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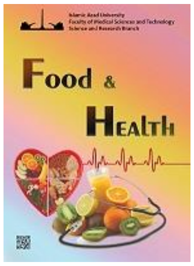

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Effects of cooling rate and aging process on crystallographic structure, whipping, rheological, textural, and thermal properties of frozen minarine

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ABSTRACT

Minarine is a cream obtained by mixing animal cream and vegetable oils. Fat crystallization is the main stage in the production of this product and affects its mouthfeel, stability, texture, and appearance. Processing conditions influencing the fat crystallization, partial coalescence, and finally, physical and structural properties of whipped cream are heat treatment (pasteurization and sterilization), homogenization, cooling rate, aging process, tempering and temperature, time and speed of whipping. This study aims to characterize the fast-cooling effects of an ice cream maker to 5 °C and an aging process at this temperature for 24 hours on frozen minarine's whipping, rheological, textural, crystallographic, and thermal properties. Results illustrated that the most desirable whipping properties (overrun=114.8% and syneresis=3.6mm) and the highest rheological and textural properties, γLVR (0.33), G' (40850 Pa) and firmness (642 g), belonged to sample FCA due to the formation of a denser crystalline network resulting from fast cooling and the aging process. Wide-angle X-ray scattering spectra show that α-crystals were mainly formed upon fast cooling. Then, a transition from α to β'-crystals occurred during the aging process. Also, according to differential scanning calorimetric results, the endothermal peak temperature was shifted to higher temperatures due to fast cooling and aging processes.

1. Introduction

Whipping cream is an oil-in-water (O/W) emulsion containing water, milk, and its derived products in the aqueous phase, as well as permitted edible oils or fats in the oil phase. This type of cream typically contains 30-40% fat content and can be whipped up to twice its volume and form firm and stable foam. The aeration process destroys the oil-in-water emulsion structure of whipping cream and forms a colloidal foam in which the dispersed phase is air, and the continuous phase is water (1, 2). Traditionally, whipping creams are made from milk fat, but recently, creams based on vegetable oils have gained more market share due to high flexibility and low prices. Minarine is a cream obtained from animal cream and vegetable oils (3). Because the fat content of this product is high, its stability and physicochemical properties are intensely affected by the fat phase. Generally, fat crystallization is the main stage in the production of this product and affects its texture, stability, mouthfeel, and appearance (4). The various forms of fatty acid crystals, called polymorphisms, include alpha, beta, and beta-prime crystals. From the point of view of triglyceride molecules packing in the crystal network, α, β', and β crystals are hexagonal, orthorhombic, and triclinic, respectively. The α crystals, which are very tiny and transparent particles with an amorphous morphology, have the least stability and the lowest density and melting point. The β´form with rectangular morphology is a bit greater than α crystal, metastable, and possesses intermediate density and melting point. The β crystal with needle-shaped morphology and coarse appearance is the most stable form and has the

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highest density and melting point. This type of crystal tends to grow into clusters that can lead to a grainy texture in the fat phase. In addition to these three main crystals, there is a gamma crystal with a glassy state. The life of this crystal is concise, and it quickly turns into α crystal (3, 5). In the whipping cream, the β' crystal is desirable due to its size, its ability to physically trap large amounts of triacylglycerol (TAG) molecules with lower melting points in the crystal lattice and increase the partial coalescence rate of fat globules (3). The fat crystal lattice properties rely on the interactions among the crystals. The size, shape, and polymorphism of crystals, dependent on the chemical composition of fats and processing conditions, can influence these interactions. Processing conditions influencing the fat crystallization, partial coalescence, and finally, physical and structural properties of whipped cream are heat treatment (pasteurization and sterilization), homogenization, cooling rate, aging process, tempering and temperature, time and speed of whipping (6, 7). As a result of different cooling rates, crystals with different sizes are formed. Fast cooling leads to the formation of a large number of small fat crystal lattices with uneven and rough surfaces, while in slow cooling, a small number of large fat crystal lattices with a smoother and more homogeneous surface are formed. Rough surfaces resulting from fast cooling with a large number of protrusions that act as active points for partial coalescence, lead to increase bonding efficiency and the production of more compact aggregates and consequently increase firmness (8, 9). Upon the fast cooling, α crystals are formed first, and then a transition from unstable crystals to more stable β' crystals occurs during the aging process at refrigerator temperature. In slow cooling, β' crystals are formed from the beginning (7, 10). Whipping cream should be stored at low temperatures (4-6 ºC) for a relatively long time (24h) to form suitable crystals in the fat structure. This leads to better aeration of the product during the aging process. During this process, proteins and stabilizers are hydrated, emulsifiers are rearranged, and fat crystallization occurs (11). The liquid phase causes the triglycerides to dissolve from the unstable crystals and recrystallize them into more stable and pure crystals. This phenomenon occurs during the aging process at refrigerator temperature at which the whipping cream is still liquid and leads to conversion of the unstable α crystals to the more stable β' crystals (12). The aging process causes a significant increase in the firmness and elastic modulus of the samples. This is attributed to the formation of a more compact crystal lattice over time and also higher number of contact points (13). The aim of this study is to investigate the effects of fast cooling by ice cream maker to 5 ºC and aging at this temperature for 24 hours, on whipping, rheological, textural, crystallographic and thermal properties of frozen minarine.

2. Materials and methods

2.1. Materials

Minarine samples were supplied by Koolak Company (Iran). This pasteurized and homogenized product contains 16% shortening, 2.6% cocoa butter substitute, 17% animal cream with 40% fat, 1.3% animal butter, 6.5% skim milk, 26% sugar, 30.4% water, and 0.22% stabilizer.

2.2. Fast cooling and aging process

After homogenization, minarine samples were subjected to different cooling rates and aging processes. The various treatments were: fast cooling by ice cream maker (BREVILLE) to 5 ºC and aging process at this temperature for 24 hours, then freezing (FCA), slow cooling at refrigerator conditions to 5 ºC and aging process at this temperature for 24 hours then freezing (SCA), fast cooling by ice cream maker to 5 ºC and then freezing without the aging process (FC) and freezing the sample without pre-cooling and aging process in accordance with the industry (BS or control sample).

2.3. Whipping operation

Thermal and crystallographic examinations were performed on frozen minarine samples before the whipping operation, but whipping, textural, and rheological examinations were performed on whipped samples. Before whipping, the frozen minarine samples were taken out of the freezer and kept at room temperature for 20 minutes until partial softening. Then, the whipping operation by a mixer (Kitchen Aid/America) was accomplished with a specific program of 5 minutes at the low speed of the mixer, 15 minutes at medium speed, and 2 minutes at high speed. Experimentally, these conditions lead to the highest overrun in the tested samples.

2.4. Measurement of overrun

The same volume (100 ml of minarine) is poured into the beaker before and after whipping by a mixer and then weighed with a scale. The overrun percent was calculated according to the following equation (Eq. (1)):

$$\%\ Overrun = \frac{M_1 - M_2}{M_2} \times 100 \qquad (1)$$

Where, M_1 (g) is the weight of 100 ml of minarine before whipping, and M_2 (g) is the weight of 100 ml of whipped minarine (14).

2.5. Measurement of syneresis

A certain amount of whipped minarine was placed on the glass filter fixed above a 100mL erlen. The erlen was placed in an oven with a temperature of 15-18°C and relative humidity of 75%, and after two hours, the amount of serum removed was measured in millimeters.

2.6. Rheological properties

Rheological measurements of whipped minarine samples were performed using a Physica MCR 301 rheometer (Anton Paar, Austria). For temperature setting, the measuring device was equipped with a Peltier system assisted by a fluid circulator. The parallel plate geometry with a 40 mm diameter and gap of 1 mm was used. After loading, the samples rested for 1 minute to achieve temperature equilibrium, eliminate induced stress, and recover the original structure. All rheological tests were performed at 5 ± 0.01°C. At first, a strain sweeps test (0.01–600%) was done at a fixed frequency of 1 Hz to detect the linear viscoelastic region. The recorded parameters were the strain corresponding to the end of the linear viscoelastic range (γ_{LVR}), the elastic modulus in LVR (G'_{LVR}), and the viscose modulus in LVR (G''_{LVR}). Frequency sweeps test was performed at the constant strain of 0.01% and the frequency range of 0.01–100 Hz to expose the frequency dependence of minarine samples. The elastic modulus (G') was modeled as a power function of angular Frequency (ω, rad/s) to calculate intercept (A), slope (b), and R^2 using the Bohlin equation (Eq. (2)).

$$G' = A \times (\omega)^b \tag{2}$$

Rheoplus software version 3.21 (Anton-Paar) was applied to gather experiment data (15).

2.7. Textural analysis

The textural properties of the whipped minarine samples were analyzed by Texture Analyzer TA-XT (Stable Micro Systems, TA. XT Plus, UK) with a Back extrusion test. A cylindrical probe (diameter: 35mm) attached to a 5 kg load cell was applied to compress the samples. The whipped minarine samples are poured into the chamber and placed under the probe in this test. Then, the probe, smaller than the diameter of the chamber, enters the sample at a certain speed, and the sample comes out of the side of the container. Then, the probe starts moving in the opposite direction and leaves the sample. Samples were compressed to 30% of height at a rate of 10 mm/min, and firmness, consistency, viscosity, and cohesiveness were measured (16).

2.8. X-ray diffractometer (XRD) analysis

Polymorphism of fat crystals was determined via X-ray diffractometer (XRD). This technique is used to identify existing crystalline phases and their orientation. The frozen minarine samples were placed in the sample holder and irradiated with an angle of 7 to 50 degrees and a scan rate of 2 degrees per second. The results were evaluated using Bragg's law (13).

2.9. Differential scanning calorimetry (DSC) analysis

To investigate the thermal behavior, 5-10 mg of the samples was placed in the DSC pan, and an empty pan was used as a reference. The minarine samples were heated from 0°C to 60°C at a heating rate of 5 °C/min. The melting temperature of the samples is determined from the obtained curves (17).

2.10. Statistical analysis

The statistical analysis was performed by analysis of variance method (the one-way ANOVA $p < 0.05$) using the SPSS, version 21, and Duncan's multiple-range test was applied to determine significant differences between means.

3. Results and discussion

3.1. Overrun

The overrun percent of samples is mentioned in Table 1. The statistical analysis of the results showed a significant difference between the overrun of minarine samples under the different cooling rates and aging processes ($p_{value} < 0.05$). The fast cooling with the ice cream maker before freezing (FC) also, slow cooling at refrigerator conditions, and the aging process at this temperature for 24 hours (SCA) led to a significant increase in overrun compared to the control sample (BS), which was frozen directly.

Table 1. Whipping properties (overrun and syneresis) of whipped minarine samples.

Sample names	Overrun (%)	Syneresis (mm)
BS	88.29±2^{d*}	7.3±0.09^{a}
SCA	103.90±2^{b}	5.4±1.00^{b}
FCA	114.80± 3^{a}	3.6±0.09^{c}
FC	99.74±1^{c}	6.7±0.08^{a}

*Values in each column with different letters are significantly different ($p<0.05$). Data are presented as mean ± standard deviation.

This phenomenon is attributed to the fat globule's partial coalescence enhancement due to the fast cooling and aging process. The effect of the aging process was more than that of fast cooling due to the formation of β' crystals (8). The highest overrun percent (114.8%) belonged to sample FCA. In this sample, α crystals are formed first, then during the aging process, the crystallization of fats increases, and a transition from unstable crystals to more stable β' crystals takes place. Consequently, a dense and compact lattice of β' crystals is formed, leading to increased connection points for partial coalescence (3, 8, 18). Therefore, the desired and optimal overrun content can be achieved by applying such thermal pretreatment. The optimal and desired amount of overrun in confectionery cream has been reported in the range of 120-115% by air volume.

3.2. 3.2. Syneresis

Serum loss in whipping cream shows emulsion instability. As demonstrated in Table 1, syneresis values of minarine samples were significantly different under the various cooling rates and aging processes ($p_{value} < 0.05$). According to the results, the highest amount of serum loss (7.3 ml) was related to the control sample (BS), and the lowest amount of serum loss (3.6 ml) belonged to the FCA sample, followed by the SCA sample, which has been subjected to the aging process. These results demonstrated that the aging process enhances emulsion stability and reduces serum loss. This can be attributed to increased fat crystallization and partial coalescence, leading to the formation of ordered structures and enhancement of viscosity (1, 9, 18). It is better to see no serum loss in the whipped cream. Regarding this, an appropriate and optimal amount of serum loss in confectionery cream has been reported in the range of 0-4 ml. Based on this, the FCA sample had a proper serum loss.

3.3. Rheological properties

Rheological attributes are one of the most important aspects to control emulsion-based food products' physical stability and shelf life. Strain sweeps and frequency sweeps are standard rheological tests performed on confectionery cream.

3.3.1. Strain sweeps rheological properties

The strain sweep test parameters of minarine samples are shown in Table 2. Up to the critical strain (γ_{LVR}), the structure of materials remains unchanged, while above this point, the structure starts to break and decompose. Therefore, this test determines the reversible deformation range of matter. In the case of whipped cream, the longer the LVR, the greater the resistance to whipping (14).

Table 2. Strain sweeps test parameters of whipped minarine samples.

Sample names	G'_{LVR} (Pa)	G''_{LVR} (Pa)	γ_{LVR} (%)
BS	22675±198^{d}	5215±72^{d}	0.10±0.000^{c}
SCA	36484±311^{b}	8026±44^{b}	0.22±0.010^{b}
FCA	40850±203^{a}	8578±93^{a}	0.33±0.000^{a}
FC	28902±241^{c}	6358±35^{c}	0.10±0.002^{c}

Values in each column with different letters are significantly different ($p < 0.05$). Data are presented as mean ± standard deviation.

In this study, the highest γLVR value (0.33%) belonged to the FCA sample, followed by the SCA sample. These results were consistent with the results of the overrun test. As previously mentioned, the FCA and SCA samples had the highest overrun values. Long et al. (14) also stated that the sample with longer LVR had greater whippability, leading to more overruns. In all the whipped minarine samples, the elastic modulus was more than viscose modulus (G′ > G’’) in linear viscoelastic region, revealing the domination of elastic behavior. The G′ values of treated samples were significantly more than that of the control sample (BS) ($p_{value} < 0.05$). It’s worth noting that a higher elastic modulus is desirable for confectionery cream, especially when used for decoration, which can keep its shape. The FCA and SCA samples showed the highest G′ values. Since both samples have been subjected to an aging process, their observed difference is attributed to their cooling rate. Rough surfaces of fat crystal lattices resulting from fast cooling provide a large number of connection points, leading to enhancement of partial coalescence, production of more compact aggregates, and consequently increment of G′ value (8). These results are in agreement with the work done by Wiking et al. (19), who reported that the complex modulus of fast-cooled milk fat was higher than that of slow-cooled milk fat. Although the FC sample was subjected to the fast cooling process, it exhibited significantly less G′ value than the FCA sample ($p_{value} < 0.05$). This difference is attributed to the effect of the aging process on minarine elastic modulus. Also, similar results were found in the study conducted by Ronholt et al. (13) on the effects of cream cooling rate and aging process on polymorphism, microstructure, and rheological properties of butter. The authors reported that the aging process of cream led to the significant increase in firmness and elastic modulus of slow cooled butter samples.

3.3.2. Frequency sweeps rheological properties

After the strain sweeps test and determination of LVR, the G′ and G″ were assessed as a function of angular Frequency. In all the samples and over the entire frequency range, the G′ was more than G’’, indicating a whipped minarine solid-like structure. Also, the variations slope of G′ curve was greater than that of G″ curve, and the G″ was almost constant and did not depend much on the frequency changes (Fig. 1).

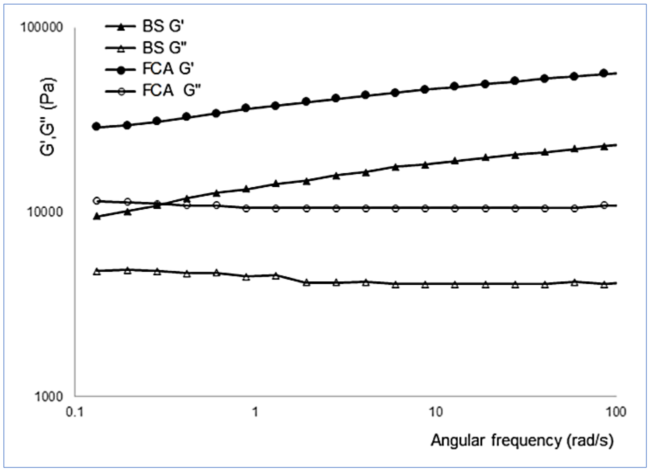

Fig. 1. Frequency sweeps test of FCA and BS samples.

With increasing Frequency, the G′ increased and elastic behavior prevails over viscose behavior. Jakubczyk and Niranjan also reported a similar result (20). The Bohlin model fitted the dependency of G′ on Frequency with a determination coefficient of R^2 >0.97. The parameters corresponding to this model are mentioned in Table 3. Based on the Bohlin model, the value of A is the measure of structural strength between rheological units. The value of b indicates the interconnected rheological units in the three-dimensional emulsion network and the emulsion frequency dependence (21). The results showed that the lowest value of b (0.09) and the highest value of A (23361 Pa.s rad^{-1}) belonged to the FCA sample. This is related to the formation of a large number of crystals during fast cooling and increasing fat crystallization during the aging process, enhancing the connection points between fat globules and the consistency (18).

Table 3. Frequency sweeps test parameters of whipped minarine samples.

Sample names	A (Pa s rad^{-1})	b	R^2
BS	15510±95^{d*}	0.13±0.00^{a}	0.98
SCA	22791±178^{b}	0.12±0.01^{b}	0.97
FCA	23361±228^{a}	0.09±0.01^{c}	0.97
FC	21259±231^{c}	0.12 ±0.00^{b}	0.98

*Values in each column with different letters are significantly different ($p < 0.05$). Data are presented as mean ± standard deviation.

3.4. *Textural properties*

The back extrusion test determines firmness, consistency, viscosity, and cohesiveness. Firmness or maximum force is the last point where the probe sinks and wants to start moving in the opposite direction. It is interesting to note that whipped cream with higher firmness value can be decorated well and is also resistant to flow. As presented in Table 4, the cooling rate and aging process significantly affected the textural properties of whipped minarine samples. In accordance with the rheological characteristics, the FCA sample showed the highest values in terms of textural parameters. As formerly mentioned, the formation of a large number of small crystals with rough surfaces during fast cooling and increasing of fat crystallization during the aging process lead to enhanced partial coalescence of fat globules and, consequently, the formation of dense and compact fat networks, which cause enhancement in textural properties (9, 18).

Table 4. Textural properties of whipped minarine samples.

Sample names	Firmness (g)	Consistency (g.s)	Viscosity (g.s)	Cohesiveness (g)
BS	576±11^{c*}	13880±323^{c}	609±19^{c}	1344±44^{c}
SCA	597±4^{b}	14324±198^{b}	630±16^{b}	1569±78^{b}
FCA	642±19^{a}	15402±306^{a}	709±30^{a}	1673±49^{a}
FC	589±17^{bc}	14608±460^{b}	642±32^{b}	1539±38^{b}

*Values in each column with different letters are significantly different (p
Data are presented as mean ± standard deviation.

3.5. *Polymorphism*

The polymorphism of fat crystals was identified by wide-angle X-ray scattering (WAXS). This technique is based on the fact that the X-ray diffraction pattern of each crystalline material is unique. Several plates or rows with different distances (d-space) can be considered for each crystal. Bragg's law is based on the distance between the crystal plates (d-space). Each crystal has one or two d-spaces. The d-space of α crystal is 4.15, β́ Crystal is 3.8 and 4.2, and the β crystal is 4.6. As displayed in Fig. 2, the FCA sample had an apparent peak at d-space corresponding to β́ crystal.

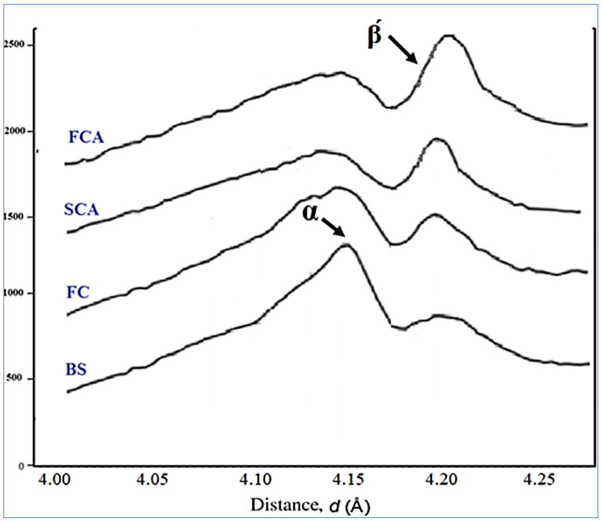

Fig. 2. Fat crystals polymorphism of frozen minarine samples.

This phenomenon can be explained by the fact that, upon the fast cooling, α crystals were formed first, and then a transition from unstable crystals to more stable β' crystals took place during the aging process at refrigerator temperature. While samples without the aging process (FC and BS) showed a clear peak at d-space corresponding to α crystal (Fig. 2). This is due to the quick solidification of these samples at freezing temperature and reduction of mobility, which leads to preventing the transition of the unstable α crystals to the more stable β' crystals. These results agree with the study done by Fredrick et al. (7) on the investigation of the isothermal crystallization behavior of milk fat in bulk and emulsified state. Whereas in a cooled sample (SCA), β' crystals were formed from the beginning, consistent with the previous studies (10).

3.6. *Thermal properties*

The results of the thermal analysis of treated minarine samples and the control sample are demonstrated in Fig. 3. According to these results, the fast cooling and aging process led to a shift in the endothermal peak temperature to higher temperatures in treated samples compared to the control sample. During the aging process, the fat crystals had enough time to rearrange and form a dense and ordered network, leading to an increase in the melting point of FCA and SCA samples compared with other samples.

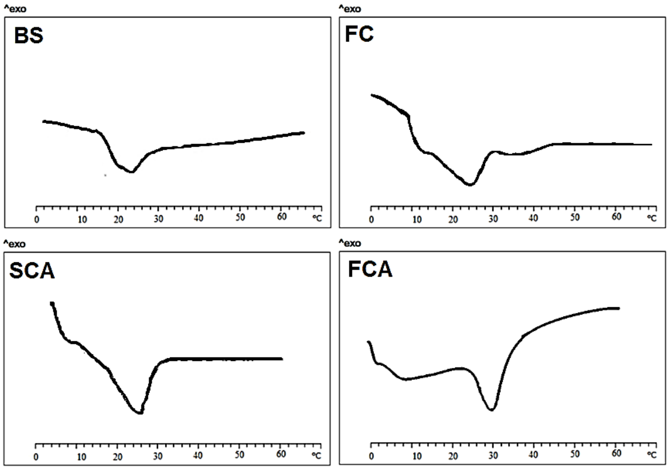

Fig. 3. Thermal profile of frozen minarine samples.

Also, the transition of the unstable α crystals to the more stable β' crystals during the aging process caused a shift in the endothermal peak temperature to higher temperatures in these samples (8, 12). The highest endothermal peak shift compared to the control sample belonged to the FCA sample. Similar results were also reported by Truong et al. (22) and Tippetts and Martini (17). The difference observed in the melting behavior of fast and slow-cooled emulsions is attributed to the formation of composite crystals during fast cooling.

4. Conclusions

Results illustrated that thermal pretreatment of fast cooling by ice cream maker to 5 ºC and aging process at this temperature for 24 hours were significantly effective on whipping, textural, rheological, crystallographic, and thermal properties of minarine samples. The most desirable whipping properties (for example, the highest overrun and the lowest syneresis) and the highest rheological and textural properties (such as γLVR, G', and firmness) belonged to the FCA sample due to the formation of a denser crystalline network resulting from fast cooling and aging process. Wide angle X-ray scattering spectra show that α -crystals were mainly formed upon fast cooling. Then, a transition from α to β'-crystals took place during the aging process. Also, according to differential scanning calorimetric results, the endothermal peak temperature was shifted to higher temperatures due to fast cooling and aging processes.

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