# Effects of homogenization pressure and sequence on textural and microstructural properties of milk-based creamy dessert

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#### ABSTRACT

Effects of homogenization sequence (before or after heating) and homogenization pressure (0, 50, or 150 bar) were studied on the certain textural properties of milk-based creamy dessert including hardness, surface tension and syneresis. Also, the microstructure of the treatments was analyzed using scanning electron microscopy (SEM). Homogenization at 50 bar after heating led to the highest hardness, whilst unhomogenized and homogenized treatments at 150 bar before heating resulted in the lowest hardness. Using pressure of 50 and 150 bar after heating led to the highest surface tension, respectively. While the highest syneresis was observed when unhomogenized treatment applied, the lowest syneresis was obtained using primarily the treatments with homogenization after heating and then the treatments with homogenization before heating. Finally, good correlation was observed between the textural and rheological results and the micrographs of microstructure obtained from SEM method.

Key words: Dessert; Homogenization; Microstructure; Rheology; SEM; Texture.

## **INTRODUCTION**

Milk-based desserts are mixtures of cocoa, chocolate, and/or fruit preparations with a high percentage of dairy ingredients with the addition of binding agents and stabilizers, sugar (sucrose) and possibly emulsifiers [1-3]. Depending on the type and usage of the dessert, numerous extra components such as flavoring agents, colorants and nuts can also be added [4]. Milk-based desserts can be categorized from various points of view. From the formulation and rheological properties point of view, they can be divided into three groups including liquid puddings with high amount of starch and low amount of carrageenan, viscosity and yield stress, flans with low amount of starch, high viscosity and yield stress and creamy desserts with intermediate mentioned characteristics. From the appearance point of view, they can be classified into three groups including desserts with creamy texture such as custards and puddings, multi-layer desserts (with e.g. whipped cream topping, chocolate bottom layer or fruit layers) and firm de-mouldable desserts such as flans and gelly desserts [2].

Nowadays, considering importance of desserts as snacks and value-added products and their organoleptic properties as their critical value, the importance and necessity of designing and producing desserts with satisfactory sensory properties are inevitable. Apart from the effects of compositional factors (especially fat, protein and sugar content, and type/amount of stabilizers and emulsifiers), process factors can also significantly influence textural and rheological properties of milk-based desserts. From these heating factors. process conditions. homogenization pressure, homogenization temperature and sequence, filling and cooling conditions, stirring and agitating, method of mixing, refrigerated storage temperature and storage time can be mentioned [2, 3, 5]. Although the effects of process factors on the textural and rheological properties of frozen dairy desserts has been comprehensively reviewed in the literatures, to the best of author's knowledge, only a few documents are available in the case of milk-based creamy desserts [2], and no information has been reported about the

interactive effects of homogenization pressure and sequence (in relation with heat treatment, *i.e.*, before or after heating) on their textural and microstructural characteristics; in contrast with the products such as dairy creams and yogurt [2-7]. Therefore, the objective of this study was to investigate the combined effect of homogenization pressure and sequence (before or after heat treatment) on the textural and microstructural.

## MATERIALS AND METHODS

#### Chemicals and sample preparation devices

Carrageenan (Types HMF) was supplied by Robertet (Can, France). Skimmed milk and cream were obtained from Pak Dairy Co. (Tehran, Iran) and natural starch from Glucosan Co. (Tehran, Iran). A batch pasteurizator (Robatmakhzan, Islamshahr, Iran), а (APV-60-10 TBS, Gaulinhomogenizator laboratory, Germany), a mixer (Shimifan, Shahriyar, Iran), a cold incubator (Irankhodsaz, Saveh, Iran) and a sealing machine (Nemoone-e-Tabriz, Tabriz, Iran) were used in this study for samples preparation.

## Sample preparation method

То prepare the samples, compositions including 83% raw fresh milk with 3.8% fat, sugar (sucrose), 4.5% starch, 0.5% 12% carrageenan and a little vanilla and salt were mixed for 30 min at 30°C. This is a popular formula for making creamy dairy dessert. Heat treatment (85°C for 1 min) or homogenization (0, 50 or 150 bar) was then performed. In the case of homogenization before heating, preheating at 60°C was applied. In converse sequence of homogenization and heat treatment. homogenization was performed after rapid cooling of samples up to 60°C. Flavouring agent was added to the formula before filling stage. After hot filling and packaging of the samples, they were rapidly cooled-down and kept at 5°C until used.

## Measurements

Hardness measurement was carried out using a Universal texturometer (Hounsfield, Germany) at 25°C according to Karami [8]. Penetration speed of the probe and the depth of penetration were 100 mm/min and 19 mm, respectively. The probe diameter was 4.5 cm.

To measure surface tension, a tensiometer (K9, KRUSS GmbH, Hamburg, Germany) with

wilhelmy plate was used according to Dickinson and Pawlowsky [9]. Before each measurement, the platinum plate was immersed in nitric acid (5%) and then in distilled water followed by a direct heating over a flame up to the blushing. After pouring 100 mL of sample in the cell of the tensiometer, plate was forced into the samples and then withdrawn from them slowly until the detachment of sample from the plate occurred. To measure syneresis, centrifugation method at 10,000 g for 10 min (ambient temperature) was applied according to Nassirpour [10], using a J-21B centrifuge system (Beckman, USA). The volume of separated serum from a certain sample mass after centrifugation was measured.

# Scanning Electron microscopy (SEM)

To obtain SEM images, a Scanning Electron Microscope model CO-C2-400 (Howard Electronic Instruments, Canada and USA) was used, according to Aichinger *et al.* [11]. Stages of sample preparation before SEM operation consisted of cutting, freeze drying, goldsputtering and silver-coating.

## Statistical analysis

Experiments were performed in triplicate. The significant differences between the means and their ranked orders were analyzed using Factorial design and Duncan's test from MSTATC software (version 2.10, Pussell D. Freed, Crop and Soil Science Department, Michigan State University).

# **RESULTS AND DISCUSSION**

## Hardness

Figure 1 indicates the effect of homogenization pressure and sequence on the hardness of desserts samples. As shown, the treatment of 50 bar after heating (50-a) led to the highest hardness. Vice versa, the lowest hardness was obtained in the case of unhomogenized treatments and homogenized ones with the pressure of 150 bar before heating (150-b). To justify the results, it is better to categorize them into the following sections according to Figure 1: i)At the same homogenization pressures (50 or 150 bar), homogenization after heating resulted in the higher hardness than homogenization before heating;

*ii*)At both homogenization sequences (before or after heating), hardness is reduced when homogenization pressure is increased.



**Figure 1**- Effect of homogenization pressure and position on hardness. a=after hating, b=before heating

\*The means shown with different letters are significantly different (p < 0.01).



**Figure 2-** Micrographs of the samples treated at the homogenization pressure of 150 bar before heating (a) and homogenization pressure of 150 bar after heating (b).



Figure 3- Effect of homogenization pressure and position on surface tension. a=after, b=before

\*The means shown with different letters are significantly different (P<0.01).



**Figure 4**- Micrographs of the samples treated at the homogenization pressure of 150 bar after heating (a) and homogenization pressure of 50 bar after heating (b).









**Figure 6**- Micrographs of the samples at the homogenization pressure of 50 bar after heating (a) with no homogenization (b) and at the homogenization pressure of 50 bar before heating (c).

The first observation can be primarily attributed to the changes occurred in the type and number of carbohydrate-carbohydrate and carbohydrate-protein bonds that can affect hardness and cohesiveness of the network structure. Based on Figure 2, it is apparent that the molecular network formed by homogenization after heating substantially consists of mass-shape and plate-form parts with high density, thickness and cohesiveness. This phenomenon increases the hardness of the network. There are various reports to the benefit of homogenization after heat treatment in the case of dairy creams. Although from hygiene view point, homogenization is preferred before heat treatment, however, homogenization after heat treatment reduces problems of milk fat hydrolytic/lipolytic rancidity due to previously denaturation of milk lipases [5]. This is the reason why this order of processing is favoured by many manufacturers. At the conditions that homogenization performed before heat treatment, heating at least at a level of pasteurization temperature done immediately must be after homogenization in order to avoid lipolytic rancidity [12]. Also, in the case of UHT sterilized cream, homogenization after heat treatment is a necessity, because of reducing the risk of protein coagulation during the heating [5]. It has been reported that applying heat treatment before homogenization in the skim milk induces casein/whey proteins complexes to be formed more extensive. This leads to the formation of artificial membrane of milk proteins on the surface of fat droplets after homogenization with significantly higher protein load, compared with converse order of [6]. Protein load processing increases especially when severe heat treatment applied before homogenization. The higher amount of protein loads around fat droplets gives the higher probability of cluster structure formation between the milk proteins, which can produce considerably firmer and more consistent structure [7]. Therefore, mentioned reports about the effects of homogenization and heat treatment sequence are in consistence with our observations.

The second observation could be because of more open and less dense molecular network formation in parallel with increase in homogenization pressure (Figure 4). The more open molecular network, the less gel strands density and junction zones formed in the microstructure, and as a result, the less hardness and consistency of the structure achieved (Figure 4). This result has been previously confirmed by Rapaille and VanHemelrijck [2], who had reported that homogenization pressure is indirectly proportional to the hardness and syneresis of milk-based desserts, respectively. However, to achieve the best smoothness and mouth-feeling of the texture, slight homogenization is required. The narrow point about the effect of homogenization pressure on the firmness/hardness of the creamy texture dairy products is its interactive effect with fat content.

In general, high amounts of homogenization pressures can degrade casein micelles, which results in the formation of weaker caseindependent structures such as yogurt network and emulsified structure of fat droplets after cream or milk homogenization, made by substantially casein micelles/particles artificial membranes [5]. This is the reason why through the production of the products such as yogurt and dairy creams, cream homogenization (separated cream) or partial homogenization (cream with certain portion of skim milk), instead of whole milk homogenization, is carried out. However, if the fat content of the product is sufficiently high (e.g., >18%) to allow clustered structure to be formed due to the generation of shared casein membranes between the fat droplets, the firmness/viscosity of the texture can be increased up to reaching a paste structure [7]. Clustering could be also formed by applying high pressures of homogenization [7], but such a treatment, as previously mentioned, leads to the degradation of casein micelles and as a result, weaker casein-based structures. Therefore, after severe heat treatment which causes the potential for the formation of artificial membrane of milk proteins on the fat droplets with higher protein loads after homogenization (due to the complex formation between the casein micelles and denatured whey proteins), if the fat content is sufficiently high but the homogenization pressure is not much severe to degrade protein complexes, the best results from the hardness and smoothness of the texture apparently is achievable. Also, two-stage homogenization must be refused to avoid degradation of the protein complexes. However, even sever homogenization pressures can be expected to make firm texture at the present of enough high fat percentage.

In our study, because the fat content was only 6.5%, it can be concluded that higher homogenization pressures weakened final product consistency.

Apart from the mechanisms explained, presence of carrageenan and starch significantly affect textural properties of milkbased creamy dessert. K-carrageenan has been proved to make electrostatic bonds with kcasein. These bonds are formed between the sulfate groups in k-carrageenan and carboxyl groups in k-casein, above the isoelectric point of casein proteins (about 4.6) by the mediation of calcium ions, and between the amine groups of k-casein and sulfate groups of k-carrageenan, below their isoelectric pH [13-15]. These reactions between the casein micelles and kcarrageenan are occurred when the amount of carrageenan does not exceed from 0.1%,

because at such a conditions, complex formation between the *k*-carrageenan polymer chains themselves would be the prominent reaction. Also, different types of complexes have been reported between k-carrageenan and whey proteins [15]. However, no data is available about the effect of homogenization and heat treatment sequence on mentioned complexes and bonds, regarding textural properties of milk-based creamy desserts. It has been reported that gentle homogenization (about 20 kg/cm<sup>2</sup>), in contrast with high homogenization, pressure as causes homogenous distribution of stabilizers within the texture of dairy desserts, results in the more smooth and coherent structure [16].

Starch also contributes in the textural and rheological characteristics of dairy desserts. An important issue regarding relation of starch with homogenization and heat treatment sequence was that when the homogenization was applied after heat treatment, lumpiness observed in the texture of final product (data not shown).

This fact has been previously reported by Rapaille and VanHemelrijck [2]. They justified the phenomenon in this way that when the gelatinized starch granules, generated by exposing to high temperatures of heat treatment, subsequently subjected to the homogenization process, considerably higher agglomeration of starch particles is occurred compared with converse sequence. Using modified starches has been recommended by the authors to overcome this problem.

According to Figure 1, homogenization treatment at 50 bar before heating resulted in the higher hardness compared with the treatment of 150 bar after heating. This observation reveals that the unsuitable effect of homogenization pressure increase on the texture consistency of the product is considerably more than performing the process sequence of homogenization before heat treatment.

As shown in the Figure 1, effect of homogenization treatment at 150 bar before heating on the hardness is statistically the same as that of unhomogenized conditions. Accordingly, as is evident in the Figure 6, no significant difference between the microstructures of two mentioned treatments was observed.

## Surface tension and syneresis

As shown in Figure 3, the homogenization pressure of 50 bar after heating and 150 bar after heating leads to the highest and lowest surface tension respectively. Micrographs of the above mentioned treatments (Fig. 4) show noticeable differences in network density, coherence and cohesiveness. Increasing cohesiveness and network density on the surface area of the texture results in higher surface tension due to the higher number and more strength of attractions within the unit volume of stressed molecules. This is consistent with the fact that the homogenization at 50 bar after heating, which gives the highest hardness, also results in the highest surface tension. Figure 5 indicates the effect of homogenization pressure and sequence on syneresis. According to the Figure, the highest syneresis is related to the unhomogenized treatments, whereas the lowest is obtained primarily the treatments using with homogenization after heating and then treatments with homogenization before heating. Homogenization pressure of 50 bar after heating which resulted in the highest hardness and surface tension led to the lowest syneresis. Justifications for the observations can be exhibited as following: the content of syneresis is substantially related to the structure hardness, size and arrangement of spaces and voids within the network and amounts of bound water. Degradation of structure due to its weakness leads to exiting free water physically entrapped in the network structure. Distribution of network spaces in the form of smaller, but with more number of voids and pores, has two advantages: first by partial degradation of the network in some parts, less amount of free water is exited and second, amount of bound water that does not contribute in syneresis is increased.

According to Figures 5 and 6, an increase in syneresis is observed in the following order: homogenization after heating<homogenization before heating<unhomogenized treatments. It should be discussed that higher hardness results in lower syneresis especially when the elastic limit of the set-structure increases with raising its elastic modulus. On the contrary, if increase in hardness leads to a more brittle structure, a stress level above the rupture point can destroy the structure and as a result, a high amount of syneresis is obtained.

## CONCLUSION

The aim of this work was to study the combined effects of homogenization pressure and sequence on the certain textural properties (hardness, surface tension and syneresis) of milk-based creamy dessert and also to justify these properties with respect to micrographs obtained by SEM method. The results demonstrated that mentioned variables significantly affect the textural and microstructural characteristics of milk-based dessert and as a result, the sensory attributes of final product. The best choice of treatments should be made according to the taste of consumers taste. Hardness and surface tension of dairy desserts have considerable effects on their sensory perception. Syneresis is generally

## REFERENCES

1.Nadison G. The interaction of carrageenan and starch in cream desserts. DAIRY IND INT 1990; 55: 12-17.

2.Rapalle A, Vanhemelrijck J. Milk based desserts. The technology of dairy products. Thomson Science, UK, 1998; 327-352.

3.Spreer E. Milk and dairy products technology. Marcel Dekker, USA 1998; 155-202.

4.Mann EJ. Dairy desserts and related products. DAIRY IND INT 1990; 55: 31-33.

5.Varnam AH, Sutherland JP. Milk and milk products. Chapman & Hall, UK 1994. 183-223. 6.Ortwijn H, Walstra P. The membrane of recombined fat globules: 2 compositions. NETH MILK DAIRY J 1979; 33: 143-154.

7.Walstra P, Van Vliet T, Kloek W. Physical chemistry of milk fat globules. Advanced dairy chemistry. Chapman & Hall, UK 1995; 131-178.

8.Karami M. 2003. Effect of type of stabilizer on the rheological properties of processed cheese. MS thesis, University of Tehran, Iran 2003.

9.Dickinson E, Pawlowsky K. Effect of carrageenan on flocculation: creaming and rheology of a protein-stabilized emulsion. J AGRIC FOOD CHEM 1997; 45: 379-383.

10.Nassirpour A. Effect of homogenization pressure and fat content on the qualitative parameters of breakfast cream. MS thesis, University of Tehran, Iran 2001.

recognized as an unpleasant parameter. According to the results from this study, it seems that the treatment of 50 bar homogenization after heat treatment can be the best choice from both sensory and economical points of view. It results in the highest hardness and surface tension, but the lowest syneresis. This treatment is also more economical than treatment at 150 bar pressure. However, in order to achieve more decisive selection, also sensory evaluation tests are also required.

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11.Aichinger PA, Michel M, Servais C, Dillmann ML, Reuvet M, Zink R, Klostermeyer H, Horne DS. Fermentation of skim milk concentrate with *Sreptococcus thermophilus* and chymosin: Structure, viscoelesticity and syneresis of gels. BIOINTERFACES 2003; 31: 243-255.

12.Fox PF, Mc Sweeney PLH. Physical properties of milk. Dairy chemistry and biochemisrty. Thomson Science Publishing, UK 1998; 437-461.

13.Hansen PMT. Hydrocolloids-protein interactions: relationship to stabilization of fluid milk products. Gums and stabilizers for the food industry. Royal Society of Chemistry, UK 1982; 127-138.

14.Lin CF. Interaction of sulfated polysaccharides with proteins. Food colloids. Royal Society of Chemistry, UK 1977; 205-236. 15 Bourrio S, Garnier C, Boublier JL. A description of micellar casein-*k*-carrageenan mixed system by means of calorimetry and rheology. Gums and stabilizers for the food industry. Royal Society of Chemistry, UK 1998; 202-222.

16.Goff HD. Instability and partial coalescence in whippable dairy emulsion. J DAIRY SCI 1997; 80: 2620-2626.