DETERMINATION METHODS OF FOOD FIBERS CHARACTERISTICS IN MILK MIXTURES WITH THE MODIFIED FAT COMPOSITION

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Abstract
The article presents main determination methods of technological parameters of food fibers in milk mixtures of the modified fat composition.

The methods of studying the ability of food fibers to water and fat-absorption are offered. The values that characterize swelling parameters of vegetable ingredients in experimental mixtures are obtained.

The effectiveness of the method of IR-spectroscopy for indentifying and comparing water-binding forms in mixtures of food fibers with water and butterdish is proved.

The indices of thermal stability and degree of liquid fat outflow for mixtures with the modified fat composition with the maximal replacement of cream butter by 25 % of oil are obtained. The research results indicate objective possibilities for the effective use of food fibers Vicetal for stabilizing the structure and preventing consistence defects of products with the modified fat composition.

Keywords: milk mixtures with the modified fat composition, food fibers, thermal stability, fat-retaining, IR-spectroscopy method.

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1. Introduction
It is expedient to introduce milk products, based on mixtures with the modified fat composition with the maximally possible replacement of milk fat by oil that doesn't need an essential technical re-equipment of an enterprise and establishment of special lines.

There was developed the technology of child spread, which recipe includes sunflower and corn oils together with cream butter [1]. The content of the latter one is 10 % of the amount of the milk-fat base. There is the information that the oils content increase results in a defect – liquid fat outflow from the monolith [2].

According to SSU, spread is a fatty food product (emulsion of “water in fat type”), consisted of milk and vegetable fat. The mass share of total fat is from 50 % to 85 %, and the one of milk fat must be no less than 25 % of total fat. The product has a dense or soft consistence with (without) food supplements, fillers and vitamins.

Substitutes of milk fat are most often used for combining with it, and also sunflower, corn, coconut, palm-kernel, palm oils and many others.

The replacement of a part of milk fat by oils at making products with the modified fat composition (MFC) results in their consistence change. Differences in fats nature are connected with changes of the process of common crystallization at the thermomechanical influence, reflected on structure parameters [3]. Thus, such changes are less noticeable for spreads, produced based on
creams and vegetable fats. When production is realized by recombination - based on melt fats and milk plasma by the butter-producing scheme, the plasticity decrease in the product, liquid fat outflow from the monolith or excessive solidity are possible. And a result is the consistence worsening of the products [4].

It is urgent to use vegetable ingredients with water- and fat-absorbing capacity, swell in water and butterdish. The products of corn processing with food fibers (Vitacel, extruded wheat flour) and wild rose meal are compatible with the milk-fat base at the organoleptic level. Table 1 presents characteristics of different sources of food fibers (FF) for possible use in milk products of MFC [5, 6].

Table 1
Characteristics of different sources of food fibers

| Source                     | FF content, % | Advantages                                                                 | Used                                                                 | Shortcomings                                             |
|----------------------------|---------------|---------------------------------------------------------------------------|----------------------------------------------------------------------|----------------------------------------------------------|
| Meal of wild rose fruits   | 39.68         | – complex use of vegetable raw materials both FF and oils in one product | necessity of continuous microbiological control; presence of alien compounds; technological effect, difficulty prognosticated |
| Food fibers Vitacel WF400  | 98.0          | – absence of harmful admixtures;                                           – high cost                                                             |
|                            |               | – microbiological purenness;                                               |                                                                      |                                                          |
|                            |               | – standardization of parameters;                                           |                                                                      |                                                          |
|                            |               | – prognosticated technological effect;                                     |                                                                      |                                                          |
|                            |               | – possibility of combination                                               |                                                                      |                                                          |
|                            |               | – preservation of native properties of crop FF;                           |                                                                      |                                                          |
|                            | 1.2           | – low cost                                                                |                                                                      |                                                          |

The aim of the work is to determine methods for characterizing technological properties of food fibers in milk mixtures with the modified fat composition with the provided replacement of milk fat by oil up to 25 %. Such approach allows to prevent consistence defects of the product.

2 Materials and Methods

At the first stage of experimental studies, there were prepared model samples of MFC mixtures with the following composition: cream butter, butterdish, oil of Rosa canina L. fruits in the amount from 10 % to 25 %, food fibers Vitacel, extruded wheat flour (EWF) and wild rose meal.

Cream butter was prepared, according to SSU 4399:2005. The products had the following parameters: fat mass share 73 %, moisture – 25 %, protein 0.8 %, carbohydrates – 1.3 %, titrated acidity – 23 °T, pH 6,2.

The main fatty acid composition of Rosa canina L. oil is presented by the following fatty acids, % of the total amount of fatty acids: oleic (C \(_{18:1}\) – 17.8; linoleic (C \(_{18:2}\) – 49.3; linolenic (C \(_{18:3}\) – 26.8 and other.

The chemical composition of Rosa canina L. fruit meal is presented in Table 2 [7].

Food fibers Vitacel WF400 (produced by J.Rettenmaier Sohne GmbH, Germany), have the following technological properties: water-retaining capacity and fat absorption (for 1 g of product) – 11 g and 12 g, respectively. The water activity is fixed at level 0.44, and pH – (6.5±1.5). The bulk mass of Vitacel is 40±2.5 g/dm\(^3\). Average sizes of 90 % of fiber particles are <300 mcm. The summary amount of food fibers is (98.0±0.5) %, including cellulose – (72.0±2.0) %, hemicelluloses – (25.5±1.5) %, lignin – 0.5±0.1 %. The following parameters of FF are at the level: mass share of fats – (0.2±0.02) %, proteins – (0.4±0.06) %, moisture – no more 8 %, ash – no more 3 %. According to the producer, Vitacel has the following microbiological parameters: NMFAIM (CFU in 0.1 g) – 5×10\(^4\), mould (CFU in 0.1 g) – no more 50, pesticides and fungicides – <0.002 mg/kg.
Pathogenic microorganisms (salmonella), in 25 g of FF, aflatoxins, CBG (coliforms) in 0.1 g are not revealed. The energetic value of 100 g of “Vitacel” is 0.09 kcal [8].

| Components                  | Mass share, % |
|-----------------------------|---------------|
| Moisture                    | 12.0          |
| Protein/fats/ash            | 3.4/1.4/3.23  |
| Food fibers:                |               |
| Soluble/insoluble           | 36.63/3.05    |
| Carbohydrates:              |               |
| fructose/arabinose          | 18.5/5.5      |
| galactose/glucose           | 2.5/2.4       |
| rhamnose                    | 1.8           |
| Organic acids:              |               |
| apple/lemon                 | 0.65/3.45     |

Extruded wheat flour (EWF) is produced in Ukraine, according to TC U 00883403.002-99. The extrusion technology (from Lat. extrudo – push out, press out) is based on the process that combines thermal, hydro- and mechanic processing of wheat flour and allows to get a product with stable properties.

Table 3 presents physical-chemical parameters of EWF [9].

| Parameter                  | Value    |
|----------------------------|----------|
| pH                         | 6.4      |
| Mass share, in %:          |          |
| Protein/fat                | 11.9/2.3 |
| Carbohydrates/cellulose    | 68.2/1.2 |

At first, there was studied the water-retaining capacity (WRC) and fat-retaining capacity (FRC) of wild rose fruit meals, Vitacel and EWF. The process was conducted at temperature (20±2) °C in different mediums.

The method of determining FRC of food fibers provides their accurate mixing with oil or liquid fat, keeping of the mixture for swelling with further centrifuging for separating free fat.

The fat-retaining capacity was calculated by the formula, in %:

\[
FRC = \frac{(c - b)}{(b - a)} \times 100 \%,
\]

(1)

where \(a\) – mass of an empty centrifugal glass, g; \(b\) – mass of a glass with a sample after centrifuging and liquid pouring, g; \(c\) – mass of a glass with an experimental sample before centrifuging, g [10].

The capacity of FF swelling in butterdish and water was determined by the weighing method after immersing them in a solvent for a certain period of time [11].

This parameter is quantitatively characterized by the swelling degree \((K)\), which indicates the relative increase of the system mass and is calculated by the formula:

\[
K = \frac{(m_1 - m_0)}{m_0} = \frac{m_p}{m_0}, \text{ mg/mg},
\]

(2)
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where \( m_0, m_1 \) – mass of the system before and after swelling, respectively, mg; \( m_r \) – mass of the absorbed solvent, mg.

Butterdish had the following characteristics: 9.1±0.2 % of dry substances; 4.7±0.1 % lactose, 3.2±0.1 % protein, 0.3 % fat, 0.7±0.1 % mineral substances, density 1028 kg/m³, titrated acidity 18±0.5 °T.

For determining moisture binding forms in milk mixtures with food fibers there were obtained spectrums of IR-Fourier electrophotometer binding «Nexus» by «Nicolet» (USA). Conditions of spectrums photo were the following: number of scans – 7 in 1 s, scanning interval – 1 cm⁻¹, scanning diapason – 400…4000 cm⁻¹.

The image of FTIR-spectrophotometer «Nexus» is presented on Fig. 1.

Fig. 1. FTIR-spectrophotometer «Nexus»

The solidity in mixtures for food products with the modified fat composition was determined on the texture analyzer «LFRA BROOKFIELD» [12]. The conditions of the conducted analysis are presented in Table 4.

Table 4

| Name        | Conditions                                      |
|-------------|-------------------------------------------------|
| Regime      | Normal (force measuring at compression)         |
| Speed, m/s  | 2                                               |
| Distance, mm| 10                                              |
| Trigger, g  | 4                                               |
| Probe       | Brookfeld TA 15-45° Perspex conical             |

All samples were placed in containers of 200 ml. After finishing the process of crystallization, all samples were led to temperature 20 °C and analyzed in threefold repetition.

At the following stage, there was determined the outflow of liquid fat and thermal stability of mixtures, and oil and food fiber contents in them were optimized.

Thermal stability is an ability of milk mixtures with MFC to preserve their form at temperature 28…30 °C. Cylinders (diameter and height 20 mm) were cut from experimental samples by sample selectors and accurately placed on the glass plate with numbers of samples at distance 2…3 cm from each other. Then the plate with samples was placed in the thermostat at temperature 28…30 °C and kept during 2 hours. After that they were placed on the filtering paper and a diameter of each cylinder base was measured. For characterizing the thermal stability of milk mixtures with MFC, the thermal stability coefficient was determined by the formula:

\[ K_m = \frac{d_0}{d}, \]  

(3)
where $K_m$ – index of thermal stability of milk mixtures with MFC; $d_0$ – initial diameter of the cylinder base, mm; $d$ – diameter of the cylinder base after thermostating, mm.

The thermal stability index close to 1 characterizes a high thermal stability, and one, deviating from 1 – a worsened one [13].

The index of outflow degree characterizes the ability of the structure of milk mixtures with MFC to retain liquid fat. A cooled experimental sample as a cube with ribs length 3,5 cm was placed on 5 layers of the filtering paper, placed in the Petri dish. Then samples were kept in the thermostat at temperature 25 °C during 30 min, and residues of milk mixtures with MFC were accurately eliminated from the paper. The amount of liquid fat, flowed out (%) was determined by the formula:

$$M=(c-a)/(b-a) \times 100,$$

where $a$ and $b$ – mass of the Petri dish with the filtering paper with and without a mixture; $c$ – mass of the Petri dish with the filtering paper and cube of the experimental sample with the filtering paper, soaked by fat [10].

Functional dependencies of the outflow degree of milk fat and thermal stability coefficient in milk mixtures with MFC on the FF amount were determined by the method of least squares. Its essence is in determining regression equation coefficients that provide the minimum of the sum of deviation squares of experimental data from values, calculated by the regression equation that is the minimum of such function [14].

For determining the functional dependence that recreates the change of parameters most distinctly, the approximation reliability coefficient ($R^2$) of each function was found, and the optimal one was found. At analyzing linear dependencies, functions growth or fall was expressed by the derivative that is speed by its physical content. The experimental data were processed by the method of mathematical statistics [15]. The main statistical parameters were determined:

– mean arithmetic of the measured value ($\bar{x}$):

$$\bar{x} = \frac{\sum x_i}{n},$$

where $x_i$ – data of parallel determinations; $n$ – number of determinations.

– deviation from the mean value

$$\bar{x}_i - x;$$

– dispersion ($S^2$):

$$S^2 = \frac{\sum (x_i - \bar{x})^2}{n-1};$$

– mean square deviation $\overline{S}$:

$$\overline{S} = \sqrt{S^2};$$

– error of the mean arithmetic value $S_x$:

$$S_x = \frac{\overline{S}}{\sqrt{n}};$$

– reliable limits: upper, lower:
The value of the reliable interval $\bar{x} \pm \varepsilon_p$ was determined at the reliable probability $B=0.95$. Just this value is accepted in chemical and biological solutions [16]:

$$
\varepsilon_p = \frac{t_p \cdot S_x}{\sqrt{n}},
$$

where $\varepsilon_p$ – value of reliable limit; $t_p$ – Student criterion, depending on the number of experiments for the reliable probability $P=0.05$.

The parameters of mixtures with the maximal replacement of cream butter by oil (25 %) is influenced by the amount of FF of Vicetal (C1, %) and meal (C2, %). The upper and lower level of C1 factor is 0.6 (max) and 0.1 (min) respectively, and for C2 – 3.5 (max) and 1 (min). The maximal and minimal values of FF amounts are chosen, taking into account producer’s recommendations and compatibility with the milk-fat base at the organoleptic level.

3. Experimental procedures

The results of determining the fat-retaining capacity of *Rosa canina* L. fruits meals, EWF and Vitacel in melt cream butter and wild rose oil and water-retaining capacity of the aforesaid FF in water and butterdish are presented in Table 5.

**Table 5**

| Vegetable ingredients with FF | Fat-retaining capacity of FF, in %, in | Water-retaining capacity, in %, in |
|------------------------------|---------------------------------------|-----------------------------------|
|                              | oil         | Cream butter | butterdish | water       |
| Vitacel                      | 55.0±1.0    | 59.0±0.5     | 95±1.0     | 99.0±1.0    |
| Extruded wheat flour         | 42.3±1.3    | 40.4±1.2     | 80.3±1.4   | 84.2±1.3    |
| Meal of *Rosa canina* L. fruits | 19.0±0.7   | 21.0±0.5     | 49.0±0.4   | 56.0±0.5    |

The minimal value of FRC was fixed for *Rosa canina* L. meal, and the maximal one was determined for Vitacel. The high WRC was inherent to Vitacel in butterdish – (95±1.0) %. Probably additional coagulating connections form in the system; they cause formation of a secondary spatial net that correspondingly allows the structure to form liquid fat. The low capacity to water retention was fixed for *Rosa canina* L. meal, at level (49.0±0.4) %. The aforesaid indices for extruded wheat flour are within the determined diapasons. The fixed values are an evidence of the effectiveness of fat- and water retention of specially processed food fibers.

The enrichment of MFC mixture with such food fibers as *Rosa canina* L. fruit meal is expedient, and the technological effect (fat- and water retention) must be intensified by adding EWF or Vitacel.

The research results that characterize the dependence of *Rosa canina* L. fruit meal EWF and Vitacel swelling in butterdish at temperature (20±2) °C on duration of the process are presented in Fig. 2.

According to the research results (Fig. 2), the rational duration of vegetable ingredients swelling is from 15 to 20 min. The swelling coefficient in butterdish for Vitacel was fixed at level 7.7±0.2 un, and for EWFF – 6.8±0.1 un, for wild rose meal – 2.9±0.1 un. The most intensive swelling takes place in first 5 min.

As to the study of forms of moisture connections in mixtures with food fibers by IR-method, the following must be outlined. In the area of OH-valent fluctuations of spectrums of the sample, (EWF and butterdish), there were observed two strips 3402 cm$^{-1}$ and 3301 cm$^{-1}$ respectively (area of the organically bound water). These strips correlate with two types of H-bound water.
Fig. 2. Dependence of *Rosa canina* L. fruit meal EWF and Vitacel swelling in butterdish at temperature (20±2) °C on duration of the process

There were studied the parameters of thermal stability and liquid fat outflow degree in mixtures of cream butter with *Rosa canina* L. oil in amount 25%. For determining the optimal amount of Vitacel in the combination with wild rose meal in MFC mixtures, there was used the full factor experiment (FFE). The experimental data were processed by the method of mathematic statistics and the adequate regression equations were obtained.

The mathematical models of the liquid fat outflow degree ($Y_1$) and thermal stability ($Y_2$) of MFC mixtures with different FF are presented below:

$$Y_1 = \frac{16.63}{C_1^{0.1} \cdot C_2^{0.06}}, \quad Y_2 = \frac{C_1^{0.04} \cdot C_2^{0.02}}{3.35}.$$ 

The optimum of values is observed for samples with the maximal possible replacement of milk fat by oil in amount 25%, that contain Vitacel in amount 0.3% and wild rose meal in amount – 2.0%. At that the liquid fat outflow degree is 19.1%. Its maximal value (22.6%) is observed at the least mass share of FF.

The thermal stability of milk mixtures with MFC is directly proportional to the FF ration and reversely proportional to the amount of introduced *Rosa canina* L. oil. The optimum of the thermal stability coefficient is within 0.81...0.82, that corresponds to the amount of Vitacel food fibers in MFC milk mixtures – 0.3 % and wild rose meal – 2.0 %. The obtained values are included in the limits of permitted ones and correlate with previous studies.

4. Conclusions

The determination of water- and fat-retaining capacity of food fibers of different processing degrees is effective for prognosticating the use of Vicetal, EWF and *Rosa canina* L. fruit meal in the composition of mixtures with the modified fat composition.

The results of IR-spectral studies for determining bound moisture allowed to estimate connection forms of vegetable ingredients with moisture (butterdish) objectively. The research results testify to creation of water polyassociates with hydrophilic functional groups of the dispersion system – fibers of vegetable ingredients with the low FF content, but active carbohydrate component.

The rational ratios of food fibers were established at the level: Vitacel – 0.3 %, extruded wheat flour – 2.0 % wild rose meal – 2.0 % for milk mixtures with cream butter replaced by 25 % of *Rosa canina* L. oil.

The presented information is recommended for substantiating technologies of milk mixtures of the modified fat composition with food fibers for preventing consistence defects at combining cream butter with oil.
The limitation for practical realization of the research results is the compatibility of vegetable ingredients and milk base at the organoleptic level.

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