA}	2.23 ± 0.52^{gG}	2.28 ± 0.32^{gG}
T3		1.25 ± 0.02^{yX}	0.77 ± 0.08^{xX}	2.02 ± 0.07^{bA}	1.45 ± 0.27^{aA}	2.35 ± 0.33^{hG}	1.82 ± 0.34^{gG}

** a, b, g, h, x, y – values with different letters within the rows (same storage time) are significantly different (α < 0.05) A, B, C, G, H, X, Y – values with different capital letters within the columns (same temperature treatment) are significantly different (α < 0.05)*

Meltdown is considered as one of the most crucial characteristics of ice cream. It is determined by the amount of air in the ice cream mass (overrun), the presence and size of ice crystals and the partially coalesced fat globules, and any modification in the composition of the ice cream formulation may affect the microstructure and thus the physicochemical parameters of the ice cream [Da Silva Junior and Lannes 2011; Daw and Hartel 2015; Muse and Hartel 2004; Wu et al. 2019]. High quality ice cream tends to melt slower than low quality ice cream which is less resistant to the temperature fluctuations and the heat shock. Several studies claim that the higher melting rate occurs in ice cream with lower overrun due to increased heat transfer through air bubbles [Alizadeh et al. 2014; Lomolino et al. 2020; Sofjan and Hartel, 2004; VanWees et al. 2019; Warren and Hartel 2018]. In addition, the heat transfer reduction and the melting rate decrease can be facilitated by a uniform distribution of both air bubbles and small ice crystals [Liu et al. 2023; Muse and Hartel 2004; Park et al. 2015]. Our results for the samples frozen-stored for 90 days are in agreement with the literature data on ice cream melting behavior. Rapid melting is also associated with low freezing point of ice cream formulations [Goff 2002; Goff and Hartel 2013a]. However, as the DSC analysis showed that all ice cream variants tested (T0, T1, T2, T3) showed negligible differences in their freezing points, this parameter was not determinant for their melting rates. Dairy ice cream is composed of components (fats, proteins) and melt more slowly than plant-based ice cream or ice cream supplemented with inulin [Junyusen et al. 2017; Lomolino et al. 2020; Ohmes et al. 1998]. Currently ongoing researches has shown the possibility of limiting the melting process by supplementing the ice cream with various available components such as hydrocolloids, mushroom powder, Moldavian balm, grape wine lees, chia seed mucilage, or microencapsulated components of plant-origin [Feizi et al. 2021; Hwang et al. 2009; Kozłowicz et al. 2021; Shamshad et al. 2023; Tsai et al. 2020]. Still, it is claimed that meltdown is likely to be affected by the temperature, i.e. heat transfer, and the storage conditions [Lomolino et al. 2020; Park et al. 2015]. In the present study, the application of heat shocks significantly changed the melting behavior of ice cream, but the ice cream supplemented with ATA showed slower melting rate and less drip-off mass were observed than in the reference sample (T0).

Several studies reported that ice cream texture, the hardness in particular, is affected by miscellaneous factors. Most often mentioned are formulation composition (fats content, emulsifiers, carbohydrate source, additives), ice crystal size and ice cream overrun, however, literature data are ambiguous [Abd El-Rahman et al. 1997; Muse and Hartel 2004; Prindiville et al. 1999; Sofjan and Hartel 2004; Syed et al. 2018]. The results of ice cream hardness and adhesiveness analysis are presented in Table 5. Ice cream which was not supplemented with vitamin component characterized with similar hardness after the 7- and 90-day storage time which decreased when the thermal stress was applied. In samples with α-tocopheryl acetate added, thermal treatment exhibited similar effect, however, it was observed that the higher ATA concentration the lower the hardness of ice cream. Surprisingly, in freshly prepared T1 ice cream samples, this parameter was significantly higher than in other samples. This, however, could be an error resulting from measurement difficulties, especially since in T1 samples stored for 90 days such a difference was not recorded. Freshly prepared ice cream with vitamin component had a higher adhesiveness than the reference ice cream (Table 5). In the samples subjected to freezing storage, the adhesiveness was lower in all the samples tested except T0 ice cream, regardless the concentration of vitamin preparation added. This suggests an instability of this parameter during storage. In general, the application of heat shock caused a decrease in the adhesiveness of the ice cream. Only ice cream with 0.01% ATA (fresh ice cream) and ice cream with 0.03% ATA (90 days storage) showed significant differences in the results.

Ice cream harness depends on several properties, however, the contradictory results concerning the air content, ice crystal size and the degree of fat destabilization are reported in the literature [Liu et al. 2023; Muse and Hartel 2004; Prindiville et al. 1999; Sakurai et al. 1996; Sofjan and Hartel 2004]. However, supplementation with other components might also affect the ice cream hardness which could be perceived by the consumers as firmness or gumminess. In the study by Akalın et al. (2018), ice cream fortified with different dietary fibers expressed differentiated hardness values which, on the contrary to our study, increased during time of frozen storage. The authors claim, that the gelling ability of dietary fibers and binding water molecules contribute to higher hardness what was observed in other study as well [Crizel et al. 2014]. Still, the potential effect of the added ingredients should be taken into account when designing new frozen dairy products, as too much hardness can have an undesirable effect on the texture of the ice cream [Dar and Light 2014

CONCLUSIONS

The objective of the study was to evaluate the effect of the addition of alpha-tocopheryl acetate (ATA) on the physicochemical parameters of dairy ice cream, as well as on the overrun, melting behavior and texture of the ice cream stored in frozen conditions and subjected to thermal fluctuations. The results obtained indicate that ATA influences some of the ice cream properties. The addition of ATA was found to increase the glass transition temperature and the onset temperature of the melting process. This could indicate that the addition of the vitamin preparation affects the transition from the rubbery to the glassy state and expresses a moderate preservative effect. Therefore, the storage stability of the ice cream both in terms of storage and temperature fluctuations, could potentially be improved. This was observed in the further part of the study concerning the meltdown analysis and the determination of the melting rates of the ice cream. The samples with a higher concentration of α-tocopheryl acetate were characterized by a lower amount of ice cream mass drip-off. On the basis of the calculated melting rates, it could be concluded that the melting process was generally less dynamic after the frozen storage and that the thermal shocks resulted in the decrease in ice cream melting rates. Moreover, the addition of the vitamin component caused the decrease in the hardness of the ice cream, which could be considered as the improvement, in sensory characteristics in particular. Unfortunately, the overrun analysis showed inconclusive results, especially for the ice cream exposed to thermal fluctuations. Therefore, in order to investigate the precise mechanisms by which ATA influences the ice cream stability during frozen storage and to verify whether the addition of ATA could show a protective effect on the ice cream overrun, further research is required. Furthermore, additional researches could possibly provide valuable data on vitamin components supplementation to ice cream in order to design functional food with health promoting properties.

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