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Rheological Properties of Carboxymethylcellulose and Whey Model Solutions before and after Freezing

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Summary

Hydrocolloids, macromolecular carbohydrates are added to many foodstuffs with the aim to achieve the appropriate rheological properties, to prevent syneresis or to increase the viscosity and stability of foodstuffs.

In this work, the influence of the type and concentration of commercial hydrocolloids (carboxymethylcelluloses) on the rheological properties of model solutions of whey, whey proteins, sucrose, sorbitol and lactose was examined. The influence of freezing on rheological properties of model solutions was also checked. Measurements were done using a Brookfield DV-III rotational viscometer at temperature of 20 °C.

The results have shown that all examined systems are non-Newtonian. Depending on the chemical composition and on the mass fraction of hydrocolloids, they exhibited pseudoplastic or dilatant properties. All CMC effected a significant increase of the model solutions viscosity.

The freezing process had no significant effect on the viscosity of the model solution prepared with water. However, whey based solutions had a greater viscosity after freezing.

The results of variance analysis showed that all examined sources of variation (composition of model solution, type of CMC and freezing process) had a significant influence on the rheological parameters.

Key words: rheological properties, carboxymethylcelluloses, whey proteins, freezing

Introduction

Hydrocolloids are mostly complex carbohydrates which are used to improve consistency and textural characteristics (rheological properties) of liquid and semiliquid foodstuffs. Their activity depends on the kind and concentration of hydrocolloids, temperature and process condition, as well as on solid matter content and chemical composition of foodstuffs. They can be added in various combinations and phases of production and may have various final effects (*1*).

The type of hydrocolloids largely determines overall appearance, texture and rheological properties of food, whereby nutritional values and sensory qualities of food products are not changed (*2–5*). Their activity could depend on the interactions among hydrocolloids and other components of food (*6–8*).

Carboxymethylcellulose (CMC), as a typical hydrocolloid, has no direct influence on the taste and flavour of foodstuffs, but at the same time has a significant effect on gel formation, water retention, emulsifying and aroma retention (*9*,*10*). In the food industry CMC is used as a stabilizer, binder, thickener, suspending and water-retaining agent, in ice-cream and other frozen des-

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serts, fluid and powdered fruit drinks, sauces and creams, cake mixes and slimming and dietary foods.

Ultrafiltrated whey and whey proteins are commonly used in the food industry for dairy desserts production. They are also gelling agents or enhance functional properties of food (*11–15*).

The functional role of proteins as a food ingredient depends on a complex interaction of various factors, such as heating or cooling rates, protein concentration, pH, ionic strength and interactions with other food components, sugars, minerals *etc.* (*16–20*).

The aim of this paper was to find out the influence of the type of carboxymethylcelulloses on the rheological properties of whey model solutions, as well as of whey protein, sucrose, sorbitol and lactose model solutions and to examine the influence of freezing process on the viscosity of the model solutions.

Materials and Methods

The studies were carried out with eight model solutions (marked as samples 1 to 8 in Table 1) prepared by mixing of following ingredients:

- Sucrose, sugar – Šećerana Županja
- Sorbitol p.a. – »Merck«
- Ultrafiltrated whey (10 % solid matter) – »Dukat« d.d.
- Whey proteins concentrate (WPC) (60 % proteins in solid matter) – »Dukat« d.d.
- Proteins free whey (10 % solid matter)
- Carboxymethylcelluloses (commercial names – YO-EH, DIKO, HVEP) – Guliver-Chemie, Wiener Neudorf

Preparation of proteins free whey

Ultrafiltrated whey was cooked in water bath at the temperature of 90 °C for 20 minutes. After that, it was cooled to 20 °C and filtrated through a gauze. The residuum on the gauze contained coagulated whey proteins. The filtrate, protein free whey with 6 % solid matter was evaporated on the water bath to the 10 % solid matter.

The rheological properties of three different types of commercial CMC (YO-EH, DIKO, HVEP) were examined. CMC dispersions with 0.1, 0.2, and 0.4 % mass fractions were prepared in distilled water by vigorous hand mixing at 20 °C.

The model solutions of sucrose, sorbitol and lactose (samples No. 5–7, Table 1) were prepared with the aim to find out the influence of carbohydrates and CMC interactions on the rheological properties of these model systems.

The samples No. 1, 2, 8, as well as No. 3 and No. 4 (Table 1), were prepared with the purpose to determine the possible interactions among proteins, inorganic compounds, CMC and carbohydrates.

Measurements

The measurements were performed using a rotational viscometer, Brookfield DV-III, with coaxial cylin-

Table 1. Composition of investigated model systems

Model system No.	Compounds						
	w_t(sucrose)	w_t(sorbitol)	w_t(lactose)	w_t(ultrafiltered whey)	w_t(proteines free whey)	w_t(WPC)	w_t(hydrocolloid)
	%	%	%	%	%	%	%
1	–	–	–	9.90	–	–	0.1
	–	–	–	9.80	–	–	0.2
	–	–	–	9.60	–	–	0.4
2	–	–	–	–	9.90	–	0.1
	–	–	–	–	9.80	–	0.2
	–	–	–	–	9.60	–	0.4
3	4.95	–	–	4.95	–	–	0.1
	4.90	–	–	4.90	–	–	0.2
	4.80	–	–	4.80	–	–	0.4
4	–	4.95	–	4.95	–	–	0.1
	–	4.90	–	4.90	–	–	0.2
	–	4.80	–	4.80	–	–	0.4
5	9.90	–	–	–	–	–	0.1
	9.80	–	–	–	–	–	0.2
	9.60	–	–	–	–	–	0.4
6	–	9.90	–	–	–	–	0.1
	–	9.80	–	–	–	–	0.2
		9.60	–	–	–	–	0.4
7	–	–	9.90	–	–	–	0.1
	–	–	9.80	–	–	–	0.2
	–	–	9.60	–	–	–	0.4
8	–	–	–	–	–	9.90	0.1
	–	–	–	–	–	9.80	0.2
	–	–	–	–	–	9.60	0.4

ders, carrying out shear stress (τ) and shear rate (γ) with shear rate increasing from the lowest value (3.9 s^{-1}) for every system to 317 s^{-1} (upwards), as well as from 317 s^{-1} (downwards) to the lowest shear rate. At the highest shear rate, shear stress lasted two minutes, and after that the rotational rate successively decreased to the initial value.

After preparations and after freezing (–20 °C) in a laboratory freezer, all measurements were made at temperature of 20 °C. All solutions were kept frozen for 24 h. After thawing at ambient temperature for 14 hours, the measurements of rheological properties were performed again at the same conditions as before freezing.

The rheological parameters (consistency coefficient and flow behavior index) were calculated by a computer program according to Ostwald de Waele power-law model (7).

$$\tau = k \cdot \gamma^{n} \qquad /1/$$

where: τ – shear stress (Pa), γ – shear rate (L/s), n – flow index, k – consistency coefficient (Pa s^n)

Apparent viscosity at 60 s^{-1} was calculated using Newtonian law:

$$\tau = \mu_{a} \cdot \gamma \qquad / 2 /$$

where: τ – shear stress (Pa), γ – shear rate (L/s), μ_a – apparent viscosity (Pa s)

The analysis of variance was used to examine the influence of sources of variation (model solution composition, type of hydrocolloid, freezing process) on the rheological parameters.

Results and Discussion

The rheological properties of food are influenced by temperature, chemical composition, solid matter content, processing, the interactions of food components and others (*1*). The aim of this work was to find out the influence of the interactions among carboxymethylcelluloses and other components in solutions (sucrose, sorbitol, whey proteins, lactose and inorganic compounds) on their rheological properties. Through the experiments an attempt to eliminate the effect of solid matter fraction was made. Therefore all model solutions had the same fraction (10 %) of solid matter (Table 1). Throughout the study it was apparent that the composition of solid matter had a significant influence on the efficiency of CMC. Model solutions prepared with water addition (samples No. 5, 6 and 7) had a higher viscosity than solutions prepared with ultrafiltrated whey (samples No. 1, 3 and 4) or concentrated whey proteins (sample No. 8) even though the solid matter content was the same in all solutions (Table 2). Solutions containing concentrated whey proteins (WPC – sample No. 8) and 0.1 % hydrocolloids (YO-EH, DIKO, HVEP) have significantly lower viscosity than those prepared with water. When the addition of hydrocolloids increased to 0.2 and 0.4 %, the viscosity, expressed as apparent viscosity as well as the consistency coefficient value of the WPC solutions significantly increased, compared to the viscosity of the so-

Table 2. Rheological characteristics of model systems with carboxymethylcellulose type HVEP addition before freezing

Model system No.	*w*(HVEP)	Apparent viscosity	Flow index	Consistency coefficient
	%	Pa s	n	*k* / Pa s^n
1 (Ultrafiltrated whey)	0.1*	3.200	1.043	0.002
	0.2	5.350	0.947	0.006
	0.4	12.100	0.937	0.013
2 (Proteins free whey)	0.1*	3.050	1.021	0.003
	0.2	5.150	0.893	0.008
	0.4	10.700	0.874	0.022
3 (Ultrafiltrated whey + sucrose)	0.1	4.050	0.983	0.003
	0.2	5.950	0.963	0.006
	0.4	12.900	0.925	0.016
4 (Ultrafiltrated whey + sorbitol)	0.1	4.200	0.973	0.003
	0.2	5.700	0.953	0.006
	0.4	13.700	0.919	0.017
5 (Sucrose)	0.1	6.550	0.876	0.011
	0.2	10.800	0.881	0.018
	0.4	20.500	0.833	0.041
6 (Sorbitol)	0.1	8.550	0.882	0.014
	0.2	13.900	0.849	0.032
	0.4	26.500	0.806	0.054
7 (Lactose)	0.1	9.950	0.800	0.025
	0.2	14.400	0.795	0.044
	0.4	27.800	0.738	0.067
8 (WPC)	0.1*	4.700	1.144	0.006
	0.2	7.650	0.987	0.013
	0.4	17.300	0.759	0.057

Flow behavior was pseudoplastic (structural-viscous behavior), except in cases, indicated by an asterix where the flow behavior was dilatant

Table 3. Rheological characteristics of model systems with carboxymethylcellulose type DIKO addition before freezing

Model system No.	*w*(DIKO)	Apparent viscosity	Flow index	Consistency coefficient
	%	Pa s	n	k / Pa s^n
1 (Ultrafiltrated whey)	0.1*	3.350	1.017	0.002
	0.2	5.150	0.913	0.014
	0.4	12.900	0.823	0.022
2 (Proteins free whey)	0.1*	2.700	1.054	0.002
	0.2	4.850	0.859	0.011
	0.4	12.200	0.809	0.019
3 (Ultrafiltrated whey + sucrose)	0.1	4.000	0.934	0.005
	0.2	6.850	0.867	0.016
	0.4	13.400	0.765	0.030
4 (Ultrafiltrated whey + sorbitol)	0.1	4.350	0.963	0.005
	0.2	7.350	0.909	0.016
	0.4	15.700	0.853	0.029
5 (Sucrose)	0.1	9.500	0.845	0.018
	0.2	14.900	0.820	0.031
	0.4	51.100	0.752	0.074
6 (Sorbitol)	0.1	9.600	0.835	0.019
	0.2	18.200	0.750	0.038
	0.4	61.100	0.763	0.084
7 (Lactose)	0.1	9.400	0.754	0.028
	0.2	15.700	0.787	0.054
	0.4	73.400	0.759	0.104
8 (WPC)	0.1*	5.800	1.060	0.006
	0.2	8.650	0.992	0.025
	0.4	42.900	0.721	0.069

Flow behavior was pseudoplastic (structural-viscous behavior), except in cases, indicated by an asterix where the flow behavior was dilatant

Table 4. Rheological characteristics of model systems with carboxymethylcellulose type YO-EH addition before freezing

Model system No.	*w*(YO - EH)	Apparent viscosity	Flow index	Consistency coefficient
	%	Pa s	n	k / Pa s^n
1 (Ultrafiltrated whey)	0.1	3.900	0.927	0.005
	0.2	6.800	0.932	0.016
	0.4	19.100	0.896	0.038
2 (Proteins free whey)	0.1	2.700	0.938	0.005
	0.2	6.250	0.818	0.009
	0.4	16.300	0.824	0.034
3 (Ultrafiltrated whey + sucrose)	0.1	4.700	0.880	0.011
	0.2	7.350	0.861	0.023
	0.4	19.800	0.899	0.042
4 (Ultrafiltrated whey + sorbitol)	0.1	4.900	0.960	0.009
	0.2	7.700	0.906	0.021
	0.4	23.000	0.900	0.046
5 (Sucrose)	0.1	10.100	0.887	0.015
	0.2	18.000	0.847	0.033
	0.4	38.600	0.838	0.077
6 (Sorbitol)	0.1	12.200	0.844	0.023
	0.2	22.900	0.823	0.044
	0.4	47.400	0.853	0.090
7 (Lactose)	0.1	10.500	0.751	0.032
	0.2	21.500	0.754	0.064
	0.4	44,200	0.839	0.091
8 (WPC)	0.1*	5.600	1.154	0.011
	0.2	8.100	0.955	0.024
	0.4	25.500	0.960	0.047

Flow behavior was pseudoplastic (structural-viscous behavior), except in cases, indicated by an asterix where the flow behavior was dilatant lution containing ultrafiltrated whey. The viscosity matched the quality of the solution prepared with water (Tables 2–4). Such behavior of hydrocolloids could be explained by interactions between proteins and hydrocolloids (*6,7*). When the hydrocolloid content is lower (0.1 %) an interaction occurs between the positive (active) groups of proteins and negative groups of hydrocolloids. When the hydrocolloid content was higher (0.2 and 0.4 %), its effectiveness increased, because the number of free, active groups of proteins decreased. This allows the hydrocolloids to bind greater amount of water and this can partially explain the differences in the viscosity between solutions prepared with ultrafiltrated whey and solutions prepared with water.

It is important to take into consideration the presence of significant amount of inorganic compounds in ultrafiltrated whey and protein free whey, as well as the presence of polyanion polysacharides (CMC) for binding greater amounts of cations. This type of interaction can improve homogenity of the systems. The contact of ion pairs (inorganic compounds – CMC) has a negative effect on the rheological properties of the model solutions which can be seen from the viscosity values of solutions containing protein free whey (containing a major quantity of inorganic compounds). The viscosity obtained was by far the lowest regardless on the quantity of hydrocolloids (Tables 2–4).

The above observation confirms the fact that all model solutions prepared with sucrose, sorbitol or lactose (samples No. 5, 6 and 7) have a distinctly higher viscosity than the other solutions. This can be explained by non-existence of the interaction between proteins and hydrocolloids or inorganic compounds and hydrocolloids.

Rheological properties of examined model solutions are adequately described according to Ostwald de Waele power-law model and expressed as consistency coefficient (*k*) and flow behaviour index (*n*).

From the shape of shear stress and shear rate curves (Fig. 1) and flow behaviour index values it is obvious that all examined model solutions exhibit a non-Newtonian character. Almost all solutions were pseudoplastic (structural – viscous behavior) except the solutions prepared with ultrafiltrated whey or WPC and with 0.1 % HVEP or DIKO addition (Tables 2 and 3) that exhibited dilatant properties.

It was mentioned that hydrocolloids have a significant influence on the flow behaviour since all the model solutions prepared with 0.2 or 0.4 % of CMC exhibited pseudoplastic properties. The increase of the hydrocolloid content in the solution made the pseudoplastic characteristics more apparent and it also increased significantly the viscosity of the model solutions.

Hydrocolloids, such as carboxymethylcellulose, are important ingredients in frozen dairy product preparations because of their ability to control crystallization and inhibit recrystallization. Therefore, the influence of the freezing process on the rheological properties of model solutions containing 0.4 % CMC was studied (Table 5). It was pointed out that after freezing all solutions had the same non-Newtonian character (structural-viscous) as before freezing (Fig. 2). The coefficient consistency values (*k*) of the model solutions prepared with water (samples No. 5–7) did not change significantly. The coefficient consistency values of the model solution prepared with whey (samples No. 1–4 and 8) increased dramatically, probably as a consequence of changes in binding capacity of whey proteins effected by water crystallization during freezing.

To verify the statistical significance of some variance sources (the composition of model solution, hydrocolloid content and freezing process) on the viscosity, the

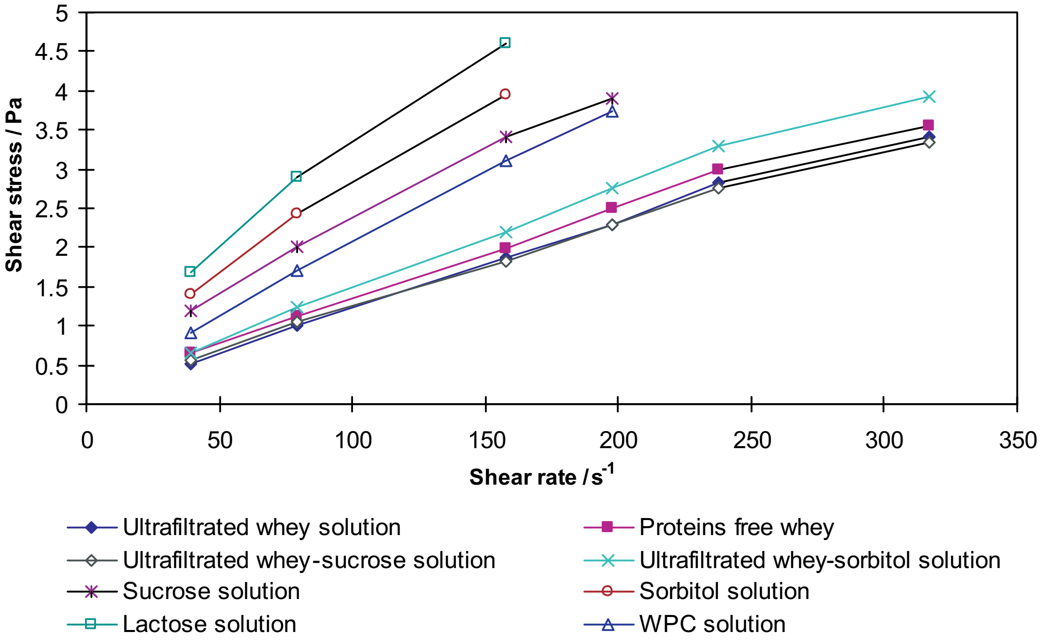

Fig. 1. Shear stress and shear rate relationship of model solutions with addition of hydrocolloid DIKO (0.4 %) before freezing

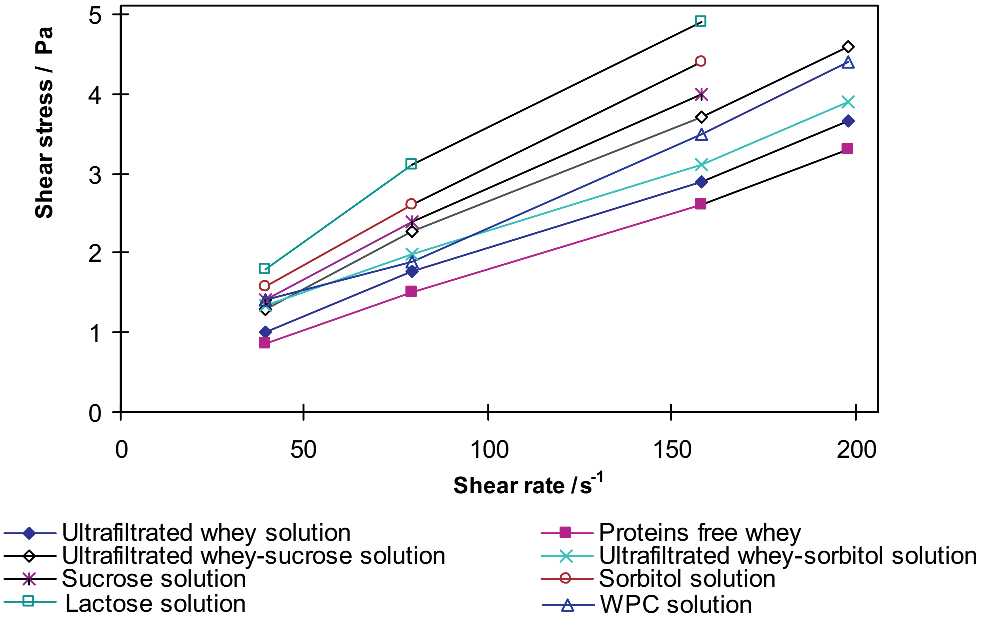

Fig. 2. Shear stress and shear rate relationship of model solutions with addition of hydrocolloid DIKO (0.4 %) after freezing

analysis of variance was performed. Variance analysis showed that all variance sources do indeed have an effect on rheological parameters.

In order to demonstrate the influence of model solution compositions, type of hydrocolloids and freezing process on the rheological properties of examined model systems, the analysis of variance and probability was executed (Table 6 and 7).

Fisher quotient values for composition of model solutions and type of hydrocolloids after freezing (F-values) were higher than the limiting values ($P \leq 0.05$) (Table 6), which means that the influence on rheological proper-

Table 5. Rheological characteristics of model systems with addition of 0.4 % CMC type (HVEP, DIKO and YO-EH) after freezing

Model system No.	Type CMC	Apparent viscosity Pa s	Flow index n	Consistency coefficient k / Pa s^n
1 (Ultrafiltrated whey)	HVEP	24.700	0.901	0.045
	DIKO	24.150	0.805	0.060
	YO-EH	30.100	0.870	0.059
2 (Proteins free whey)	HVEP	20.360	0.864	0.042
	DIKO	22.300	0.820	0.053
	YO-EH	28.050	0.810	0.063
3 (Ultrafiltrated whey	HVEP	25.800	0.837	0.0533
	DIKO	28.600	0.812	0.0647
+ sucrose)	YO-EH	40.700	0.904	0.0676
4 (Ultrafiltrated whey	HVEP	25.400	0.861	0.0503
	DIKO	24.400	0.843	0.0537
+ sorbitol)	YO-EH	37.800	0.839	0.0755
5 (Sucrose)	HVEP	26.400	0.890	0.0415
	DIKO	42.400	0.803	0.0981
	YO-EH	44.900	0.849	0.0879
6 (Sorbitol)	HVEP	28.000	0.840	0.0557
	DIKO	47.300	0.742	0.1131
	YO-EH	39.200	0.825	0.0913
7 (Lactose)	HVEP	30.100	0.737	0.0720
	DIKO	50.200	0.757	0.0900
	YO-EH	44.700	0.830	0.0890
8 (WPC)	HVEP	26.000	0.770	0.060
	DIKO	41.000	0.730	0.078
	YO-EH	35.000	0.830	0.050

Table 6. Analysis of variance for rheological parameters of examined hydrocolloids (0.4 %) before freezing

Source of variance	SQ	Degrees of freedom	MQ (Variances)	F	Probability
Between model systems	826.48	7	118.07	4.12	0.01166
Between hydrocolloids	608.36	2	304.18	10.61	0.00156
Error	401.23	14	28.66	–	–
Total	1836.08	23	–	–	–

$F_{0.05}$ (7/14) = 2.77; $F_{0.05}$ (2/14) = 3.74

Table 7. Analysis of variance for rheological parameters of examined hydrocolloids (0.4 %) after freezing

Source of variance	SQ	Degrees of freedom	MQ (Variances)	F	Probability
Between model systems	4356.72	7	622.39	6.56	0.00145
Between hydrocolloids	1285.69	2	642.85	6.78	0.00873
Error	1327.41	14	94.81	–	–
Total	6969.82	23	–	–	–

$F_{0.05}$ (7/14) = 2.77; $F_{0.05}$ (2/14) = 3.74

ties of examined model systems is statistically significant.

Conclusion

Coefficient consistency and flow behavior index values of CMC solutions are determined by Ostwald de Waele power-law.

When the amount of hydrocolloids was low (0.1 % w_t), model solutions prepared with ultrafiltrated whey and WPC, exhibited dilatant characteristics. By increasing the amount of hydrocolloid (0.2 and 0.4 % w_t) all the model solutions had a pseudoplastic flow (structural – viscous behavior).

As a result of interactions between whey proteins and hydrocolloids, or inorganic compounds and hydrocolloids, the viscosity of solutions prepared with ultrafiltrated whey was significantly lower then those of model solutions prepared with water.

Freezing process increased the viscosity of whey solution, while the viscosity of solutions prepared with water did not change significantly.

The results of variance analysis showed that all the examined model solutions had a significant influence on the rheological parameters.

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Reološka svojstva modelnih otopina karboksimetilceluloze i sirutke prije i nakon zamrzavanja

Sažetak

Hidrokoloidi, makromolekularni spojevi iz skupine ugljikohidrata, dodaju se mnogim prehrambenim proizvodima da bi se postigla određena reološka svojstva, spriječila sinereza, te povećala viskoznost i stabilnost proizvoda. U ovom je radu ispitivan utjecaj vrste i koncentracije hidrokoloida iz skupine karboksimetilceluloze na reološka svojstva modelnih otopina na bazi sirutke odnosno sirutkinih proteina, te saharoze, sorbitola i laktoze. Ispitivan je i utjecaj zamrzavanja na reološka svojstva modelnih otopina. Mjerenja su provedena rotacijskim reometrom Brookfield DV-III pri temperaturi od 20 °C. Rezultati su pokazali da svi ispitivani sustavi imaju nenewtonske značajke te da, ovisno o sastavu modelne otopine i udjelu hidrokoloida, pokazuju pseudoplastična ili dilatantna svojstva. Sve vrste CMC bitno su utjecale na povećanje viskoznosti ispitivanih otopina. Proces zamrzavanja nije jače utjecao na viskoznost modelnih otopina pripremljenih s vodom za razliku od onih pripremljenih sa sirutkom, koje su nakon zamrzavanja imale kudikamo veću viskoznost. Rezultati provedene analize varijancije pokazali su da svi ispitivani izvori varijacija (sastav modelne otopine, vrsta CMC i proces zamrzavanja) bitno utječu na reološke parametre.