Investigation of the stability of frozen food emulsions

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Thesis book

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Introduction and Objectives

In recent years, ice creams and frozen desserts were sold in large quantities in the frozen food market, not only globally but also in Europe. The largest producers of frozen desserts -like ice cream, frozen yogurt and frozen cakes – in the world are North America and Australia, while in Europe, the United Kingdom, Germany and Denmark. These giant companies produce desserts on an industrial scale, apply a wide variety of texture modifiers and by a proper measurement instrument park they are able to get a clear picture of quality changes in frozen products. So their frozen products can be stored for long time without degradation.

The domestic refrigeration industry produces even today mainly frozen vegetables and fruits, and in small quantities semi-processed and ready meals. After the campaign period (June-November), however, the high capacity freezing equipment are unused till the next season, in the plant they mostly package the products for the consumers, which were manufactured in summer. Like the foreign frozen dessert manufacturers, the Hungarian industry also could use their infrastructure to produce frozen desserts to take advantage of this market gap. However, there is not much information about the storage of frozen cakes. The developments of foreign companies are secret, so it is important to examine the stability of these products during freezing and frozen storage.

The majority of foods are complicated complex systems, and we must recognize and know the changes occurring during processing and storage. In addition, we need to know during product development, how the added components affect the properties of the food.

Nowadays the demand for artisanal products (even without additives) is steadily growing, and it is also true for confectionery products. The various cakes filled with confectionery cream and ice creams are very popular. However, the handling and development of these have been established only empirically in small scale plants. It is important to know for example, how the unused creams could be preserved in such a manner as to maintain the formed stable structure, and obtain appropriate organoleptic properties of the products during future use. Development of ice cream is also an actual topic, there are countless variations of flavouring, only limited by imagination, if we have a proper ice cream base. However, if we change the basic ice cream recipe we will have a product with completely different melting and textural characteristics. Therefore, I chose as the topic of my dissertation the research of the stability of artisanal confectionery creams and ice creams with instruments capable of thermo-physical and rheological measurements. I tried to find out how are thermo-physical and textural characteristics affected by freezing and frozen storage in creams and the addition of acid whey in ice creams.

My objectives based on that were the followings:

In case of artisanal confectionery creams:

- To determine for confectionery creams, whether there is a difference in the methods of freezing and the period of frozen storage, in particular with regard to monitoring the changes in thermo-physical and structural characteristics, and which methods are the most suitable ones to detect these differences,
- To determine whether the thermo-physical and rheological measurement methods are capable of detecting the changes in confectionery cream stability caused by freezing,
- To define the proper technique of freezing and the maximum frozen storage period based on the measurements.

In case of ice creams

- To examine whether the acid whey concentration affects the structure and physical properties of ice creams,
- To determine whether the thermo-physical and rheological measurement methods are capable of detecting the changes that occur, and mapping the changes of physical properties and structural characteristics of ice creams;
- To determine the maximum useable amount of acid whey concentrate which is suitable from sensory and technological perspective.

Materials and methods

Emulsions used in the experimental work

During my work I used many types of emulsions and studied their stability from rheological and thermo-physical perspective. For the different food emulsions a variety of model foods were used. These were the followings:

- margarines: 8 types of Norwegian and a special cream margarine
- confectionery creams: based on traditional recipes and corn starch and pudding powder were added
- ice creams: milk-based ice creams that are supplemented with different amounts of acid whey concentrate
- freezing and frozen storage of confectionery creams

Two types of confectionery creams (one made with corn starch, the other with pudding powder and whipped with a special cream margarine) were frozen with three different freezing methods and stored frozen for 6 months at -24 °C.

The instruments and methods which were used in the experimental work

Determination of the thermo-physical properties

Measurements such as the initial temperature of intensive melting, the glass transition temperature and unfreezable water content of the different milk-based ice cream samples and frozen confectionery cream samples (one made with corn starch, the other with pudding powder and whipped with a special MeisterCreme margarine) were carried out with a Setaram DSC 131 evo-type instrument.

During the examination of the samples the temperature was reduced by a rate of 5°C/min from + 30°C to-50°C and held for 15 minutes at -50 °C before the heat-up phase. Then temperature of ice cream samples was increased with 2°C/min heating rate from -50°C to + 30°C by the instrument, while the temperature of the confectionery cream was increased to +50°C also by 2°C/min heating rate . During the measurement changes of the heat flow data were recorded as a function of the sample’s temperature. The evaluation was carried out in the warm-up phase of the measured heat flow recorded as a function of the sample’s temperature with the program Callisto Processing 1.076 version.

Rheological examinations

Rheological properties of margarines, confectionery creams and ice creams were measured by oscillation rheometry. The used measure methods were as follows: amplitude sweep, temperature sweep, frequency sweep and torque test in rotary measurement mode.

Rheological measurements of margarines and confectionery creams

The three types of Norwegian cup margarines were measured by the method of the amplitude sweep.

The measurement settings were the followings: Physica USD200 type rheometer, plate-plate measuring system, MP 31/P type, 50 mm diameter rough (sandblasted) surfaced plate, measurement temperature: 4°C, the distance between the plate-plate was 1,00 mm. The value of the angular frequency constant was 10 1/s; the amplitude was 0.01% - 200% of degree of the distance between the two plates.

All types of margarines and the 5 Norwegian margarines recommended for cream making, and the confectionery creams made of MeisterCreme margarine with corn starch and pudding powder were measured by the temperature sweep method.

The measurement settings were the followings: Physica USD200 type rheometer, cone-plate measuring system, MK 22 type cone head (1°), 50 mm diameter, distance between the cone and plate was 0,05 mm. During the measurement the amplitude constant was 0,1 %, angular frequency constant was 10 1/s. With this measurement we can examine the changes of texture characteristics during the freezing and thawing. The temperature program consists of three phases. The first phase is cooling from 20 °C to -18 °C by a cooling rate of 4 °C/min, the second stage is holding at -18 °C for 10 min and the third phase is heating from -18°C to 20°C, at a heating rate of 4 °C/min.

Whipped MeisterCreme margarine and two types of confectionery creams (made of MeisterCrème margarine with corn starch and pudding powder) were measured by the amplitude sweep method.

The measurement settings were the followings: MCR51 type Physica rheometer, measurement temperature: in case of margarine 12 °C, 14 °C, 16 °C, 18 °C and 20 °C, in case of confectionery creams 4 °C, 6 °C, 8 °C, 10 °C, 12 °C, 14 °C, 16 °C, 18 °C, 20 °C, plate-plate measuring system type MP 31/P, 50 mm diameter rough (sandblasted) surfaced plate, the distance between the plate-plate was 1,00 mm. The value of angular frequency constant was 10 1/s; the amplitude was 0.01% -200% of degree of the distance between the two plates.

The examined confectionery cream samples were stored frozen for 6 months. I measured the frozen samples monthly. The samples were thawed and rheological characteristics were measured at 10 °C, and at the end of 6 months of storage at 4 °C, 10 °C and 20 °C. Rheoplus software was used for data analysis.

Rheological measurements of ice creams

Ice creams were measured by Physica MCR51 type viscometer, with plate-plate measurement system type PP50/S, 50 mm diameter rough (sandblasted) surfaced plate, distance between the plates was 2 mm. I used four measuring methods.

For the torque test constant angular frequency (10 1/s) and constant torque (250 μNm) values were used at varying temperatures, the temperature program was as follows: heating from -15 °C to 0 °C, cooling from 0 °C to -15 °C.

During the amplitude sweep method the level of amplitude was varied from 0.001 to 40% of degree of the distance between the plates, with a constant 10 1/s angular frequency value at -10 °C.

During the frequency sweep measurements were carried out under constant amplitude 0.002% and increasing frequency values from 10 to 100 Hz at constant -10 °C measurement temperature.

During the temperature sweep measurements the amplitude value was constantly 0,005%, the angular frequency was constant 10 1/s. The temperature values were increased from -15 °C to 0 °C. Data evaluation was carried out by Rheoplus software.

Measurement of texture characteristics

The texture characteristics of the whipped MeisterCreme margarine and confectionery creams made with MeisterCreme margarine were measured at 4 °C, 6 °C, 8 °C, 10 °C, 12 °C and 20 °C by TA.XTPlus (Stable Micro System) type precision texture analyser instrument. For the experiment I used the TTC Spreadability Rig. The probe is 50 mm high and passes 48 mm deep in the test sample. The load cell force was 500 N, and the probe moved at a speed of 2 mm/s in the sample. Exponent software was used for data analysis.

Sensory analyses

To examine the capability of frozen storage of confectionery creams, I made a layered cake from the confectionery cream samples and sponge cake, and stored it frozen for the sensory tests. The sensory evaluation was performed with an average of 10 trained assessors monthly, scoring methods were used and the confectionery cream samples were evaluated based on three criteria, which were the homogeneity, light creaminess and hardness. The temperature was the same in all cases, 10 °C.

For the sake of comparability, I made a fresh sample each month. The samples were frozen with three different methods, frozen stored for 1-6 months and then thawed. Stored and a freshly made samples were tested always together.

Scoring method was used during the ice-cream sensory evaluation to determine the amount of sour whey concentrate in ice cream which could be already perceived and tolerated by consumers. 15 expert judges tasted the six ice cream samples. The evaluation criteria were the taste, color, texture, overall impression, scoopability, meltability, homogeneity and creaminess.

Results

I began my research by examining the margarine assortment to determine what types of margarines can be used to create confectionery creams, which form a stable system in spite of freezing. To determine this I used the temperature sweep method, and I got the result that a special margarine (Meister Creme) could be a good basic material for further experiments. I whipped the selected margarine and studied its temperature-dependent behavior with the amplitude sweep method and by texture measurement using a spreadability rig. Then I prepared the confectionery cream cooked from pudding gel and whipped margarine. I measured the temperature-dependent behavior with the two above mentioned methods and compared the margarine and the confectionary cream and the results of the two measurement methods. I determined that textural properties, such as firmness or spreadability, can be monitored with the two measurement methods. The optimal processing temperature can be set, which is between 10 and 14 °C, based on my measurements. The samples in this temperature range can be mixed and spread well. Comparing the measurement parameters of the two measurement methods, I found strong correlation between initial modulus of elasticity and loss modulus determining the firmness and cohesivity of samples, and between modulus of elasticity, measured in the intersection, and extrusion and adhesion properties measured by texture analysis. In addition, extrusion force and work values, determining the spreadability, showed a strong correlation with the slopes fitted to the curve sections of the modulus of elasticity and the loss modulus after their intersection. So spreadability is measurable and quantifiable for stable emulsions using the oscillating method, too. In the next series of experiments, I froze confectionery creams made by pudding powder and corn starch by liquid nitrogen (cryogenic freezing mode), air blast and slow freezing method, then stored them at -24 °C for 6 months. Samples were taken in every month. The thermo-physical parameters were determined by a differential scanning calorimeter, changes in the textural properties were measured by an oscillatory viscometer and a texture analyser. I found that the samples prepared with pudding powder were more stable than those made by native corn starch. DSC measurements detected changes in structure occurring during frozen storage such as starch retrogradation, and destabilization of the fat phase, and as a result, phase inversion. Changes occurred in the parameters measured by oscillatory viscometer. From the fifth month on S1 and S2 parameters, typical of the spreadability, could not be measured because phase inversion occurred as the effect of large deforming force, and the probe slipped on the sample. We can demonstrate the effect of freezing methods on the sample structure with the results of texture analysis, and determine the application of the appropriate freezing technology. The sensory test results show the differences between the two spreads and the samples having satisfactory organoleptic properties can be detected. These results established that freezing in air blast gives better results than the cryogenic and slow freezing. After air blast freezing, this type of confectionery cream can be stored frozen for up to 4 months, to ensure an adequately stable structure for further use and consumption.

In the next series of experiments I investigated the effect of addition of whey concentrate on the changes in the properties of ice cream. I performed measurements with DSC, oscillatory viscometer in three measurement modes, and sensory test was also carried out. It was proved that the addition of whey reduces the initial temperature of melting and the amount of unfreezable water, which affects the melting and textural characteristics of ice cream. I pointed out that the whey concentrate positively affected the creaminess of ice cream and the development of its soft character by decreasing the shear stress value measured in the intersection of the two curves. The frequency sweep method refers to the stability and the time-dependent behavior of the ice cream. Measurements showed that increasing the amount of whey concentrate slightly reduced the stability of the ice cream, but the sample can endure short and long-term storage and transportation without phase separation. Temperature sweep method showed that by the addition of whey concentrate, handling of ice cream is possible in a wider temperature range. The sensory tests have shown that the addition of whey had little impact on the creaminess, the homogeneity and the scoopability of ice cream, but its addition above 20% is not recommended, because whey concentrate significantly alters the flavor of ice cream. I made correlation analysis and I found that the thermo-physical and rheological properties showed a strong correlation with each other, so the parameters determined by DSC and temperature sweep method completely characterize the most important quality properties of ice cream.

New scientific results

1. I have found that the amplitude sweep method is suitable to measure the spreadability of emulsions used by the confectionery industry such as water-in-oil (eg. margarines) and oil-in-water (eg. margarine and starch-gel based confectionery creams). Strong correlation can be found in the large deformation range between the slopes of lines well-fitting on the curves of elastic modulus (G ') and storage modulus (G”) and the texture properties measured by the spreadability rig.

2. I have found that the freezing method of disperse systems has effect on the properties of the product. My measurements proved that the ultra-fast / cryogenic freezing adversely affects the texture properties of the confectionery creams. On the basis of my measurements I suggest the application of air blast freezing with 3-4 m/s air velocity at -35 °C.

3. Based on my measurements I proved that after frozen storage at -18 °C for 4 months the rheological properties change adversely, therefore the maximum period of frozen storage in case of confectionery products prepared with confectionery cream is 4 months, due to changes in the rheological properties.

4. It was found that between -50 °C and + 50 °C the structural changes of confectionery creams (oil-in-water emulsion) caused by freezing and frozen storage are detectable from the change in the enthalpy values (J/g) measured by differential scanning calorimetry (DSC) measurement (increase in the value of water melting enthalpy and at the same time the decrease in the value of fat melting enthalpy), and the shifting of peak temperatures in negative direction.

5. I have found that the thermo-physical and textural characteristics of milk-based ice creams, measured by amplitude sweep and temperature sweep methods, are affected by the whey concentrate. At -10 °C, by the effect of increasing the whey concentrate, elasticity and storage modulus decrease, the complex viscosity values measured by the temperature sweep method are also reduced and of thermo-physical characteristics the initial temperatures of intensive melting decrease as well, so ice cream will be softer and creamier.

6. I have found that the examined rheological parameters have a strong correlation with thermo-physical properties in case of ice creams. In practical terms, texture characteristics, determining organoleptic quality, can be estimated from the thermo-physical properties and the complex viscosity values measured by temperature sweep method.

Primary publications related to the dissertation

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