

Обґрунтовано доцільність введення дієтичних добавок на основі хелатного комплексу в харчові системи та забезпечення рівномірності їх розподілення. Мета полягає у вивченні рівномірності розподілу мікроелементів, сорбованих на макромолекулах-носіях, та дослідженні впливу добавки на функціонально-технологічні властивості тістових заготовок. Предметом дослідження були тістові заготовки із дріжджового, листкового та прісного тіста з введеною добавкою на основі хелатного комплексу.

Проведено дослідження розподілення дієтичної добавки з хелатним комплексом в об'ємі харчової системи та її впливу на функціонально-технологічні властивості досліджуваних харчових систем. При цьому використовували методи ядерного магнітного резонансу, електронного парамагнітного резонансу, низькотемпературний калориметричний метод та реологічні методи дослідження.

Встановлено, що порошкоподібна добавка зі стабілізованим хелатом впливає на рухливість й взаємодію молекул води з оточенням у тістових заготовках. Доведено, що внесення хелату металу у вигляді порошкоподібної харчової добавки дозволяє забезпечити рівномірність розподілу металу за об'ємом досліджених тістових заготовок. Визначено, що для зразків тіста за умови внесення дієтичної добавки на основі хелатного комплексу відбувається збільшення кількості невимороженої води порівняно з контролем. Встановлено свідчить про збільшення кількості зв'язаної води за умови внесення добавки. Відзначено, що стабілізований хелат знаходиться в основному у насиченому стані, оскільки вода тістових заготовок знаходиться у «зв'язаному» стані. Встановлено, що в тістових заготовках з дієтичною добавкою на основі хелатного комплексу відбувається зміна пружних властивостей. Результат пояснюється зміною співвідношення вільна – зв'язана вода у досліджуваних харчових системах.

Доведена перспективність використання порошкоподібної добавки зі стабілізованими хелатами металів в технологіях збагачення харчових продуктів. Відзначено, що перспективою подальших досліджень є визначення розподілу по об'єму харчових систем інших мікроелементів хелатів, які являють основу дієтичної добавки

Ключові слова: тістові заготовки, добавка на основі хелатного комплексу, об'ємне розподілення мікроелементу

UDC 664.651.2: 664.48

DOI: 10.15587/1729-4061.2018.140134

STUDY OF MICROELEMENT DISTRIBUTION UNIFORMITY IN A BULK OF DOUGH ENRICHED WITH DIETARY SUPPLEMENTS

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1. Introduction

The lack of macro- and micronutrients in human nutrition is one of the main and topical issues of universal significance. Lack of microelements often causes many diseases [1]. To maintain normal level of micronutrients, it is necessary

to eat varied and healthy food. However, given the difficult economic situation in Ukraine and the world as a whole, majority of population cannot afford exclusively natural and adequate food products. That is why the most simple and available way to improvement of quality of everybody's diet is food enrichment with additional vitamins and minerals [2].

2. Literature review and problem statement

Enrichment of food products is based on biological, medical and technological requirements [3]. The most difficult-to-obtain micronutrients are necessarily to be used. These include calcium, iodine, selenium, potassium, magnesium, iron, vitamins of A, C, E and B groups [4]. At the same time, there should be no threat of excess micronutrients, as it can provoke a number of violations of the human body activity [5].

The threat of growth of risk of spreading diseases caused by lack of micronutrients in the human body motivates scientists around the world to study [6] causes of micronutrient deficiency [7]. This topic has been reflected in many scientific papers. However, as scientists point out, «importance of mineral substances (especially microelements) and the necessity of mandatory entry of these minerals into the human body are definitely underestimated» [8].

It should be noted that microelements of inorganic origin are usually poorly assimilated in the human body and just maintain their certain content. From the point of view of solving the problem of assimilation of micro elements, one of the most rational ways of introducing these nutrients into the human body is their taking with food products. The choice of carriers of chemical elements and uniform distribution of these carriers in products in conditions of industrial production remains to be a pressing problem of the technology of enriched food products [9]. Microelements are introduced to food products by dissolving in a liquid phase [10], dry mixing with food ingredients [11], applying special coating [12], etc. Ensuring homogeneity of distribution of microelements in a bulk of a food product is the issue that remains unresolved by any of the above-mentioned methods. The use of dietary supplements based on chelate complexes in food technologies is a promising method from this point of view as it ensures the issue solution [13]. This line is new in food technologies. Therefore, experimental studies of distribution of dietary supplement microelements based on chelating complexes and their effect on functional and technological properties of various food systems are topical.

3. The aim and objectives of the study

The study objective was to elucidate uniformity of distribution of microelements sorbed by macromolecular carriers and study the effect of supplements on functional and technological properties of dough preparations. This will make it possible to control functional and technological properties of dough preparations with dietary supplements based on chelate complexes in food enrichment technologies.

To achieve this objective, the following tasks were solved:

- to study interaction of water molecules with each other and environment in the food system (dough preparations) with application of the nuclear magnetic resonance (NMR) method;
- to estimate homogeneity of volume distribution of supplements based on magnesium-containing chelating complex in the food system (dough preparations) with application of the electron paramagnetic resonance (EPR) method;
- to study the change of functional and technological properties of food systems (dough preparations) containing the chelating complex with application of the low-temperature calorimetric method;

- to study the change of functional and technological properties of food systems (dough preparations) containing the chelating complex with application of rheological methods.

4. The study material and methods

The study of distribution of powdered dietary supplements based on the chelating complex in the volume of food system and effect of supplements on functional and technological properties of the studied food system was carried out in four stages according to the method applied. The following analysis methods were used: NMR, EPR, low-temperature calorimetric method and rheological study methods. These methods were aimed at obtaining of original tomograms that carry information on molecular mobility of aqueous medium of the rough-dispersed system and metal ion distribution in its volume. The metal ion distribution of which was studied was Mn^{2+} . The Mn^{2+} ion was also used as a spin marker.

The food raw materials with an introduced supplement based on the Mn^{2+} chelating complex represented the food systems that were the subject of studies by rheological, and low-temperature calorimetry, NMR and EPR methods:

- model system No. 1 (yeast dough) with moisture content of 0.43 rel. un.;
- model system No. 2 (puff pastry dough) with moisture content of 0.29 rel. un.;
- model system No. 3 (unleavened dough) with moisture content of 0.35 rel. un.

The above model systems without supplements based on Mn^{2+} chelating complex were used as control systems.

A laboratory pulse NMR radio spectrometer with fixed frequency of 16.5 MHz was used in the NMR studies [14].

To study system water of food raw materials and products, low-temperature calorimetric method was used [15].

Rheological studies were carried out using a Tolstoy plane-parallel elastoplastometer [16].

A radiospectrometer operating at a fixed wavelength $\lambda = 3.2$ cm was used to record EPR spectra. The spectra were recorded as the first derivative of VHF energy (E) absorption by the studied paramagnetic substance during scanning of constant magnetic field (H) [17].

The methods used to test dough preparations with dietary supplements based on chelate complexes are described in detail in [18].

5. Results obtained in studying the properties of dough preparations enriched with dietary supplements

Stage 1. Interaction of water molecules contained in food systems (dough preparations) with each other and environment was studied by the NMR method.

A precision weighted sample of dough (100 g) containing a daily human body demand of micronutrient (K^+) simulated by Mn^{2+} ion was taken for the studies. For NMR studies, the sample was divided into 8 conditional portions of a parallelepiped form. Three samples were taken from each portion and sent for NMR analysis.

The results of NMR studies are shown in Table 1.

The data shown in Table 1 represent spin-spin (T_2) and spin-lattice (T_1) relaxation time. The time of spin-spin relaxation is responsible for interaction of water molecules

with each other. The time of spin-lattice relaxation is the time of relaxation with other molecules, i.e. the powdered dietary supplement molecules in this case [19].

Table 1

The results of NMR studies of unleavened, puff pastry and yeast dough

T_n, s	Unleavened dough		Puff pastry dough		Yeast dough	
	control	w/supplement	control	w/supplement	control	w/supplement
T_1	0.03	0.05	0.025	0.008	0.06	0.065
T_2	0.013	0.004	0.01	0.03	0.016	0.015

Stage 2. Homogeneity of volume distribution of a supplement based on Mn containing chelating complex introduced into the food system (dough preparations) was estimated by the EPR method. Mn^{2+} ion was used as a spin marker [20].

The investigated food system was tightly packed in a container of a parallelepiped form (Fig. 1) with overall dimensions of $10 \times 60 \times 60$ mm. The container was conventionally sectioned into elementary portions with characteristic dimensions of $10 \times 20 \times 20$ mm (Fig. 1). A sample was taken from each elementary portion, a_{ij} (where $i=0, 1, 2; j=0, 1, 2$) to study by the EPR method.

Estimation of homogeneity of volume distribution of a supplement based on the Mn containing chelating complex introduced to the food system was performed by the method of EPR-spin markers as shown in Fig. 2–4.

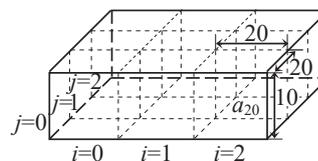


Fig. 1. The container for the food systems studied by the EPR method

Fig. 2, a, 3, a, 4, a show values of areas under the spectrum represented by a broad single line proportional to the amount of system water that does not dissolve ionic salt.

Fig. 2, b, 3, b, 4, b show tomograms of these areas. The given data were normalized for the maximum value of area under a single line for the sample of food raw material. It is clear from the results that the areas under the EPR spectrum for the raw material from different elementary volumes with overall dimensions of $10 \times 20 \times 20$ mm differ by no more than 4...5 %, that is, within the error limits. The results demonstrate homogeneity of supplement distribution in the volume of the food systems under study.

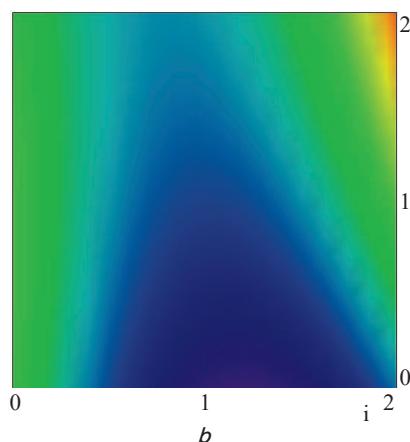
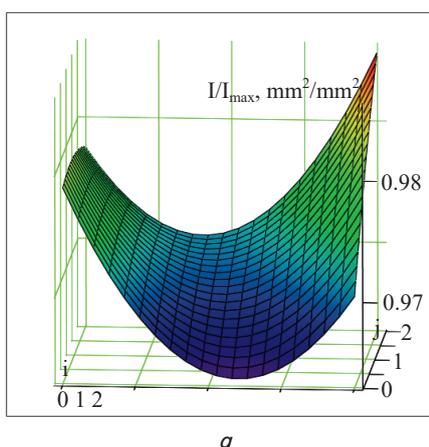


Fig. 2. Results of EPR studies: areas under the spectrum at different coordinates of the sample of the model food system No. 1 (yeast dough) (a); the surface tomogram (b)

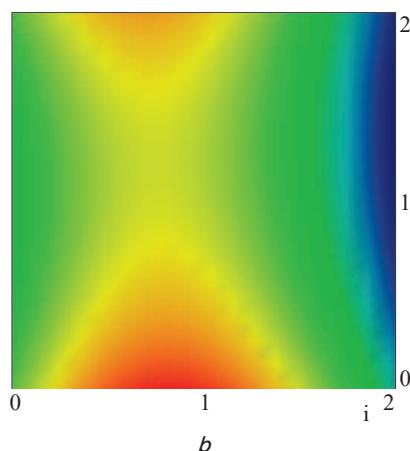
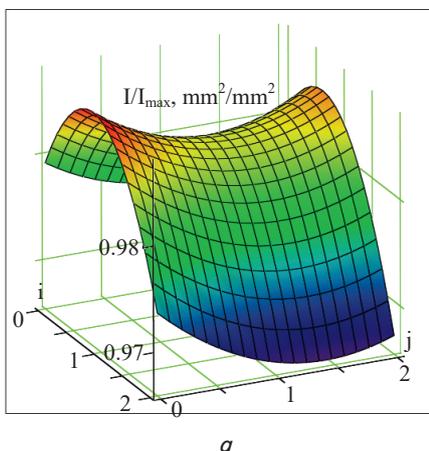


Fig. 3. Results of EPR studies: areas under the spectrum at different coordinates of the sample of the model food system No. 2 (puff pastry dough) (a); the surface tomogram (b)

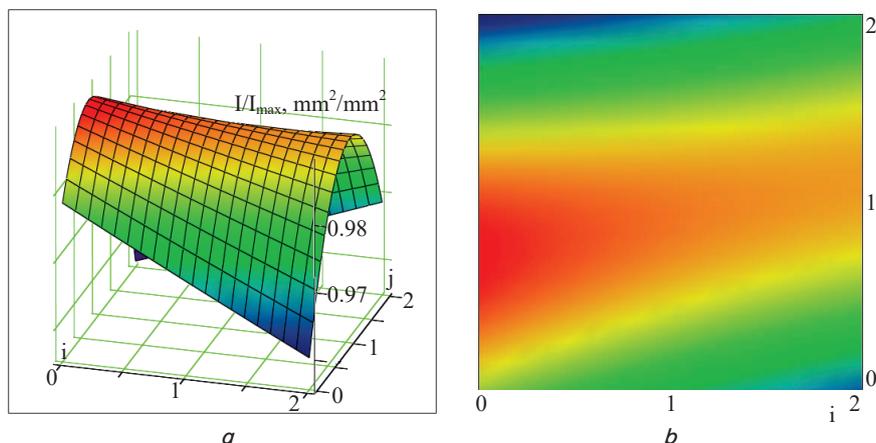


Fig. 4. Results of EPR studies: the areas under the spectrum at different coordinates of the sample of the model food system No. 3 (unleavened dough) (a); the surface tomogram (b)

Stage 3. Low-temperature calorimetry study of the state of water of the food systems (dough preparations) containing a supplement based on the chelating complex. Fig. 5 shows the thermograms obtained for the studied food systems with a supplement (2) and without it (1).

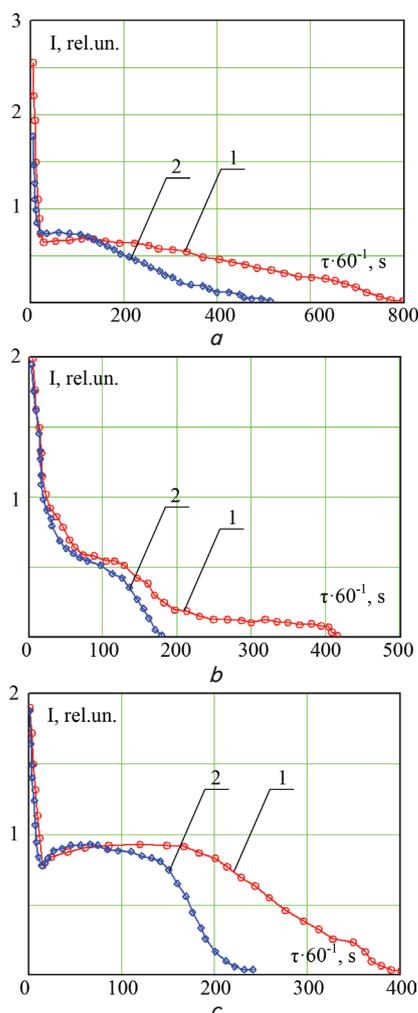


Fig. 5. Thermograms obtained by the calorimetric method at calorimeter temperature of $-12\text{ }^{\circ}\text{C}$ for model food systems: No. 1 (yeast dough) (a); No. 2 (puff pastry dough) (b); No. 3 (unleavened dough) (c)

Table 2 shows relative fractions of system water in the studied samples. The data were obtained by the low-temperature calorimetric method at an equilibrium temperature of the calorimeter of $-12\text{ }^{\circ}\text{C}$. It should be noted that there were two relative mole fractions of system water, A_i , at this thermostat temperature [21]. Denote them as A_{k0} and A_{k1} where index «0» corresponds to the fraction of the system water for which there was a phase transition of the first type at the specified calorimeter temperature. Index «1» corresponds to the fraction of system water remained in a liquid state.

The sum of relative molar fractions is equal to one:

$$\sum_{i=0}^m A_i = 1, \tag{1}$$

where m is determined by the method and procedure of system water study. Accordingly, for calorimetric studies at equilibrium calorimeter temperature of $-12\text{ }^{\circ}\text{C}$, $m=1$, the quantity of m is 2.

Table 2

Relative fractions of chilled (A_{k0}) and nonchilled (A_{k1}) water in model food systems at calorimeter temperature of $-12\text{ }^{\circ}\text{C}$

Sample	A_{k1} , kg/kg	A_{k0} , kg/kg
Control sample:		
Model system No. 1 (yeast dough)	0.37	0.63
Model system No. 2 (puff pastry dough)	0.66	0.34
Model system No. 3 (unleavened dough)	0.47	0.53
With a supplement based on the chelating complex:		
Model system No. 1 (yeast dough)	0.64	0.36
Model system No. 2 (puff pastry dough)	0.77	0.23
Model system No. 3 (unleavened dough)	0.68	0.32

It is seen from Table 2 that there was a 1.75 time increase in the fraction of system water for which there was no phase transition of the first type at the calorimeter temperature of $-12\text{ }^{\circ}\text{C}$ for food system No. 1 (yeast dough). There was a 1.16 time increase in the fraction of system water for food system No. 2 (puff pastry dough) and a 1.45 time increase was observed for food system No. 3 (unleavened dough).

Stage 4. Rheological studies of changes in structural and mechanical properties of food systems (dough preparations)

with introduced supplements based on the chelating complex. Preparations of yeast and unleavened dough with supplements based on the chelating complex were studied. Respective dough preparations without supplements were used as control samples.

The puff pastry dough was not studied by the rheological method at all because its composition and inhomogeneous consistency (caused by presence of layers of margarine/butter in dough) is not subject to this type of study. Input parameters for this study stage are shown in Table 3.

The obtained structural and mechanical indicators for the studied food systems are shown in Table 4.

Fig. 6 shows the change in time of shear stress for samples of the studied food systems obtained with the help of the Tolstoy elastoplastometer.

Table 3

Input parameters for rheological studies of dough preparations

Input parameters:	Dough preparations:			
	Control for dough No. 1	Dough No. 1	Control for dough No. 3	Dough No. 3
Load, kg	0.002	0.002	0.002	0.002
Sample height, m	0.008	0.008	0.008	0.008
Scale division value, $\times 10^{-4}$ m	5	5	5	5

Table 4

Structural and mechanical indicators of dough preparations with and without supplements

Indicator name	Dough preparations:			
	Control for dough No. 1	Dough No. 1	Control for dough No. 3	Dough No. 3
Reversible strain, 10^{-3}	0.04	0.01	0.06	0.04
Irreversible strain, 10^{-3}	0.01	0.04	0.02	0.05
Total strain, 10^{-3}	0.04	0.05	0.08	0.08
Shear stress, Pa	13.08	13.08	13.08	13.08
Relative resilience, %	19.27	13.19	21.74	25.60
Relative plasticity, %	20,11	86,54	20,96	54,22
Relative elasticity, %	60.61	27.0	57.30	20.18

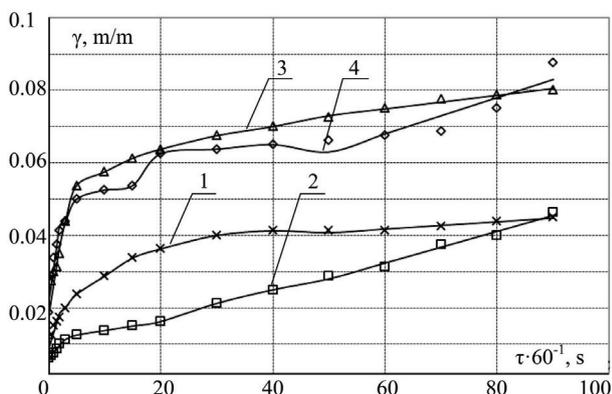


Fig. 6. The change of shear stress in time for model food systems: control sample of dough No. 1 (yeast dough) (1); sample of dough No. 1 (yeast dough) (2); control sample of dough No. 3 (unleavened dough) (3); sample of dough No. 3 (unleavened dough) (4)

6. Discussion of results obtained in the study of properties of dough preparations enriched with dietary supplements

Analysis of the results obtained in the NMR studies has shown the following. In unleavened dough, interaction of water molecules with each other in a sample called «control» (without a dietary supplement based on the chelating complex) was weaker than that in a sample of dough with supplement (0.004 s) and was 0.013 s. Regarding spin-lattice relaxation time, interaction of dough with other molecules (environmental molecules) was decreased in unleavened dough with a supplement and the spin-lattice relaxation time was increased: from 0.03 to 0.05 s.

For puff pastry dough, the results of the NMR study were opposite to the results obtained for unleavened dough. For puff pastry dough, interaction of water molecules with each other (identified by the time of spin-spin relaxation) manifested itself to a greater extent for a dough sample with no supplement. At the same time, spin-spin relaxation time was 0.01 s and if a supplement was added, it was 0.03 s. Concerning dough interaction with other molecules, it was greater in the puff pastry dough sample, exactly with supplement (0.008 s), compared with the sample without supplement (0.025 s).

For yeast dough, the results of NMR studies were similar to those obtained for unleavened dough. In yeast dough, interaction of water molecules with each other in a sample without supplement has manifested itself to a lesser extent (0.016 s) compared to a dough sample with the same supplement (0.015 s). Regarding spin-lattice relaxation time, interaction of dough with other (environmental) molecules decreased with supplement introduction and the spin-lattice relaxation time has increased from 0.06 to 0.065 s.

It should be noted that the smaller the spin-spin relaxation time (T_2), the stronger interaction of water molecules with each other. Accordingly, the less the spin-lattice relaxation time (T_1) the stronger water interaction with environment. In this case, 'environment' refers to air molecules, particles of a powdered dietary supplement based on the chelating complex, fat, protein and the like. This result is one of the starting conditions during formation of functional and technological properties of dough preparations enriched with dietary supplements based on the chelate complexes.

The studies by the method of EPR-spin markers have shown that the samples did not show cleavage of ultra-fine structure of the EPR spectrum. This means that the stabilized chelate was mainly in a saturated state in this state of samples since the solvent (water) in the amount introduced into the dough preparation system was in a «bound» state. Such a state corresponds to a decrease in level dielectric permeability of medium which results in a loss of chelate solubility.

The EPR studies have shown that amount of supplement in the volume of food system was not more than 4...5%. This result proves homogeneity of the dietary supplement microelement (Mn) distribution in the volume of dough preparations. The aim of further studies is determination of distribution of other microelements (other than Mn) of the dietary supplements based on the chelate complex in the volume of food systems.

The results obtained by the low-temperature calorimetric method show that addition of dietary supplements based on the chelating complex leads to a decrease in amount of chilled

system water, that is, to an increase in the fraction of non-chilled water at a specified calorimeter temperature for all model food systems under study. The amount of nonchilled water increased in 1.7 times for yeast dough, 1.2 times for puff pastry dough and 1.4 times for unleavened dough. This indicates an increase in amount of bound water provided a supplement is introduced. The established result serves as a reference condition in determination of the dough preparation storage conditions. It should be noted that dough preparations with dietary supplements based on the chelating complex have an extended shelf life compared to the dough preparations without supplements. However, when establishing the recommended shelf life, microbiological properties of above food systems should be taken into account which requires further studies.

It is evident from the results obtained on the Tolstoy elastoplastometer that reversible strain in unleavened dough was greater in the control sample amounting to $0.04 \cdot 10^{-3}$ compared with the sample with a supplement where it was $0.01 \cdot 10^{-3}$. In yeast dough, situation with the reversible strain was the same: $0.06 \cdot 10^{-3}$ in the control sample and $0.04 \cdot 10^{-3}$ in the sample with a supplement. Irreversible strain in unleavened dough was inversely proportional to reversible strain in yeast dough. Irreversible strain in the control sample was also smaller ($0.02 \cdot 10^{-3}$) than in the sample with a supplement ($0.05 \cdot 10^{-3}$). General strain in samples of the same dough type was almost the same: $0.04 \cdot 10^{-3}$ in the control sample of unleavened dough, $0.05 \cdot 10^{-3}$ in the sample with supplement and $0.08 \cdot 10^{-3}$ in yeast dough for both cases. Shear stress was everywhere the same: 13.08 Pa because all values in the formula will be the same for calculation of shear stress in all samples. Relative elasticity was determined in samples. Accordingly, the highest relative elasticity was in a sample of yeast dough: 25.60 %. The control sample of yeast dough also had a high relative elasticity: 21.74 %. As for unleavened dough, relative elasticity was much larger in the control sample: 19.27 % compared to the sample with supplement: 13.19 %. Relative plasticity both in unleavened and yeast dough was greater in samples containing supplement. For example, it was up to 86.54 % in unleavened dough and 54.22 % in yeast dough. In control samples, it was 20.11 % and 20.96 %, respectively. On the contrary, relative elasticity was greater in control samples in both cases. For example, it was 60.61 % in unleavened dough and 57.3 % in yeast dough. It was 27.0 % and 20.18 %, respectively, in samples with supplements.

Rheological studies of dough preparations with and without supplements evidenced changes in elastic properties of the food systems under study with dietary supplements based on the chelating complex. For example, there was a 4 and 1.5 time decrease in reversible strain, a 4 and 2.3 time increase in relative plasticity, a 2.1 and 2.8 time decrease in relative elasticity, respectively for unleavened and yeast dough with supplements. The above was caused by the change of the free to bound water ratio in dough preparations with supplements as confirmed by NMR, EPR and low-temperature calorimetric studies.

Thus, the studies of functional and technological properties of dough preparations with supplements have established

that introduction of supplements contributed to the change of the chilled to nonchilled water ratio and elastic properties of the food systems under study.

Limitation of this work results consists in that the studies have proved homogeneity of distribution of dietary supplements in the volume of semi-finished food products (dough preparations) but only for Mn^{2+} microelement. Hence, the issue of distribution of other microelements of dietary supplements based on the chelating complexes in dough preparations remains to be solved in further studies. Also, the task of further studies consists in determination of distribution of dietary supplement microelements in other semi-finished and finished food products. In this case, the applied procedures require adaptation to properties of the studied food systems and the chelates as the basis of dietary supplements.

7. Conclusions

The study of functional and technological properties of dough preparations enriched with dietary supplements based on the chelating complex with the use of NMR, EPR, low-temperature calorimetric and rheological methods have resulted in the following.

1. It was established that the spin-spin and spin-lattice relaxation time has increased by 3 and 1.7 times, respectively, for unleavened dough. A 3-fold reduction of spin-spin and spin-lattice relaxation time was observed for puff pastry dough. The spin-spin relaxation time has decreased by 1.1 times and the spin-lattice relaxation time has increased by 1.1 times for yeast dough. It was pointed out that the obtained results determine properties of water of the dough preparations before its interaction with air molecules, particles of a powdered dietary supplement based on the chelating complex, fats, proteins, etc.

2. It was established that amount of supplement introduced in the food system volume varied by no more than 4–5 %, which proved the homogeneity of distribution of dietary supplements based on the chelating complex containing Mn in the volume of dough preparations. It was pointed out that stabilized chelate was generally in a saturated state since water of the dough preparations was in a «bound» state.

3. It was determined that for the samples of dough with an introduced dietary supplement based on the chelating complex, there were the following values of increase in amount of nonchilled water compared with control samples: 1.7 times for yeast dough, 1.2 times for puff pastry dough and 1.4 times for unleavened dough. The established data indicate an increase in amount of bound water provided a supplement is introduced.

4. A change in elastic properties was established in dough preparations with a dietary supplement based on the chelating complex. There was a 4 and 1.5 time decrease in reversible strain, a 4 and 2.3 time increase in relative plasticity and a 2.1 and 2.8 time decrease in relative elasticity in samples of unleavened and yeast dough, respectively. The established data are explained by the change of free to bound water ratio in the studied food systems.

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