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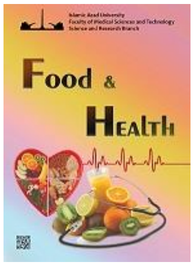

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Effects of cooling rate and aging process on crystallographic structure, whipping, rheological, textural and thermal properties of frozen minarine

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ABSTRACT

Minarine is a cream obtained by mixture of animal cream and vegetable oils. Fat crystallization is the main stage in the production of this product and affects its mouth-feel, 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. The objective of this study is to characterize the effects of fast cooling by ice cream maker to 5 ºC and aging process at this temperature for 24 hours, on whipping, rheological, textural, crystallographic and thermal properties of frozen minarine. 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), were belonged to sample FCA due to the formation of the denser crystalline network resulting from fast cooling and aging process. Wide-angle X-ray scattering spectra shows that α -crystals were mainly formed upon fast cooling then, a transition from α to β'-crystals took place during 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

Studies Whipping cream is an oil-in-water (O/W) emulsion containing water, milk, and or its derived products in the aqueous phase and permitted edible oils or fats in 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 price. Minarine is a cream obtained by mixture of animal cream and vegetable oils (3). Due to the fact that 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, mouth-feel, and appearance (4). The various forms of fatty acid crystals, also 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. While the β crystal with needle-shaped morphology and coarse appearance is the most stable form and has the highest density and melting point. This type of crystal tends to grow into clusters that can

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lead to a grainy texture in the fat phase. In addition to these three main crystals, there is also gamma crystal with a glassy state. The life of this crystal is very short 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 that are 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 takes place during aging process at refrigerator temperature. Whereas in slow cooling, β' crystals are formed from the beginning (7, 10). The whipping cream should be stored at low temperature (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 and is called the aging process. During this process, proteins and stabilizers are hydrated, emulsifiers are rearranged, and fat crystallization occurs (11). The presence of the liquid phase causes the triglycerides to dissolve from the unstable crystals and recrystallize them into more stable and pure crystals. This phenomenon takes place during aging process at refrigerator temperature at which temperature 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 is a pasteurized and homogenized product containing 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 the homogenization stage minarine samples were subjected to deferent cooling rate and aging process. 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 the whipping process, the frozen minarine samples were taken out of the freezer and kept at room temperature for 20 minutes until partial softening and then whipping operation by a mixer (Kitchen aid/America) was accomplished with a specific program of 5 minutes at 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. Overrun percent was calculated according to the following equation:

$$\%\,Overrun = \frac{M_1 - M_2}{M_2} \times 100$$

where, M1 (g) is the weight of 100 ml of minarine before whipping, and M2 (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 which was 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 Peltier system assisted by a fluid circulator. The parallel plate geometry with 40 mm diameter and gap of

1 mm was used. After loading, the samples were rested for 1 minute in order to achieve temperature equilibrium, eliminate induced stress and recovery of the original structure. All rheological tests were performed at 5 ± 0.01°C. At first, strain sweeps test (0.01–600%) was done at fixed frequency of 1 Hz in order to detect the linear viscoelastic region. The recorded parameters were the strain corresponding to the end of the linear viscoelastic range (γLVR), the elastic modulus in LVR (G′ LVR), and the viscose modulus in LVR (G″ LVR). Frequency sweeps test was performed at constant strain of 0.01% and the frequency range of 0.01–100 Hz to expose 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 R2 using the Bohlin equation: $G' = A \times (\omega)b$
Rheoplus software version 3.21 (Anton-Paar) was applied to gather the data of experiments (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 Back extrusion test. A cylindrical probe (diameter: 35mm) attached to a 5 kg load cell was applied to compress the samples. In this test, the whipped minarine samples are poured into the chamber and placed under the probe. Then, the probe, which is 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 and 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 the parameters of 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 orientation of them. 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 the Duncan's multiple-range test was applied to determine significant differences between means.

3. Results and discussion

3.1. Overrun

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 rate and aging process (p<0.05). The fast cooling with ice cream maker before freezing (FC) and also slow cooling at refrigerator conditions and aging process at this temperature for 24 hours (SCA), led to significant increase in overrun compared to the control sample (BS) which was freezed directly. This phenomenon is attributed to enhancement of fat globules partial coalescence as a result of fast cooling and aging process. The effect of aging process was more than that of fast cooling due to formation of β´ crystals (8). The highest overrun percent (114.8%) was belonged to sample FCA. In this sample, α crystals are formed first, then during aging process, crystallization of fats increases and a transition from unstable crystals to more stable β' crystals takes place.

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.

Consequently, a dense and compact lattice of β' crystals is formed which leads to increase connection points for partial coalescence (3, 8, 18). Therefore, by applying such thermal pretreatment, the desired and optimal overrun content can be achieved. The optimal and desired amount of overrun in confectionery cream has been reported in the range of 120-115% by volume of air.

3.2. Syneresis

Serum loss in whipping cream shows the emulsion instability. As demonstrated in Table 1, syneresis values of minarine samples were significantly different under the various cooling rate and aging process (p<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 FCA sample followed by the SCA sample which has been subjected to aging process. These results demonstrated that aging process causes enhancement of emulsion stability and reduction of serum loss. This can be attributed to increase of 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. With regarding this, the 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 the physical stability and shelf life of emulsion-based food products. Strain sweeps and frequency sweeps are common rheological tests performed on confectionery cream.

3.3.1.Strain sweeps rheological properties

The strain sweeps test parameters of minarine samples are shown in Table 2. Up to the critical strain (γLVR), 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 resistant to whipping (14). In this study, the highest γLVR value (0.33%) belonged to FCA sample followed by SCA sample. These results were consistent with the results of the overrun test, as formerly mentioned, the FCA sample and then the SCA sample had the highest overrun values. Long et al. (14) also stated that the sample with longer LVR had greater whippability leading to more overrun. In all the whipped minarine samples the elastic modulus was more than viscose modulus (G′ > G") in linear viscoelastic region that is revealing domination of elastic behavior. The G′ values of treated samples were significantly more than that of control sample (BS) ($p<0.05$).

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.

It's worth noting that, higher elastic modulus is very desirable for confectionery cream, especially when it is used for decoration, which can keep its shape. The FCA sample and then the SCA sample showed the highest G′ value. Since both samples have been subjected to aging process, the observed difference between them 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 FCA sample ($p<0.05$). This difference is attributed to the effect of aging process on minarine elastic modulus. Also, similar results were found in the study conducted by Ronholt et al (13) on effects of cream cooling rate and aging process on polymorphism, microstructure and rheological properties of butter. The authors reported that aging process of cream led to 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" which is indicative of whipped minarine solid like structure. Also, the slope of the variation 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).

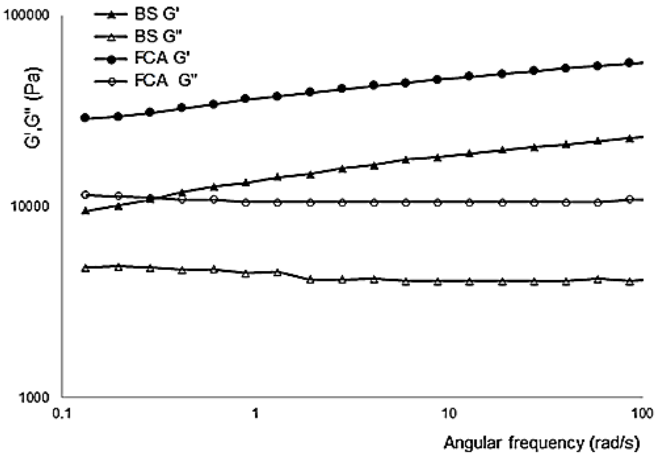

Fig. 1. Frequency sweeps test of FCA and BS samples.

With increasing frequency, the G′ increased and elastic behavior prevails over viscose behavior. A similar result was also reported by Jakubczyk and Niranjan (20). The dependency of G′ on frequency was fitted by Bohlin model with a determination coefficient of R2 >0.97. The parameters corresponding to this model are mentioned in Table 3.

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.

Based on the Bohlin model, the value of A is the measure of structural strength between rheological units and the value of b indicates the number of interconnected rheological units in the three-dimensional emulsion network, as well as the emulsions 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) were belonged to FCA sample. This is related to formation of large number of crystals during fast cooling and increasing fat crystallization during aging process leading to enhance connection points between fat globules and the consistency (18).

3.4. Textural properties

The purpose of back extrusion test is to determine the firmness, consistency, viscosity, and cohesiveness. Firmness or maximum force is actually 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 also it is resistant to flow. As presented in Table 4, the cooling rate and aging process had significant effects on 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, formation of large number of small crystals with rough surfaces during fast cooling and increasing of fat crystallization during aging process lead to enhance partial coalescence of fat globules and consequently formation of dense and compact fat network, 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 ($p<0.05$). 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. For each crystal, several plates or rows with different distances (d-space) can be considered. As 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. 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 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 quick solidification of these samples at freezing temperature and reduction of mobility which leads to prevent transition of the unstable α crystals to the more stable β´ crystals. These results are in agreement with the results of study done by Fredrick et al (7) on investigation of isothermal crystallization behavior of milk fat in bulk and emulsified state. Whereas in slow cooled sample (SCA), β' crystals were formed from the beginning, which is consistent with the previous studies (10).

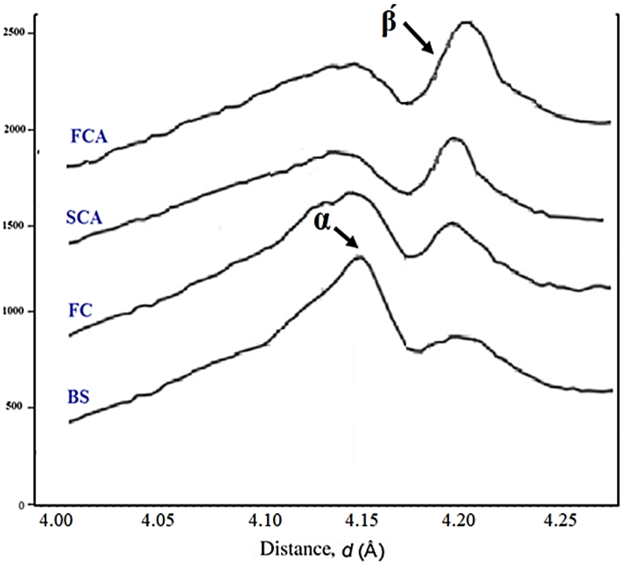

Fig. 2. Fat crystals polymorphism of frozen minarine samples.

3.6. Thermal properties

The results of 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 shift the endothermal peak temperature to higher temperatures in treated samples compared to the control sample.

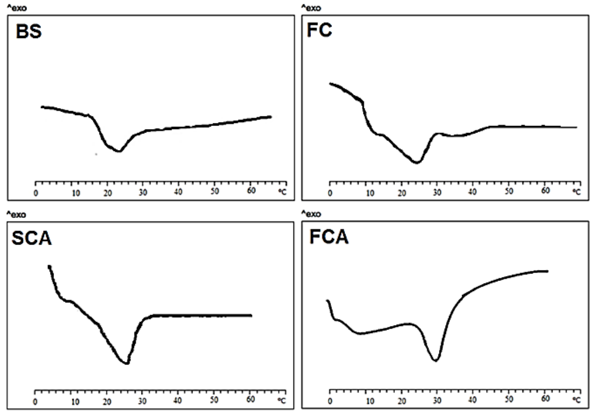

Fig. 3. Thermal profile of frozen minarine samples.

During the aging process, the fat crystals had enough time to rearrangement and formation of dense and ordered networks leading to increase the melting point of FCA and SCA samples in comparison with other samples. Also, the transition of the unstable α crystals to the more stable β´ crystals during the aging process caused to shift the endothermal peak temperature to higher temperatures in these samples (8, 12). The highest endothermal peak shift compared to the control sample was belonged to 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) were belonged to FCA sample, due to the formation of a denser crystalline network resulting from fast cooling and aging process. Wide-angle X-ray scattering spectra shows that α -crystals were mainly formed upon fast cooling then, a transition from α to β'-crystals took place during 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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