Rheological and thermophysical properties of dairy desserts before and after freezing*

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Summary

Because of its significant amount of proteins, lactose, vitamins and minerals, whey is very important byproduct in cheese technology. However, it isn't enough used in human nutrition although it could be used as a component of various food products like ice cream, cakes, bread, drinks etc. In this investigation it was used as one of the main ingredients in dairy desserts preparation. As such products are usually preserved by freezing, and in this work were examined their thermophysical and rheological properties. Rheological properties were measured at 20°C using rotational viscometer and phase transitions (freezing and thawing) temperatures were determined using differential thermal analysis. Freezing temperatures of desserts varied from - 2 to - 5°C, and thawing temperatures from -9 to -5°C. All desserts were pseudoplastic and their consistency depended on fraction and on the composititon of solids. After freezing and 30 days' storage at - 18°C, their freezing temperatures decreased but melting temperatures did not change. Consistency increased, but dairy desserts pseudoplascic character remained unchanged.

Additional index words: whey, dairy desserts, freezing, thermophysical properties, rheological properties

1. Introduction

Whey is very valuable by-product in cheese manufacture and could be used in bread, sausages, ice cream, dairy desserts and fruit drinks production, and also as one of ingredients in serial food products. As it contains valuable proteins, minerals, vitamins and lactose, it improves the foods nutritional value. It also decreases due amount of other sweeteners in products. Concerning suggestions of world's health institutions to consumers, to use foods containing lower fat, cholesterol and sucrose content, a quite number of products appeared marked as light or low energy (Anon, 1989; Lachance, 1989; Hatchwell, 1994.). These products have lower energetic value (no more than 1.6 kJ/g) and reduced fat content (at least 50% of reduction) (Shank and Carson, 1991; Anon, 1990; Campbell, Ketelsen and Antenucci, 1994.).

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The aim of this work was to prepare a new product, cream dessert type, containing a great fraction of whey as fundamental characteristic and small fraction of fat that increase its nutritive value. Important property of such product beside flavor, is its consistency (Tharp and Gottemoller, 1990; Specter and Setser, 1994). In majority of products belonging to the group of dairy creams or ice-creams, specific consistency could be achieved using adequate fraction of solids, as well as milk fat and different additives (emulsifiers, stabilizers) (Hegedušić, Lovrić and Prlog, 1991; Giese, 1993). For all that, it is indispensable to investigate their rheological properties owing to their influence on technological processes and their faculty to determine product's quality (Parnell-Clunies, Kakuda and Deman, 1986; Chaffai-Hamza, 1990; Ramaswamy and Basak, 1992). With regard to non-Newtonian's product characteristics it is recommended to use different types of rheometers when determining their consistency. The best one for such measurements are those of rotating type.

Dessert creams are of limited shelf life due to their composition and relatively high water content (according to the activity of microorganisms). For the purpose to enable longer shelf life of creams, it is necessary to freeze them. With regard to that, it is important to know thermophysical properties of dessert creams, particularly freezing and thawing temperatures, as these parameters determine conditions of freezing process, as well as storage temperature of frozen products (Hegedušić, Lovrić and Tkalec, 1993). Product's structure could be disrupted during freezing process due to water crystallization and forming ice crystals of different sizes causing changes in its physico-chemical characteristics (Herald, Osorio and Smith, 1989; Hegedušić and Lovrić, 1990). Consequently, taking aim in determining the intensity of these changes, it is important to determine rheological characteristics of products after thawing (Noda nad Shiinoki, 1986; Hegedušić, Lovrić and Parmać, 1993; Hegedušić, Piližota and Šubarić, 1994; Šubarić, Piližota and Lovrić, 1994).

2. Materials and methods

Dairy desserts preparation

Investigations were carried out with ten dairy desserts (marked as sample 1 to 10), prepared after recipes from Table 1. The following ingredients were used:

- sucrose, N crystal
- "Sladial", "Pliva", Croatia
- egg yolk (S class of eggs from local market)
- concentrated sweet whey (45% solid matter) "Zdenka", Croatia
- whole milk powder, "Zdenka", Croatia
- wheat flour, Type 400
- sodium alginate, "Grindsted", Denmark
- glycerol, "Kemika", Croatia

Table 1 Ingredients used for dairy desserts preparation
Tablica 1. Sastojci upotrebljeni za pripremu mliječnih krema

Ingredient % Sastojak	Sample Uzorak 1	2	3	4	5	6	7	8	9	10
Sucrose Saharoza	9.00	-	-	-	-	-	-	-	-	-
"Sladial"	-	9.00	8.74	8.74	8.74	8.74	9.00	7.14	7.14	7.14
Egg yolk Žumanjak	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00
Whey (conc.) Sirutka	20.20	20.20	20.20	20.20	20.20	20.20	20.20	30.00	30.00	30.00
Whole milk powder Punomasno mlijeku u prahu	11.00	11.00	11.00	-	-	10.86	11.00	10.00	-	-
Wheat flour Pšenično brašno	1.80	1.80	1.80	1.80	1.80	1.80	-	1.65	1.65	1.65
Glycerol Glicerol	-	-	-	-	-	-	-	-	6.50	-
Sodium algal Natrijev alginat	-	-	0.26	0.26	0.66	0.40	0.66	0.13	0.13	0.13
Water Voda	80.00	80.00	80.00	91.00	90.60	80.00	81.14	73.08	76.58	83.08

As it could be seen from Table 1, in cream desserts preparation, sucrose (sample No. 1) and natural sweetener "Sladial" (samples No. 2 to No. 10) with the purpose to investigate their influence on cream's sensory properties, were used. With the aim to increase cream's nutritive value, concentrated whey due to its significant minerals (especially Ca and Mg) as well as vitamins, lactose and proteins content was used. For that purpose the highest quantity of whey (20.2 to 30%) still having positive effect on the cream's taste, was added.

Sucrose (or "Sladial") was mixed with Na-alginate and egg yolks in a blender. A suspension of flour, milk powder and water was added into a mixture. At the end, it was added whey and as much water to obtain the mixture that weighed 130 grams. The mixture was put in a hot water bath and cooked exactly for 7 minutes at 80°C. In this way 23% of water was evaporated and cream mass was reduced to 100 grams. Stirred cream was cooled to 10°C and subjected to sensory evaluation by 7 assessors, using 20 point's scale system. Average points of sesnoric evaluation are presented in Table 2.

Prepared creams were analyzed to determine rheological properties and temperatures of phase transition. Some of the creams (No. 3, 5, 6, 7, 9 and 10) were put in polyethylene containers (cream mass being 100 grams), cupped and frozen in a freezer at -18°C. At that temperature, creams were stored for 30 days, and after that rheological properties as well as temperatures of phase transition were determined again.

Phase transition temperatures

Phase transition temperatures were determined by differential thermal analysis using apparatus MP DT PtL (Faculty of Food Technology and Biotechnology, Zagreb), by continuously scanning of temperature as well as temperature difference between sample and referent material (quartz sand). Cooling was performed by ethanol and liquid nitrogen (cooling rate = 4°C/min), and thawing by ambient air, temperature 20°C (thawing rate = 0,5°C/min). (Hegedušić and Lovrić, 1985.). As the results of measurements, DTA curves were obtained. The beginning of DTA peaks was determined as the beginning of phase transition (freezing, thawing).

All measurements were made three times and as result the arithmetic mean value was used.

Rheological properties

Rheological properties were determined using a rotational rheometer (VT 500, Haake, Germany) with concentric cylinders fixtures type SV DIN. Shear stress against the increasing shearates from 0 to 100 l/s and from 0 to 500 l/s (rising measurements) were measured. All measurements were carried out at 20°C and in an interval of 2 minutes. Shear stress and shear rate values were recalculated

Table 2 Average points of sensoric evaluation of dairy desserts
Tablica 2. Prosječni broj bodova senzorske procjene mliječnih krema

Quality parameter (maxima points) Parametri kakvoće (maksimalan broj bodova)	Sample Uzorak 1	2	3	4	5	6	7	8	9	10
Taste (8) Okus (8)	5.3	5.4	7.0	4.4	6.1	6.9	6.3	7.1	6.8	5.2
Consistency (6) Konzistencija (6)	4.1	2.6	4.3	2.3	4.6	3.9	4.9	5.4	3.8	2.0
Odour (4) Miris (4)	3.1	3.0	3.3	2.7	2.9	3.3	3.1	3.4	3.4	3.3
Colour (2) Boja (2)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Total (20) Ukupno (20)	14.5	13.0	16.6	11.4	15.6	16.1	16.3	17.9	16.0	12.5

Table 3 Composition and energetic value of dairy desserts (in 100 g)*
Tablica 3. Sastav i energetska vrijednost mliječnih krema (u 100 g)*

Sample Uzorak	Solid matter % Suha tvar	Protein g Bjelančevine	Total fat g Ukupna mast	Total carbohydrate g Ukupni ugljikohidrati	Lactose g Laktoza	Sucrose g Saharoza	Energetic value kJ Energetska vrijednost
1	34.18	5.09	5.53	21.10	10.97	9.00	646
2	34.20	5.09	5.53	21.10	10.97	-	646
3	34.07	5.09	5.53	21.10	10.97	-	612
4	23.73	2.47	2.62	17.11	6.78	-	426
5	24.15	2.47	2.82	17.51	6.78	-	440
6	33.90	5.09	5.53	21.25	10.97	-	619
7	33.28	4.90	5.51	20.43	10.97	-	597
8	35.79	5.42	5.47	22.23	13.84	-	668
9	32.14	2.92	2.67	24.86	10.17	-	578
10	25.69	2.92	2.67	18.37	10.17	-	466

*Based on chemical composition of ingredients
*Utemeljena na kemijskom sastavu sastojaka

in flow index and consistency coefficient value after Ostwald's model, using Haake's computer program.

$$\tau = k\, y^n \qquad /1/$$

where:
τ = shear stress (Pa)
y = shear rate (l/s)
n = flow index
k = consistency coefficient (Pa s^n)

Apparent viscosity, both at shear rate 100 l/s and 500 l/s was calculated as a ratio between shear stress (measuerd value from flow curve) and corresponding shear rate. Suitability of Ostwald's model for data analyzing was determined after regression analysis according to the least squarer method using a computer program (Haake).

3. Results and discussion

Table 2 presents average points of cream sensory evaluation done by 7 assessors. The most important trait of these products being flavor, it was awarded 8 points. Cream No. 8 was evaluated as the best ragarding its flavor (it contained higher fraction of whey and lower fraction of flour and Na-alginate). Only a bit inferior were creams No. 3 and No. 7. The worst was cream No. 4 because of its higher fraction of water. Evaluated cream consistency was dissimilar. The consistency of cream No. 8 was the best, and the worst one was that of cream No. 10. The reason being dissimilar partition of dry matter (Table 3), as well as their composition. More proteins in cream No. 8 very probably favored firmer and more homogenous system. Considering total rank of points, cream No. 8 was superior (17.9 points). Very good were also creams No. 3, 6 and 7. The worst was cream No. 4 (11.4 points) owing to its low dry matter content and lack of milk powder.

Because of creams nutrivite and energetic values (Table 3), lower energetic values of creams No. 4,5 and 10 were due to remarkable fat content (2.65 - 2.82% that is around 50% less than the rest) and carbohydrate partition. According to it, these creams, as well as remaining analyzed creams (about 5.5% fat) could be ranked in the category "Light dairy desserts", their fat contents being less than 10% (minimal fat quantity in case of ice cream) (Shank and Carson, 1990). The energetic values of the rest of creams varied from 578 to 668 kJ/100 g. As from FDA's perspective, the term "low calorie" means no more than 1.6 kJ/g, these dairy desserts are not included in this category, but only in the category of "light frozen dairy desserts" (Shank and Carson, 1990.).

Table 4 Phase transition temperatures of dairy desserts before freezing and after 30 days of storage at -18°C
Tablica 4. Temperature faznih promjena mliječnih krema prije zamrzavanja i poslije 30 dana skladištenja pri -18°C

Sample Uzorak	Before freezing Prije zamrzavanja		After storage at -18°C Nakon skladištenja pri -18°C	
	Freezing temp. (°C) Temperatura zamrzavanja	Thawing temp. (°C) Temperatura odmrzavanja	Freezing temp. (°C) Temperatura zamrzavanja	Thawing temp. (°C) Temperatura odmrzavanja
1	-5.0	-8.0	-	-
2	-4.0	-6.0	-	-
3	-4.0	-9.0	-4.0	-7.0
4	-3.0	-6.0	-	-
5	-4.0	-7.0	-4.5	-8.0
6	-4.0	-7.0	-4.5	-7.0
7	-4.5	-8.0	-5.0	-9.0
8	-3.0	-5.0	-	-
9	-4.0	-6.0	-5.5	-7.0
10	-2.0	-4.5	-3.5	-7.0

Carbohydrates make 50% of cream's total dry matter, and fats and proteins 25% each, in keeping line with nutritionist's recommendations (A n o n, 1989). The highest carbohydrate content (50%) is due to lactose, and the rest to "Sladial".

Phase transition temperatures

The phase transition temperatures (freezing, melting) of investigated creams were determined using DTA. The data do suggest that initial freezing temperatures of creams vary between -2.0 to -5.0°C. This result does not enable to think on the influence due to some compound parameters on freezing temperatures owing to the fact that cream's composition was rather similar and temperature differences small. Only dead certainty is to establish the fact that glycerol presence in cream No. 9 influenced decrease of freezing temperature (-4.0°C) in relation to the freezing temperature of sample No. 10 (-2.0°C) having identical composition minus glycerol.

Thawing temperatures were considerably lower (about 2 to 3°C) than their freezing temperatures varying from -9.0 to -5.0°C, meaning that in the case of storing frozen creams, it is necessary to keep them at temperatures below -10.0°C.

Table 5 Rheological parameters of dairy desserts after Ostwald's model
Tablica 5. Reološki parametri mliječnih krema prema modelu Ostwalda

Sample Uzorak	Shear rate 1/s Smična brzina	Flow index n Indeks tečenja	Consistency coefficient k (Pa s^n) Koeficijent konzistencije	Regression coefficient R^2 Koeficijent regresije	Apparent viscosity at max. γ,μ_a (Pa s) Prividni viskozitet uz maks. γ
1	0-100	0.25	22.31	0.99	0.96
	0-500	0.36	19.04	0.98	0.39
2	0-100	0.11	24.05	0.98	0.40
	0-500	0.22	22.99	0.99	0.18
3	0-100	0.08	37.34	0.96	0.55
	0-500	0.14	31.85	0.98	0.16
4	0-100	0.26	25.23	0.96	0.90
	0-500	0.32	23.14	0.97	0.34
5	0-100	0.28	29.16	0.96	1.16
	0-500	0.35	26.05	0.99	0.48
6	0-100	0.27	65.27	0.96	2.15
	0-500	0.19	92.26	0.96	0.62
7	0-100	0.24	45.51	0.99	1.42
	0-500	0.26	44.32	0.99	0.45
8	0-100	0.24	39.68	0.99	1.17
	0-500	0.18	54.30	0.99	0.33
9	0-100	0.21	29.05	0.98	0.76
	0-500	0.25	31.21	0.99	0.30
10	0-100	0.22	26.30	0.99	0.73
	0-500	0.35	24.20	0.99	0.43

Table 6 Rheological parameters of dairy desserts after freezing calculated using Ostwald's model (shear rate range: 0-100 l/s)
Tablica 6. Reološki parametri mliječnih krema poslije zamrzavanja izračunati prema modelu Ostwalda (raspon smične brzine: 0-100 l/s)

Sample Uzorak	Flow index n Indeks tečenja	Consistency coefficient k (Pa s^n) Koeficijent konzistencije	Regression coefficient R^2 Koeficijent regresije	Apparent viscosity at max. γ, μ_a (Pa s) Prividni viskozitet uz maks. γ
3	0.18	45.19	0.97	1.04
5	0.21	63.27	0.98	1.68
6	0.17	75.83	0.96	1.65
7	0.15	76.84	0.90	1.53
9	0.21	37.15	0.95	0.98
10	0.20	34.38	0.98	0.86

Rheological properties

Determination of cream's rheological properties is done in the area of shear rate from 0 to 100 l/s and from 0 to 500 l/s. Rheological characteristics were determined using Ostwald model owing to the fact that regression coefficients in this case were very high (between 0.96 to 0.99). Table 5 shows that flow index values, **n**, were below 1. meaning that the character of all creams was pseudoplastic. Coefficients of consistency depended on shear rate measuring area, and in general were higher in the area from 0 to 100 l/s. The lowest **k** value had sample No. 1 (prepared adding sucrose) although its apparent viscosity at 100, respectively, 500 l/s was above some alternative cream samples. Sample no. 6 had the highest consistency coefficient value, containing the highest part of dry matter and added Na-alginate.

Sample No. 8 got the best sensory evaluation score for consistency (Table 2) attained value **k** 39.68 Pa s, respectively, 54.30 Pa s, a little less than sample No. 6. According to it one could deduce that optimal viscosity as expressed by cream's consistency coefficient would be at 40.00 or 55.00 Pa s.

Creams containing higher fraction of dry matter in general demonstrated higher viscosity.

Particular ingredients addition influenced viscosity differently so that creams containing "Sladial" were more viscous than that containing sucrose. Na-alginate increased cream viscosity depending on its quantity, as well as on presence of other ingredients, primarily on flour and milk powder.

Creams No. 9 and No. 10 prepared without addition of milk powder and having reduced fraction of flour and Na-alginate. However, their roughly equal consistency was nearly comparable to creams No. 4 No. 5 meaning that increased share of whey favorably influenced consistency. Addition of glycerol increased also cream's No. 9 consistency.

Apparent viscosity of creams was determined using maximal shear rate i.e. 100 and 500 l/s. Data show 2 to 3 times greater viscosity at 100 l/s than at 500 l/s, meaning that these creams had typical pseudoplastic character particulary at smaller shear rates.

Some of prepared creams thawed after freezing and storing at -18°C during 30 days, were subject to repeated rheological properties' determination. Data are represented in Table 6 and Fig. 1 and 2. According to Fig. 2 it is perceptible that creams retained pseudoplastic character after freezing, indicated by values of flow index (Table 6). However, cream's consistency increased owing to the consistency coefficient value and to apparent viscosity, as well as according to Fig. 2. The reason for it is to be searched for in some increase of cream's dry matter and in the structure system interruption provoked by water crystallization.

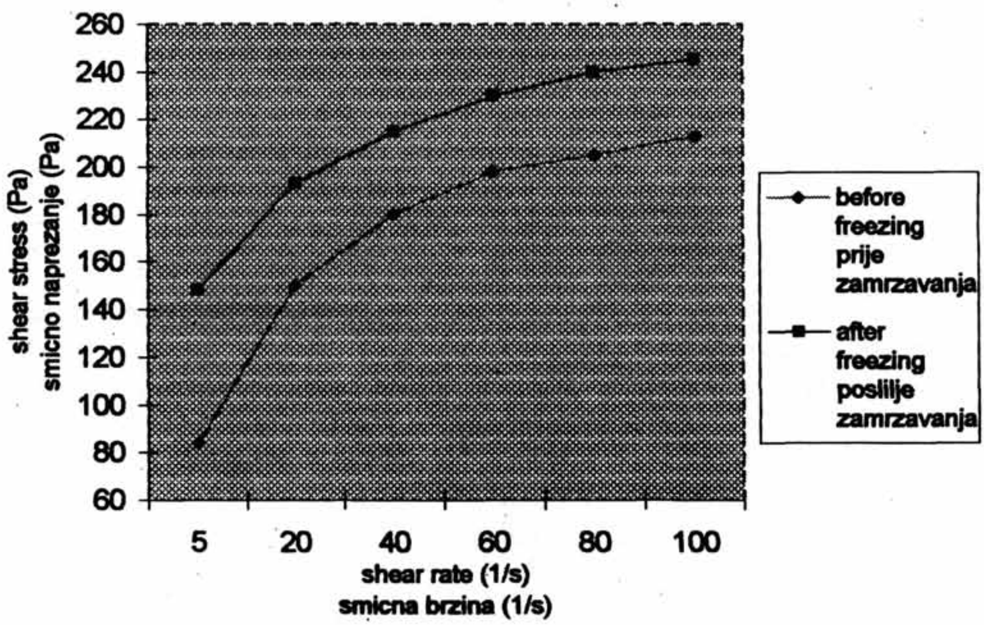

Fig. 1 Shear stress and shear rate ralationship of sample 6 before and after freezing
Slika 1. Odnos smičnog naprezanja i smične brzine uzorka 6 prije i poslije zamrzavanja

4. Conclusion

All prepared dairy desserts, due to their low milk fat content, may be considered as "light" products, as their energetic value ranged from 5.78 to 6.68 kJ/g. Desserts exhibited non-Newtonian pseudoplastic character unchanged after freezing and during storage. Consistency was dependent both, on the solid matter content and on the composition. Glycerol and sodium alginate addition had a significant influence on desserts consistency increasing.

Freezing temperatures of dairy desserts varied between -2 and -5°C, while their thawing temperatures were lower (from -5 to -9°C). After freezing and 30 days storage at -18°C, phase transition temperatures of investigated dairy desserts decreased insignificantly, and consistency increased considerably.

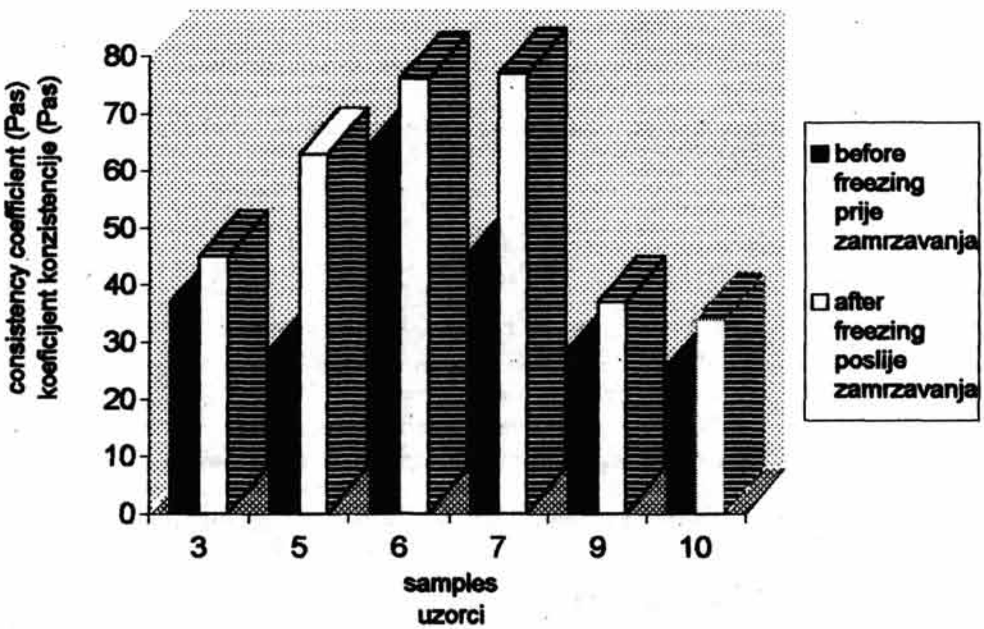

Fig. 2 Consistency coefficient values (k) of samples 3, 5, 6, 7, 9 and 10 before and after freezing
Slika 2. Vrijednosti koeficijenta konzistencije (k) uzoraka 3,5,6,7,9 i 10 prije i poslije zamrzavanja

TERMOFIZIČKA I REOLOŠKA SVOJSTVA SMRZNUTIH MLIJEČNIH DESERATA

Sažetak

Sirutka je zbog svog značajnog udjela proteina, laktoze, vitamina i mineralnih tvari značajan nusprodukt proizvodnje sira. Ipak, još se uvijek ne koristi dovoljno u prehrani ljudi, iako se može upotrebljavati kao dodatak različitim vrstama hrane kao što su sladoled, kolači, keksi, kruh, pića i dr. U ovom istraživanju sirutka je upotrebljena kao jedna od glavnih komponenti u pripremi mliječnih krema. Kako se te kreme obično konzerviraju smrzavanjem, u ovom radu su istražena njihova termofizička i reološka svojstava. Reološka svojstva mjerena su pri 20°C u rotacionom viskozimetru, a temperature faznih promjena (smrzavanja i odmrzavanja) uz pomoć diferencijalne termičke analize. Njihove temperature smrzavanja bile su između -2 i -5°C, a temperature odmrzavanja od -9 do -5°C.

Svi deserti bili su pseudoplastična karaktera, a njihova konzistencija je ovisila o udjelu i sastavu suhe tvari. Nakon smrzavanja i 30 dnevnog skladištenja pri -18°C njihove temperature smrzavanja su se snizile, ali je pseudoplastičan karakter ostao nepromijenjen.

Riječi natuknice: sirutka, mliječni deserti, smrzavanje, termofizička svojstva, reološka svojstva

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