

Structural changes in milk-protein concentrates with purple corn powder

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Abstract

Introduction. Milk protein concentrates derived from goat milk with purple corn powder represent promising multicomponent systems; however, the mechanisms underlying their structural rearrangement remain insufficiently understood. The aim of this study was to establish relationships between water state, protein–carbohydrate interactions, and the rheological behavior of these systems.

Materials and methods. Purple corn (*Zea mays* L.) powder and model milk-protein, water-plant, and dairy-plant systems were studied. Structural changes were assessed by FTIR spectroscopy in the range of 400–4000 cm^{-1} using spectral normalization, Voigt-profile fitting of overlapping bands, and integration of spectral regions. Rheological properties were determined using a *Kinexus Pro+* rheometer at 25 °C over a shear rate range of 0.1–100 s^{-1} .

Results and discussion. The incorporation of purple corn powder promoted water redistribution in the milk-protein system toward a more structurally associated state. In the 3600–3000 cm^{-1} range, the total band area increased from 147 ± 7 in the control to 273 ± 14 in the sample with powder, while the area of the low-frequency component S_2 increased from 86 ± 4 to 200 ± 10 . The share of S_2 rose from 58 ± 3 to $73 \pm 4\%$, the moisture redistribution index $R = S_2/S_1$ increased from 1.41 to 2.06, and the $\nu(\text{O-H})$ band maximum shifted from 3280 to 3245 cm^{-1} , indicating enhanced hydrogen bonding. The amide region of 1700–1500 cm^{-1} remained almost unchanged: 48.52 in the control and 47.51 in the sample with powder, confirming preservation of the protein matrix. At the same time, the area of the carbohydrate region of 1200–900 cm^{-1} increased from 28.3 to 57.6, i.e. 2.03-fold, and the protein-to-carbohydrate phase ratio index $I_{P/C}$ decreased from 1.71 to 0.83. This confirms the formation of a modified protein-carbohydrate matrix enriched with polysaccharide components. Both systems showed pseudoplastic flow behavior. The milk-protein concentrate with purple corn powder had higher effective viscosity throughout the shear rate range of 0.1–100 s^{-1} : 1.57–2.06-fold higher on the upward curve and 1.91–2.47-fold higher on the downward curve. The integral hysteresis loop parameter ΔS increased from 843.95 to 1399.32 Pa; however, the relative hysteresis area decreased from 69.79 to 62.08%, and the structural recovery coefficient increased from 30.21 to 37.92%. This indicates increased resistance to flow and improved structural recovery after shear loading.

Conclusions. The combination of FTIR spectroscopy and rheological analysis enabled the elucidation of the structure-forming mechanism of purple corn powder in milk protein concentrates. The scientific novelty lies in demonstrating the relationship between water redistribution, the formation of a protein–carbohydrate matrix, and enhanced thixotropic recovery of the system.

Introduction

Milk-protein concentrates containing plant ingredients are of considerable scientific interest as multicomponent matrices in which protein-carbohydrate interactions determine the water-holding capacity, textural properties, and rheological behavior of the product (Corredig et al., 2011; Dickinson, 1998). It has been established that the incorporation of plant-derived carbohydrate components into a dairy base modifies the microstructure and hydration properties of protein matrices as a result of intermolecular interactions (Abubaker et al., 2026; Babu et al., 2024). In milk-protein concentrates, plant ingredients play an important role in water redistribution and biopolymer compatibility, contributing to the stabilization of dairy matrices (Buldo et al., 2016; Rosa-Sibakov et al., 2016).

Milk-protein concentrates derived from goat milk are a promising basis for the development of multifunctional dairy-plant products, since the incorporation of plant ingredients makes it possible to modify their structural-mechanical and functional-technological properties. Among such components, purple corn (*Zea mays* L.) has attracted particular attention (Lao and Giusti, 2018; Salvador-Reyes and Clerici, 2020). It contains proteins, lipids, carbohydrates, dietary fiber, anthocyanins, flavonoids, and other phenolic compounds (Cristianini and Guillén-Sánchez, 2020; Deng et al., 2023), which determines its potential for use in food technologies. In addition, purple corn exhibits a broad spectrum of biological properties associated with its phenolic complex, as well as the possibility of concentrating its protein fraction while preserving antioxidant potential (Kim et al., 2023; Vilcacundo et al., 2020).

Previous studies on goat milk-protein concentrates have already established the technological feasibility of using purple corn powder and determined its optimal incorporation level within the range of 6–8% (Grek et al., 2024). However, the reported results mainly reflected changes in functional-technological properties and did not sufficiently reveal the structural prerequisites for these changes, particularly water redistribution and the nature of protein-carbohydrate interactions.

To elucidate the mechanisms underlying structural changes in milk-protein systems, the application of FTIR spectroscopy is appropriate, as this method enables proteins and other components to be studied directly within a complex food matrix without their prior isolation (Sadat and Joye, 2020). It has been shown that the interpretation of overlapping absorption bands in protein systems requires the use of spectral derivatives and peak-fitting procedures, which allow superimposed peaks to be resolved and the contribution of individual spectral components to be quantified (Sadat and Joye, 2020). At the same time, in food protein-carbohydrate systems, spectroscopically detected structural changes should be consistent with the macroscopic behavior of the matrix, particularly its viscosity-related properties, gelation capacity, and structural recovery after mechanical treatment (Sun et al., 2025; Xiang et al., 2025).

Therefore, combining FTIR spectroscopy with rheological analysis is an appropriate approach for evaluating structural changes in goat milk-protein concentrates with purple corn powder, for which the relationships between water state, protein-carbohydrate interactions, and rheological behavior remain insufficiently clarified. Thus, the aim of this study was to investigate structural changes in milk-protein concentrates with purple corn powder using FTIR spectroscopy and rheological analysis.

Materials and methods

Materials

Purple corn (*Zea mays* L.) powder (RS. E4513813N - KBEOSA), ground to a powdered state, was used in the study, along with model milk-protein and dairy-plant systems prepared on its basis.

Methods

FTIR spectra of the samples were recorded using a Nexus FTIR spectrophotometer (Nicolet, USA) in the wavenumber range of 400–4000 cm⁻¹. The spectra were acquired at a resolution of 1 cm⁻¹ and a spectral interval of 1 cm⁻¹. To improve the signal-to-noise ratio, seven scans were averaged for each spectrum. The samples were prepared as a pressed drop placed between KRS-5 windows, without the use of matrix media.

To ensure correct comparative analysis, the spectral curves were preliminarily intensity-normalized by scaling them to a common range of values while preserving the relative ratios between peaks within each spectrum. This normalization enabled comparison of the shape of the absorption bands and their relative contribution irrespective of the absolute signal level (Sadat and Joye, 2020).

Mathematical processing of the FTIR spectra was performed using peak fitting of overlapping absorption bands, which is an appropriate approach for interpreting complex protein spectra in multicomponent food systems (Sadat et al., 2019; Sadat and Joye, 2020). To identify the positions of hidden maxima, second-derivative spectral analysis was applied, which is widely used to resolve overlapping bands in the amide region of proteins (Susi and Byler, 1983; Dong et al., 1990). The Voigt profile, a convolution of Gaussian and Lorentzian functions, was used as the fitting function (Di Rocco et al., 2001). This approach makes it possible to simultaneously account for instrumental band broadening and the physical mechanisms involved in the formation of the band profile.

In general form, the signal was represented as a sum of Voigt profiles:

$$y = y_0 + \sum_1^n A \frac{2 \ln 2}{\pi^{3/2}} \frac{W_L}{W_G^2} \int_{-\infty}^{\infty} \frac{e^{-t^2}}{\left(\sqrt{\ln 2} \frac{W_L}{W_G} \right)^2 + \left(\sqrt{4 \ln 2} \frac{x - x_c}{W_G} \right)^2} dt \quad (1)$$

where y – experimentally measured absorption intensity; y_0 – baseline offset; n – number of fitted peaks; A_i – area of the i -th peak; $x_{c,i}$ – wavenumber position of the peak center; $W_{G,i}$ – full width at half maximum (FWHM) of the Gaussian component; $W_{L,i}$ – full width at half maximum (FWHM) of the Lorentzian component; t – integration variable.

The application of the Voigt function makes it possible to resolve overlapping spectral bands and to quantitatively assess the relative contribution of spectral components within the corresponding ranges based on the area of the fitted peaks (Byler and Susi, 1986; Sadat and Joye, 2020).

For the practical implementation of the fitting procedure, the spectrum was represented as a sum of Voigt profiles:

$$y = y_0 + \sum_{i=1}^n A_i V(x; x_{c,i}, W_{G,i}, W_{L,i}) \quad (2)$$

where $V(x)$ is the Voigt profile function, defined as a convolution of Gaussian and Lorentzian functions (Di Rocco et al., 2001).

After fitting, the integrated intensities of the spectral bands were determined within the specified wavenumber ranges. The integrated intensity over the interval from ν_1 to ν_2 was calculated as the area under the absorption curve:

$$A = \int_{\nu_1}^{\nu_2} I(\nu) d\nu \quad (3)$$

where $I(\nu)$ – normalized absorption intensity at the corresponding wavenumber ν .

The integral characteristics of individual spectral regions corresponding to vibrations of O–H, N–H, C–H, C=O, and C–O bonds were determined by numerical integration of the normalized spectral curves within the respective wavenumber intervals using the trapezoidal rule. The integrated area was denoted as S . Since the analysis was performed using normalized intensity without absolute calibration of optical density, the obtained values were dimensionless and were used for comparative evaluation of the samples.

The rheological properties of the milk-protein concentrates were determined using a *Kinexus Pro+* rotational rheometer (Malvern Instruments, UK) equipped with a Starch Paddle Plastic 2 Blade measuring geometry, which represents a coaxial-type system with a two-blade paddle stirrer (PC34 SL0007 SS) mounted on a vertical shaft. The prepared sample was placed into the measuring cylinder, after which the measuring element was immersed and the sample was held at 25 °C for 5 min to stabilize its structure.

Flow curves and effective viscosity dependences were recorded over a shear rate range of 0.1–100 s⁻¹ under a sequential increase in shear rate (upward curve), followed by its decrease within the same range (downward curve) (Vukić et al., 2018). The discrepancy between the upward and downward branches of the rheological curve was considered as evidence of the thixotropic behavior of the system. The degree of structural rearrangement under mechanical loading was quantified by the hysteresis loop area, defined as the area between the upward and downward curves (Alvarez et al., 2015; Ilić et al., 2024).

The integral parameter of the hysteresis loop, ΔS , was determined as the difference between the integral values under the upward (S_1) and downward (S_2) curves:

$$\Delta S = S_1 - S_2 \quad (4)$$

where S_1 and S_2 – integral values under the upward and downward curves, respectively (Ilić et al., 2024).

The integral values S_1 and S_2 were determined using the trapezoidal rule:

$$S = \sum_{i=1}^{n-1} \frac{\eta_i + \eta_{i+1}}{2} (\dot{\gamma}_{i+1} - \dot{\gamma}_i) \quad (5)$$

where η_i and η_{i+1} – effective viscosity values at two adjacent points of the curve, Pa·s; $\dot{\gamma}_i$ and $\dot{\gamma}_{i+1}$ – corresponding shear rate values, s⁻¹.

Since the integration was performed in the coordinates «effective viscosity-shear rate», the dimensionality of the integral parameters S_1 , S_2 , and ΔS corresponds to Pa.

For correct comparison of samples with different absolute viscosity levels, the relative hysteresis area, H , was additionally calculated:

$$H = \frac{\Delta S}{S_1} \cdot 100 \quad (6)$$

where H — relative area of the hysteresis loop, %.

The degree of structural recovery after mechanical treatment was evaluated using the structural recovery coefficient, K :

$$K = \frac{S_2}{S_1} \cdot 100 \quad (7)$$

where K – structural recovery coefficient, % (Zarzycki et al., 2019).

Statistical analysis

Data were expressed as means±standard deviations for triplicate determination. Statistical analysis was performed using Microsoft Excel. Differences were considered to be significant at validity of $\alpha=0.95$.

Results and discussion

Analysis of FTIR spectra of the milk-protein system

As a result of the FTIR spectroscopic study, absorption spectra of the samples were obtained in the range of 4000–400 cm^{-1} (Fig. 1). The spectral profiles were characterized by bands corresponding to stretching and bending vibrations of functional groups of protein and carbohydrate origin, as well as hydroxyl groups (Kong et al., 2007). For convenience of analysis, the spectra are presented in two fragments: the range of 4000–2000 cm^{-1} , which mainly covers stretching vibrations of O–H, N–H, and C–H bonds, and the range of 1800–400 cm^{-1} , which includes the region of carbonyl and amide bands of proteins, as well as vibration regions of the carbohydrate component.

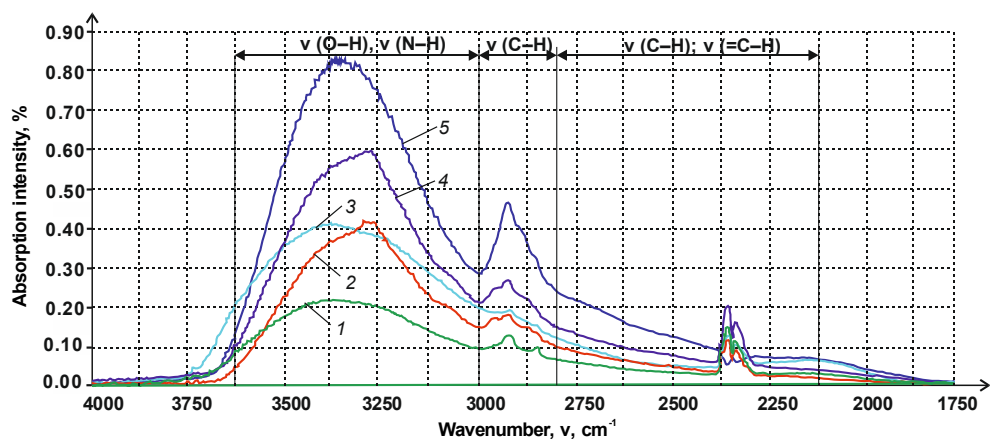
Further analysis was performed through a stepwise examination of individual spectral regions, taking into account the characteristic types of vibrations.

The spectral curves of sample 2, corresponding to the milk-protein concentrate, were characterized by intense amide bands, namely amide I and amide II, associated with vibrations of the peptide bond (Kong et al., 2007). The addition of purple corn powder to the milk-protein base, as in sample 5, was accompanied by changes in the intensity and relative ratio of individual absorption bands, which may indicate modification of the spectral profile of the protein–carbohydrate system and redistribution of intermolecular interactions (Sadat and Joye, 2020).

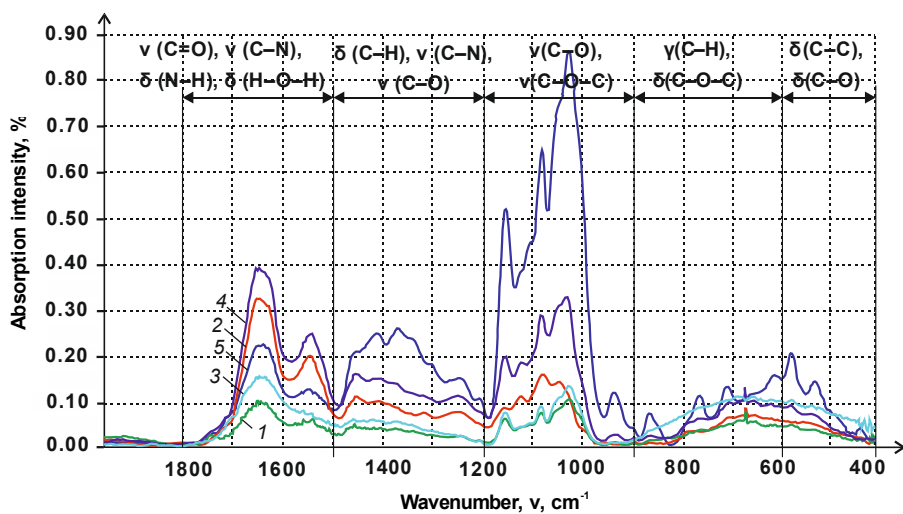
Detailed spectral analysis was started with the 3600–3000 cm^{-1} region, which covers stretching vibrations of hydroxyl and amide groups and is sensitive to the state of hydrogen bonding in the studied systems (Dong et al., 1990; Susi et al., 1983).

To evaluate moisture redistribution and the nature of intermolecular interactions, the total area of the broad $\nu(\text{O–H})$ and $\nu(\text{N–H})$ bands in the 3600–3000 cm^{-1} region was analyzed. Considering the overlap of the amide A band with the $\nu(\text{O–H})$ band, changes in this region were interpreted as an integral indicator of the overall level of hydrogen bonding in the system. To differentiate the degree of water association, this region was conditionally divided into two subregions: the high-frequency subregion S_1 (3600–3400 cm^{-1}), associated mainly with weakly bound, physically adsorbed water, and the low-frequency subregion S_2 (3400–3000 cm^{-1}), corresponding to more structurally associated, physicochemically bound water involved in intermolecular hydrogen interactions with amide ($-\text{NH}-$), carbonyl (C=O), and hydroxyl (O–H) groups of milk proteins and carbohydrate components of purple corn powder (Kong et al., 2007; Sadat and Joye, 2020).

The areas of the S_1 and S_2 subregions were determined by numerical integration of the normalized spectral curves. The obtained values were used to calculate their relative contribution to the total band area (ΣS) and to determine the moisture redistribution index, $R = S_2/S_1$ (Table 1).



a



b

Fig. 1. FTIR absorption spectra of the experimental samples in the regions of stretching vibrations: a) 4000–2000 cm^{-1} and b) 1800–500 cm^{-1} :

- 1 – purple corn powder;
- 2 – milk-protein concentrate without purple corn powder;
- 3 – water-plant system: water + purple corn powder;
- 4 – dairy-plant system: milk + purple corn powder;
- 5 – milk-protein concentrate with purple corn powder.

Table 1

Areas of the subregions of the 3600–3000 cm⁻¹ band and the moisture redistribution index in the studied samples (n = 3)

Sample No.	Sample	S ₁ (3600–3400)	S ₂ (3400–3000)	ΣS (3600–3000)	Share of S ₂ , %	R = S ₂ /S ₁
1	Purple corn powder	48±2	95±5	143±7	66±3	1.98
2	Milk-protein concentrate	61±3	86±4	147±7	58±3	1.41
3	Water-plant system	72±4	150±8	222±11	68±3	2.08
4	Dairy-plant system	83±4	175±9	258±13	68±3	2.11
5	Milk-protein concentrate with purple corn powder	97±5	200±10	273±14	73±4	2.06

A decrease in the wavenumber of the $\nu(\text{O-H})$ band maximum and an increase in the share of the low-frequency component S_2 indicate an increase in the degree of hydrogen bonding in the system (Sadat and Joye; Kong et al., 2007). In sample 5, corresponding to the milk-protein concentrate with purple corn powder, the band maximum shifted to 3245 cm⁻¹ compared with sample 2, the milk-protein concentrate, for which ν_{max} was approximately 3280 cm⁻¹. This decrease by 35 cm⁻¹ was accompanied by a 15% increase in the share of the low-frequency component S_2 and a 46% increase in the moisture redistribution index R . The strengthening of intermolecular interactions leads to a decrease in the vibrational energy of the O–H bond, which is spectroscopically manifested as a shift of the maximum toward lower wavenumbers.

In the presence of protein and carbohydrate components, water can form hydrogen bonds both with hydroxyl groups of polysaccharides and with amide and carbonyl fragments of proteins (Babu et al., 2024; Zhang et al., 2022). The 2.3-fold increase in the integrated area of the low-frequency component S_2 and the 1.9-fold increase in the total band area ΣS in sample 5 compared with sample 2 are consistent with the formation of a more developed hydration structure of the system.

Thus, changes in the spectral parameters in the 3600–3000 cm⁻¹ region correlate with moisture redistribution toward a more structurally associated state and indicate the involvement of biopolymer components, namely milk proteins and carbohydrates of purple corn powder, in the stabilization of bound water in the dairy-plant system. Water redistribution may contribute to the formation of a stabilized hydration structure, reduced mobility of free water, and improved structural stability of the system.

The 2800–1800 cm⁻¹ range was not included in the quantitative analysis because it did not contain pronounced characteristic bands directly related to the evaluation of the protein–carbohydrate component of the studied systems. This region was used for visual control of the spectral profile and for detecting possible extraneous or anomalous features.

The 3000–2800 cm⁻¹ range is dominated by stretching vibrations of aliphatic C–H bonds, mainly –CH₂– groups, with a possible contribution from –CH₃ fragments (Kačuráková et al., 2001). In this region, two main maxima were recorded for all samples near 2925 and 2854 cm⁻¹, and their positions did not change substantially, indicating the absence of noticeable transformation in the nature of aliphatic fragments and the predominantly quantitative character of the observed changes. At the same time, the intensity of the $\nu(\text{C-})$

H) bands in the samples containing purple corn powder was higher than that in the milk-protein concentrate without the additive: 1.3-fold for the dairy-plant system, 1.8-fold for the milk-protein concentrate with purple corn powder, and approximately 2.3-fold for the water-plant system. This is consistent with an increased proportion of polysaccharide components of purple corn powder, whose structure contains numerous C–H fragments (Babu et al., 2024; Zhang et al., 2022), and indicates quantitative enrichment of the system with organic biopolymers, which may contribute to structure strengthening, enhanced stability, and changes in rheological characteristics.

In the 1700–1500 cm^{-1} range, amide bands of proteins were recorded: amide I (1700–1600 cm^{-1}), associated with stretching vibrations of the C=O group of the peptide bond, and amide II (1600–1500 cm^{-1}), arising from N–H bending vibrations combined with C–N stretching vibrations (Sadat and Joye, 2020; Kong et al., 2007). The intensity of this region substantially depends on the presence of the milk protein phase. The most pronounced amide I maximum was observed near 1646 cm^{-1} in samples 2, 4, and 5, with peak heights of 0.183, 0.199, and 0.180, respectively, whereas in samples 1 and 3 it was lower, approximately 0.129 and 0.139. To quantitatively assess the contribution of the protein component, the areas of the 1700–1600 cm^{-1} subrange (amide I) and the 1600–1500 cm^{-1} subrange (amide II) were determined (Table 2).

Table 2

Areas of the amide region 1700–1500 cm^{-1} in the studied samples

Sample No.	Sample	S (1700–1600), amide I	S (1600–1500), amide II	ΣS (1700–1500), amides	S (1700–1600)/ S (1600–1500), amide I / amide II
1	Purple corn powder	13.41	13.50	26.91	0.99
2	Milk-protein concentrate	21.60	26.91	48.52	0.80
3	Water-plant system	14.36	13.70	28.06	1.05
4	Dairy-plant system	20.19	23.38	43.57	0.86
5	Milk-protein concentrate with purple corn powder	21.39	26.12	47.51	0.83

The highest total areas of the amide region were found for samples 2 and 5, amounting to 48.52 and 47.51, respectively. These values were almost 1.8-fold higher than that of sample 1 and 1.7-fold higher than that of sample 3. For sample 4, the total area was 43.57, which was approximately 10% lower than that of sample 2. The addition of purple corn powder to the milk-protein base practically did not change the total contribution of the amide region, as the difference between samples 2 and 5 was approximately 2%. This indicates the preservation of the protein matrix as the dominant structural component of the system (Sadat and Joye, 2020). The amide I/amide II ratio in the samples containing the dairy component ranged from 0.80 to 0.86, whereas in samples 1 and 3 it was close to 1.00, which is consistent with the lower proportion of the protein phase.

In the spectral interval of 1500–1200 cm^{-1} , bands associated with δ (C–H) bending vibrations and ν (C–N) and ν (C–O) stretching vibrations are formed; therefore, this region is informative for protein–carbohydrate systems (Kačuráková et al., 2001). In the milk-protein concentrate without the plant component (sample 2), bands near 1450–1415 and 1240–1230 cm^{-1} were recorded, corresponding to bending vibrations of –

CH₂–CH₃ groups of proteins and lipid fragments, as well as ν (C–N) vibrations of amide origin. In purple corn powder (sample 1) and the water-plant system (sample 3), bands at 1410–1380 and 1230–1200 cm⁻¹, characteristic of the polysaccharide phase, were more pronounced (Coates, 2006; Kačuráková et al., 2001). At the same time, the positions of the maxima did not change substantially, indicating preservation of the nature of the functional groups. In samples 4 and 5, this region showed the combined contribution of protein and carbohydrate components, whereas the enhancement of the bands in the 1410–1380 and 1240–1230 cm⁻¹ regions compared with sample 2 indicated an increase in the proportion of polysaccharides and the formation of a combined protein–carbohydrate structure. This is consistent with the increased complexity of the dispersed matrix and may contribute to improved structural stability, better consistency, and reduced susceptibility of the system to syneresis (Babu et al., 2024; Zhang et al., 2022).

In the spectral range of 1200–900 cm⁻¹, stretching vibrations of ν (C–O), ν (C–O–C), and glycosidic bonds of polysaccharides were recorded; therefore, this region is characteristic of the carbohydrate phase of the system (Kačuráková et al., 2001; Coates, 2006). The lowest area in this region was found for the milk-protein concentrate (sample 2), amounting to 28.3, which reflects the predominantly protein nature of the system (Table 3).

Table 3

Areas of the carbohydrate region 1200–900 cm⁻¹ in the studied samples

Sample No.	Sample	<i>S</i> (1200–900 cm ⁻¹)	Relative to sample 2, fold
1	Purple corn powder	65.4	2.31
2	Milk-protein concentrate	28.3	1.00
3	Water-plant system	49.7	1.76
4	Dairy-plant system	54.2	1.92
5	Milk-protein concentrate with purple corn powder	57.6	2.03

For purple corn powder, this value was 65.4, which was 2.31-fold higher than that of sample 2. For the water-plant system, it was 49.7, corresponding to a 1.76-fold increase; for the dairy-plant system, 54.2, corresponding to a 1.92-fold increase; and for the milk-protein concentrate with purple corn powder, 57.6, corresponding to a 2.03-fold increase. This indicates a systematic increase in the contribution of the carbohydrate phase upon the incorporation of purple corn powder.

To assess the ratio between the protein and carbohydrate components, the protein/carbohydrate index, *I*_{P/C}, was calculated (Table 4):

$$I_{P/C} = \frac{S(1700 - 1500)}{S(1200 - 900)}$$

The highest value of this index was found for the milk-protein concentrate, reaching 1.71, which indicates the predominance of the protein component. In contrast, for purple corn powder, the index was 0.41, reflecting the dominance of the carbohydrate phase. In samples 4 and 5, the index values were 0.80 and 0.83, respectively, indicating the formation of a modified protein–carbohydrate system. The increase in the area of the carbohydrate region in the samples containing purple corn powder is consistent with an increased proportion of hydrophilic polysaccharide components and creates prerequisites for the formation of a combined structural matrix, enhanced stability, reduced susceptibility to syneresis, and changes in rheological characteristics (Babu et al., 2024; Zhang et al., 2022).

Table 4

Protein-to-carbohydrate phase ratio index

Sample No.	Sample	$\Sigma(1700-1500)$	$\Sigma(1200-900)$	$I_{P/C}$
1	Purple corn powder	26.91	65.4	0.41
2	Milk-protein concentrate	48.52	28.3	1.71
3	Water + purple corn powder	28.06	49.7	0.56
4	Milk + purple corn powder	43.57	54.2	0.80
5	Milk-protein concentrate + purple corn powder	47.51	57.6	0.83

In the spectral range of 900–600 cm^{-1} , vibrations of polysaccharide origin associated with glycosidic bonds and the structural organization of the carbohydrate framework are localized (Coates, 2006; Kačuráková et al., 2001). The most pronounced profile in this region was characteristic of purple corn powder, corresponding to the predominance of the polysaccharide component.

In the water-plant system, the overall spectral structure was retained; however, the intensity of individual bands slightly decreased, which is consistent with hydration of polysaccharide chains and changes in intra- and intermolecular interactions (Babu et al., 2024; Zhang et al., 2022). The dairy-plant system and the milk-protein concentrate with purple corn powder were characterized by a further decrease in the prominence of individual peaks compared with the powder, indicating the incorporation of polysaccharide structures into the protein-carbohydrate matrix and restricted vibrational mobility of the corresponding fragments.

For the milk-protein concentrate without the plant component, the intensity in the 900–600 cm^{-1} range was minimal. Thus, changes in this region are consistent with the integration of the polysaccharide component into the milk-protein system and, together with the increased proportion of bound water, indicate the formation of a more ordered protein-carbohydrate structure, which may contribute to enhanced textural stability and improved rheological characteristics (Zhang et al., 2022).

In the spectral range of 600–400 cm^{-1} , low-frequency bending vibrations of C–C, C–O, and C–N bonds were recorded, reflecting general features of the spatial organization of macromolecular structures (Coates, 2006). For biopolymer systems, this region has an auxiliary, structure-confirming character.

The most differentiated spectral profile in this region was characteristic of purple corn powder, which is consistent with the complex polysaccharide nature of the plant raw material. In the water-plant system, the prominence of individual maxima decreased, whereas in the dairy-plant system and the milk-protein concentrate with purple corn powder, the profile became even less contrasting. This indicates the incorporation of polysaccharide structures into the combined protein-carbohydrate matrix and restriction of their vibrational mobility.

For the milk-protein concentrate without the plant component, the intensity in the 600–400 cm^{-1} range was minimal. Due to the low signal intensity and substantial band overlap, quantitative integration in this region was not performed; its analysis was qualitative and was used as additional confirmation of the structural changes identified in the high- and mid-wavenumber regions of the spectrum.

Rheological properties and thixotropic restructuring of milk-protein concentrates

According to the FTIR spectroscopy data, the incorporation of purple corn powder (*Zea mays* L.) was accompanied by changes in the state of water and enhanced structural interactions between the components of the milk-protein matrix. These changes were expected to be reflected in the rheological parameters of the system (Babu et al., 2024; Zhang et al., 2022). This is related to the degree of moisture binding and the strength of the spatial network of the milk-protein concentrate, which determine its resistance to flow and its recovery behavior after mechanical treatment. Therefore, at the next stage, the dependence of effective viscosity on shear rate was investigated, and the hysteresis loop was analyzed as an indicator of structural breakdown and recovery under shear loading, i.e., its thixotropic restructuring (Zarzycki et al., 2019).

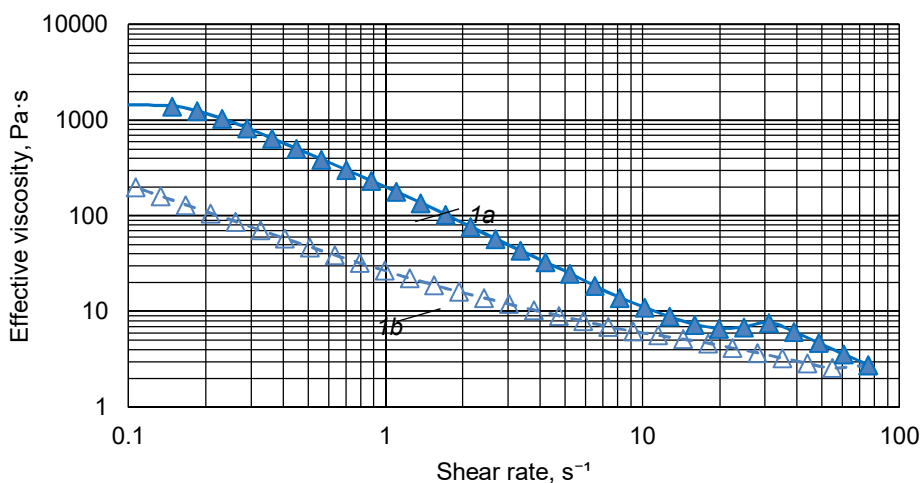
Analysis of the rheological parameters showed that the milk-protein concentrate (MPC) obtained by acid-rennet coagulation of goat milk proteins, both without the plant component and with the addition of purple corn powder (*Zea mays* L.), was characterized by a decrease in effective viscosity with increasing shear rate. This indicates the pseudoplastic flow behavior of the studied systems (Fig. 2a, b) (Nguyen et al., 2023; Vukić et al., 2018). At the same time, throughout the entire investigated shear rate range, the MPC with purple corn powder exhibited higher effective viscosity values compared with the control.

For a generalized assessment of the effect of purple corn powder on the rheological behavior of MPC, the mean effective viscosity values were additionally analyzed in three shear rate ranges: 0.1–1, 1–10, and 10–100 s⁻¹ (Table 5). It was found that, on the upward curve, the effective viscosity of MPC with purple corn powder exceeded that of the control by 1.57–2.06 times, with the fold increase rising as the shear rate increased. A similar trend was observed on the downward curve, where the effective viscosity of the enriched sample was 1.91–2.47 times higher than that of the control.

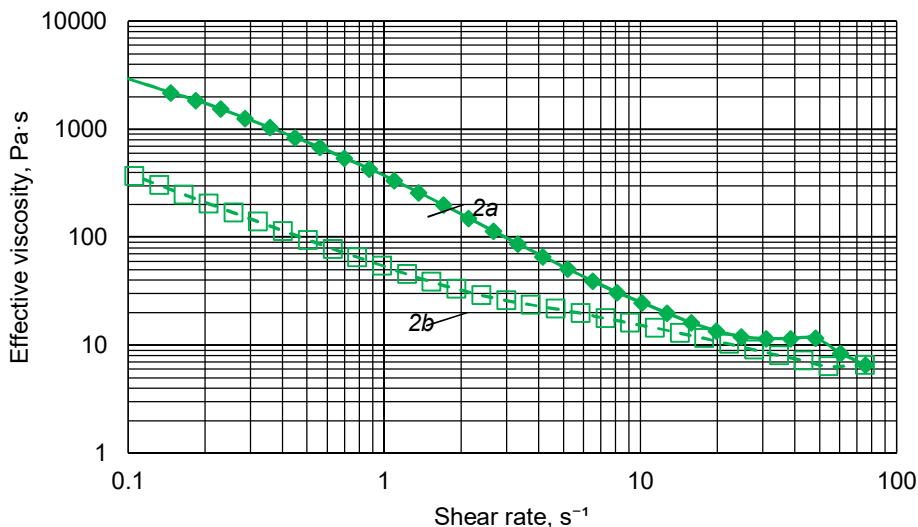
Table 5
Mean effective viscosity values of MPC in different shear rate ranges

Curve direction	Shear rate range, s ⁻¹	Effective viscosity of MPC, Pa·s – control	Effective viscosity of MPC, Pa·s – with purple corn powder	Increase in effective viscosity, fold
Upward	0.1–1	737.00	1155.76	1.57
	1–10	69.26	133.56	1.93
	10–100	6.61	13.59	2.06
Downward	0.1–1	87.72	167.79	1.91
	1–10	12.50	27.25	2.18
	10–100	3.92	9.66	2.47

Thus, the incorporation of purple corn powder into MPC exhibited structure-forming properties, resulting in increased resistance to flow. This effect was associated with the binding of part of the free moisture, enhancement of intercomponent interactions, and formation of a more densely organized milk-protein matrix, as confirmed by the FTIR spectroscopy results (Babu et al., 2024; Zhang et al., 2022).



a



b

Fig. 2. Effective viscosity of milk-protein concentrates as a function of shear rate: a – MPC (control); b – MPC with purple corn powder (*Zea mays* L.);
 1 – milk-protein concentrate (control); 2 – milk-protein concentrate with purple corn powder;
 a – upward curve; b – downward curve.

The discrepancy between the upward and downward curves (Fig. 2 a,b) indicates the thixotropic character of structural rearrangement in MPC under shear loading. During the increase in shear rate, gradual breakdown of the internal structural network of the milk-protein system occurred, whereas during the downward curve, recovery of the structural network was incomplete, resulting in the formation of a hysteresis loop. To quantitatively assess this rearrangement, the integral hysteresis loop parameter ΔS , the relative hysteresis area H , and the structural recovery coefficient K were determined (Table 6).

Table 6

Parameters of thixotropic restructuring of milk-protein concentrates

Milk-protein concentrate	S ₁ , Pa – upward curve	S ₂ , Pa – downward curve	ΔS, Pa – hysteresis loop area	H, % – relative hysteresis area	K, % – structural recovery coefficient
Control	1209.36	365.40	843.95	69.79	30.21
With purple corn powder	2254.18	854.86	1399.32	62.08	37.92

It was found that the ΔS value for MPC with purple corn powder was 1399.32 Pa, whereas for the control it was 843.95 Pa. At the same time, compared with the control, the relative hysteresis area decreased by 11.05%, while the structural recovery coefficient increased by 25.52% (Nguyen et al., 2023; Zhang et al., 2022). The obtained results indicate that the incorporation of purple corn powder reduces the intensity of structural changes under shear forces and improves structural recovery after mechanical treatment (Guan et al., 2020). Thus, purple corn powder in MPC exhibits pronounced structure-forming properties.

Conclusions

According to the FTIR spectroscopy data, the incorporation of purple corn powder caused structural rearrangement of the milk-protein concentrate, manifested by water redistribution toward a more bound state. In the high-wavenumber region of 3600–3000 cm^{-1} , the share of the low-frequency component increased 1.46-fold, the moisture redistribution index reached 2.06, and the maximum of the $\nu(\text{O-H})$ band shifted by 35 cm^{-1} toward lower wavenumbers, indicating enhanced hydrogen bonding and intercomponent interactions in the system.

In the mid-wavenumber regions of the FTIR spectrum, the formation of a modified protein-carbohydrate matrix was confirmed. In the amide region of 1700–1500 cm^{-1} , the protein-to-carbohydrate phase ratio index decreased 2.3-fold, whereas the integrated area of the carbohydrate region of 1200–900 cm^{-1} increased 2.03-fold. In the 3000–2800 cm^{-1} region, the integrated intensity increased 1.82-fold without changes in the positions of the maxima, while the spectral regions of 900–600 and 600–400 cm^{-1} qualitatively confirmed the integration of polysaccharide structures into the milk-protein matrix.

Rheological analysis showed that the incorporation of purple corn powder was accompanied by enhancement of the structural-mechanical properties of milk-protein concentrates, as manifested by an increase in effective viscosity throughout the entire investigated shear rate range. The experimental samples were characterized by a lower relative hysteresis area and a higher structural recovery coefficient compared with the control, indicating reduced intensity of structural changes under shear loading and improved recovery capacity of the system after mechanical treatment. Taken together, the obtained results confirm the pronounced structure-forming properties of purple corn powder in the composition of milk-protein concentrate.

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