Chemical compositions of dairy products and raw milk from cows kept at different technologies in farms of Volgograd region

I F Gorlov¹², M I Slozhenkina¹², A E Serkova¹²*, N I Mosolova² and A N Sivko²

¹ Volgograd State Technical University, 400005, 28 Lenin av., Volgograd, Russia
² Volga region research Institute of production and processing of meat and dairy products, 400131, 6 Rokossovsky str., Volgograd, Russia

* E-mail: serkova.anastasiya@gmail.com

Abstract. The article presents chemical compositions of dairy products and milk from dairy cattle kept under different technological conditions, i.e. stable keeping at AE LLC Donskoye, Kalachevsky District, Volgograd Region and stall-camp keeping at JSC Kirov Farm, Staropoltavsky District, Volgograd Region. To determine chemical compositions, methods of inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) were applied. There was used an Elan 9000 quadrupole mass spectrometer (Perkin Elmer, USA) and an Optima 2000 DV atomic emission spectrometer (Perkin Elmer, USA). The article also provides a comparative description of macro- and microelement compositions of samples (milk, sour cream, cream, whey, cottage cheese, and butter) and concentrations of heavy metals. The obtained results allowed a conclusion that stall keeping is preferable for obtaining high-quality dairy raw material because ruminants’ microflora in forestomachs cannot quickly adapt to summer pasture rations after winter stall keeping.

1. Introduction

Dairy cattle breeding has been the most important sector of the national economy that provides the population with biologically complete food products [1]. The chemical composition of milk depends on numerous factors, including livestock conditions, i.e., lactation, breed, animal health, keeping technology, and others [2].

The effectiveness of the keeping technology is seen in the productivity of dairy cattle and chemical composition and technological properties of milk. It is extremely difficult to predict the quality of finished products without necessary vegetable feed, appropriate mineral additives, local dried medicinal herbs, and some additional factors, contributing to improved composition of cow's milk at different periods of keeping [3, 4]. Therefore, the study of mineral compositions of milk and its products from cows kept in farms at different conditions and feeding is relevant and appropriate.

Milk contains macro- and microelements in the form of cations and anions in the composition of proteins, enzymes, nucleic acids, etc. The salt composition of milk and its products can be estimated by the contents of cations and anions in regular and colloid solutions [4].

The purpose of the work was to study chemical compositions of milk and its products and conduct a comparative assessment of the macro- and microelement compositions of raw materials with respect to...
the keeping technology of dairy cattle—stall keeping at LLC “Donskoye” and stall camp at JSC Kirov Farm.

At LLC “Donskoye”, Kalachevsky District, Volgograd Region, experimental cows (Test Group I) were fed with balanced rations in accordance with detailed VIZH standards [5, 6]. The farm used a variety of high-quality feeds, i.e., haylage, silage, sudanese hay, alfalfa hay, mixed grass, and straw. Moreover, the animal rations contained soybean meal, crushed corn grain, and vitamin and mineral premix 3P60-3 (MegaMix). Feeding observed the calcium-phosphorus and sugar-protein ratios due to the appropriate supplements.

At JSC Kirov Farm, Staropoltavsky District, Volgograd Region, experimental animals (Test Group II) were fed according to stall-camp keeping. In addition to green top dressing and concentrated feed (a mixture of barley, wheat, and peas) in feeding troughs, experimental animals were grazed on near-farm natural and seeded pastures. In October, the cows continued to graze on the near-farm pastures and received supplementary feeding of green fodder in troughs in the walking yard. From October to April, the lactating cows were in stall keeping and received feed from feeders in the walking yard during the day.

2. Material and methods
Mineral compositions of milk obtained from cows in Test Group I and Test Group II and its products after processing were examined.

We used modern analytical and experimental methods and certified equipment in the accredited laboratory at LLC Micronutrients in Moscow.

Sampling was conducted according to GOST 26809-86 “Milk and dairy products.” To determine the chemical compositions of milk and products after its processing, the method of inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) were applied. We also used an Elan 9000 quadrupole mass spectrometer (Perkin Elmer, USA) and an Optima 2000 DV atomic emission spectrometer (Perkin Elmer, USA).

The statistical data were processed on the Statistical program (Statistica, version 6.0 (Dell, USA). The data were presented as average values. Differences between the samples were estimated using an unpaired t-test. Correlation analysis with the calculated pair correlation coefficient was used to establish the relationship between the parameters. The significance was determined by Student's test, the value was considered reliable at $p\leq0.95$. The study was repeated three times.

3. Results and discussion
One of the most important points in determining the quality of raw milk was the analysis of its elemental composition. The data on the quantitative content of macro- and microelements, including heavy metals, are important because they ensure milk’s safety and its potential contribution to the physiological needs of the human body. After a series of analyzes, we obtained data on the concentration of chemical elements in raw milk from cows in Test Groups I and II. The results are presented in tables 1 and 2. A diagram was constructed for a visual comparison of the macroelement compositions of raw milk in both Test Groups (figure 1).

**Table 1. Concentration of chemical elements in cow's milk.**

| Chemical element | Test Group I | Test Group II |
|------------------|--------------|---------------|
| Al               | <0.09        | <0.09         |
| B                | 0.24±0.029   | 0.24±0.029    |
| Ca               | 1284±128     | 1204±120      |
| Co               | 0.004±0.0008 | 0.003±0.0006  |
| Cr               | 0.03±0.004   | 0.04±0.006    |
| Fe               | 0.26±0.078   | 0.38±0.115    |
| I                | 0.18±0.022   | 0.15±0.018    |
Table 1. Compared macroelement compositions of milk in Test Groups I and II.

| Element | Test Group I | Test Group II |
|---------|--------------|--------------|
| K       | 2140±257     | 2090±251     |
| Li      | 0.002±0.0005 | 0.004±0.0008 |
| Mg      | 101±10       | 96.1±9.61    |
| Mn      | 0.03±0.005   | 0.02±0.003   |
| Na      | 337±34       | 343±34       |
| Ni      | 0.06±0.009   | 0.05±0.008   |
| P       | 1005±121     | 946±142      |
| Se      | 0.04±0.006   | 0.04±0.005   |
| Si      | 0.83±0.248   | 0.92±0.277   |
| Sn      | 0.0009±0.00027 | 0.0009±0.00028 |
| Sr      | 0.65±0.078   | 0.54±0.065   |
| V       | 0.001±0.0003 | 0.004±0.0009 |

**Figure 1.** Compared macroelement compositions of milk in Test Groups I and II.

The Na:K ratio in milk of lactating cows in Test Groups ranged from 0.157:1 (Test Group I) to 0.164:1 (Test Group II). In other words, the Na:K ratio in milk of cows in both groups did not correspond to generally accepted indicators of mineral nutrition and quality and was less than 0.3-0.5:1.

The potassium content in both samples was higher than the regional average (113-117 mg%). Since potassium and sodium salts ensure the salt balance of milk, that is, a certain ratio between calcium (magnesium) cations and anions of phosphates and citrates, an increased content of one of these macronutrients can affect the regulation of ionized calcium in milk that in turn affects the size and stability of casein micelles. The salt imbalance of milk can cause undesirable reactions at various stages of milk processing (for example, coagulation of proteins during milk sterilization).

The correct calcium-phosphorus balance in diets, when lactating cows were changing stall to pasture keeping, was evaluated by the Ca:P ratio in milk that ranged from 1.27:1 to 1.28:1 in the samples studied. Both proportions in each milk sample were well-balanced, which indicated their high digestibility. The optimal calcium-phosphorus ratio in milk is 1:1-1.4:1.

The calcium and phosphorus contents in milk in Test Groups was in the range of average values for these elements, however, Test Group I milk surpassed Test Group II milk in terms of both indicators. This can be explained by the full-fledged ration of dairy cattle at stall-pasture keeping.

In terms of the iodine and magnesium contents, milk in Test Group I slightly exceeded milk Test Group II. Only traces of aluminum and arsenic were found in both samples.

Table 2 shows that the copper content was below the MAC 12.5 times in Test Group I and 14.28 times in Test Group II; and zinc was lower than the MAC 1.14 and 1.2 times, respectively. The lead content in milk was 100 times lower than the MPC in both groups. There were registered only traces of...
cadmium, arsenic, and mercury in the samples. In other words, such milk could not adversely affect human health and was suitable for consumption and processing.

Table 2. Concentrations of heavy metals in milk.

| Chemical element | MAC | Contents of heavy metals (μg/g) |
|------------------|-----|---------------------------------|
|                  |     | Test Group I | Test Group II | Test Group I | Test Group II |
| Zn               | 5.0 | 4.37±0.44     | 4.17±0.42     | <0.0042      | <0.0042      |
| As               | 0.05| <0.00012      | 0.0002±0.00005| <0.00054     | <0.00054     |
| Cd               | 0.03| 0.08±0.012    | 0.07±0.01     | <0.00005     | <0.00005     |
| Hg               | 0.005| <0.00054     | 0.001±0.00029 | 0.001±0.00033|
| Pb               | 0.1 | 0.001±0.00029| 0.001±0.00033|              |              |

Most vital mineral milk substances, transiting to its products after processing, also evidenced high quality of technological keeping lactating cows. The concentration of chemical elements in cottage cheese and curd whey is presented in tables 3 and 4.

Table 3. Concentrations of chemical elements in cottage cheese and whey.

| Chemical element | Measurement result (μg/g) |
|------------------|--------------------------|
|                  | cottage cheese | whey          |
|                  | Test Group I | Test Group II | Test Group I | Test Group II |
| Al               | 0.72±0.087 | 1.26±0.13     | 0.03±0.007 | 0.05±0.012     |
| B                | 0.12±0.015 | 0.18±0.021    | 0.18±0.036 | 0.18±0.036     |
| Ca               | 1125±113   | 1183±118      | 121±18    | 118±18         |
| Co               | 0.003±0.0007 | 0.003±0.0007 | 0.0005±0.00022 | 0.0006±0.00025|
| Cr               | 0.01±0.002 | 0.02±0.002    | 0.001±0.00038 | 0.002±0.0006   |
| Fe               | 1.59±0.4  | 2.07±0.52     | 1.47±0.51 | 0.0002±0.00009 |
| I                | 0.08±0.011 | 0.06±0.009    | 0.05±0.014 | 0.05±0.014     |
| K                | 890±133    | 1093±131      | 206±51    | 192±48         |
| Li               | 0.003±0.0006 | 0.006±0.0012 | 0.02±0.004 | 0.01±0.003     |
| Mg               | 89.04±8.9 | 98.86±9.89    | 29.07±4.36 | 26.89±4.03     |
| Mn               | 0.06±0.01  | 0.1±0.015     | 0.008±0.0025 | 0.0002±0.00009|
| Na               | 202±20     | 264±26        | 10±1      | 11±1           |
| Ni               | 0.11±0.013 | 0.1±0.012     | 0.01±0.0029 | 0.0002±0.00009|
| P                | 2840±341   | 3118±374      | 242±60    | 223±56         |
| Se               | 0.12±0.015 | 0.13±0.016    | 0.06±0.016 | 0.08±0.02      |
| Si               | 6.31±1.58  | 8.16±2.04     | 3±0.3     | 0.04±0.03      |
| Sn               | 0.03±0.004 | 0.02±0.003    | <0.00015  | 0.0002±0.00006 |
| Sr               | 0.65±0.078 | 0.69±0.083    | 0.17±0.034 | 0.17±0.033     |
| V                | 0.001±0.0002 | 0.002±0.0004 | 0.0002±0.00007 | 0.0002±0.00009|

The data obtained enabled constructing diagrams (figure 2), containing a comparative assessment of the contents of the main macroelements in cottage cheese from raw milk in Test Groups I and II.

The study found that the content of basic macronutrients in Test Group II cottage cheese was higher than in Test Group I cottage cheese. Calcium is the most important mineral component in the acid-rennet production of cottage cheese. The first stage of rennet coagulation (k-casein hydrolysis by chymosin or another coagulator) was followed by formation of rennin curd that depended on a sufficient amount of soluble calcium and corresponding colloidal calcium content that allowed the micelles to remain intact. Calcium ions affect the coagulability by neutralizing negative charges on casein micelles and, probably, forming bonds (calcium phosphate bridges) between negatively charged phosphate groups on casein micelles. The
activity of calcium ions in milk affects the duration of rennet coagulation and clot density. The calcium distribution is strongly influenced by pH; pH decrease makes colloidal calcium dissolve to complete dissolution at a pH of 4.6. This is of particular importance for the cottage cheese structure, since the amount of calcium in the curd clot depends on the pH value in whey excreting. Other things being equal, cottage cheese with high calcium content has a more elastic texture than cottage cheese with low calcium content [8].

![Graph showing macroelement compositions of cottage cheese compared in Test Groups I and II.](Image)

**Figure 2.** Macroelement compositions of cottage cheese compared in Test Groups I and II.

The optimal ratio between calcium and phosphorus in the cottage cheese is 1:1.5-1:2. However, in the cottage cheese made from milk of the Test Groups, this indicator made 1:0.38-0.39, which was significantly lower than the optimum one.

**Table 4.** Concentrations of heavy metals in cottage cheese.

| Chemical element | MAC for cottage cheese | Contents of heavy metals (μg/g) | Test Group I  | Test Group II |
|------------------|------------------------|---------------------------------|---------------|---------------|
| Zn               | 50.0                   | 20.18±2.02                      | 14.26±1.43    |
| As               | 0.2                    | 0.01±0.0002                     | 0.002±0.0003  |
| Cd               | 0.2                    | 0.0008±0.00025                  | 0.001±0.0003  |
| Cu               | 4.0                    | 0.36±0.043                      | 0.25±0.03     |
| Hg               | 0.02                   | <0.00054                        | <0.00054      |
| Pb               | 0.3                    | 0.01±0.002                      | 0.01±0.002    |

The data obtained indicated heavy metals, not exceeding the MAC for the curd. According to MAC, milk in Test Groups I and II had lower values of zinc content 2.5 and 3.5 times, arsenic 20 and 100 times, cadmium 250 and 200 times, copper 11.1 and 16 times, and lead 30 times, respectively. Only traces of mercury were found in both samples.

The study of cottage cheese produced from raw milk in Test Groups I and II found that both samples were safe, however, in terms of the main macronutrients, cottage cheese in Test Group I was inferior to cottage cheese in Test Group II.

After butter was produced from raw milk in both Test Groups, the content of mineral substances was determined. The research results are presented in Tables 5 and 6. A comparative diagram of the main macronutrients is shown in figure 3.
Table 5. Concentration of chemical elements in butter.

| Chemical element | Test Group I | Test Group II |
|------------------|--------------|---------------|
| Al               | 0.87±0.105   | 0.98±0.119    |
| B                | 0.07±0.011   | 0.07±0.011    |
| Ca               | 340±34       | 279±28        |
| Co               | 0.002±0.00029| 0.004±0.0008  |
| Cr               | 0.02±0.003   | 0.02±0.003    |
| I                | 0.02±0.004   | 0.03±0.005    |
| K                | 477±72       | 433±65        |
| Li               | 0.003±0.0006 | 0.004±0.0008  |
| Mg               | 32.69±3.27   | 28.64±2.86    |
| Mn               | 0.05±0.008   | 0.07±0.01     |
| Na               | 122±12       | 151±15        |
| Ni               | 0.07±0.01    | 0.16±0.019    |
| P                | 336±50       | 308±46        |
| Se               | 0.005±0.001  | 0.008±0.0015  |
| Si               | 13.06±2.61   | 10.77±2.15    |
| Sn               | 0.03±0.005   | 0.01±0.002    |
| Sr               | 0.24±0.029   | 0.18±0.022    |
| V                | 0.002±0.0003 | 0.001±0.0003  |

Figure 3. Macroelement compositions of butter compared in Test Groups I and II.

Based on the data obtained, it can be concluded that butter, like its base raw milk from cows in Test Group I, was better in terms of basic macroelements.

Table 6. Concentrations of heavy metals in butter.

| Chemical element | MAC | Contents of heavy metals (μg /g) |
|------------------|-----|---------------------------------|
| Zn               | 5.0 | 2.1±0.21                         |
| As               | 0.1 | 0.001±0.0003                     |
| Cd               | 0.03| 0.001±0.00029                    |
| Cu               | 0.4 | 0.08±0.011                       |
| Hg               | 0.03| <0.00054                         |
| Pb               | 0.1 | 0.01±0.002                       |
| Fe               | 1.5 | 2.59±0.65                        |
According to SanPiN 2.3.21078-2001 [9], butter should additionally meet iron content regulations. Based on the results obtained, the maximum permissible concentration of this element was found to exceed MAC 1.7 times in milk of Test Group I and 8 times in milk of Test Group II.

At the iron content of not more than 0.5-0.9 μg/g, noticeable deviations in the sensory properties of butter are not detected. Iron in an amount of 2 μg/g and more causes some defects—metallic and greasy tastes manifested during long-term storage.

Transition metals exhibit different activity in oxidative processes. Considering the degree of activity decrease, metal oxidation catalysts are usually arranged in the following order: Cu, Fe, Co, Ni, and Mn. Iron in ionic form is more active than copper. In butter, iron immediately changes into a form of protein binding. The activity of this complex is many times less compared with copper protein compounds. However, a high content of both metals causes a decrease in the stability of butter during storage [10].

Thus, butter produced from raw milk from both Test Groups can harm the body if consumed excessively. Test Group II sample should be noted to have iron content that was significantly higher than the MAC, and macronutrients content that, on the contrary, was lower than the reference.

To study the concentration of chemical elements in cream and sour cream, raw milk in both Test Groups was separated. Then, the cream was examined in terms of the content of mineral substances and fermented with a mixture of strains of live mesophilic lactobacilli *Lactococcus lactis*, *Lactococcus cremoris*, *Lactococcus diacetylactis*, and *Leuconostoc cremoris* to obtain sour cream. The produced sour cream was also examined. The results are presented in table 7 and figures 4, 5.

| Chemical element | Measurement result (μg/g) |
|------------------|----------------------------|
|                  | Test Group I | Test Group II | Test Group I | Test Group II |
| Al               | 2.15±0.21    | 1.9±0.19      | 2.32±0.23    | 0.73±0.088    |
| B                | 0.12±0.015   | 0.13±0.016    | 0.19±0.022   | 0.1±0.012     |
| Ca               | 795±79       | 703±70        | 710±71       | 548±55        |
| Co               | 0.003±0.0007 | 0.004±0.0007  | 0.004±0.0008 | 0.002±0.0003  |
| Fe               | 4.65±1.16    | 4.46±1.11     | 22.52±4.5    | 2.86±0.71     |
| Fe               | 0.03±0.004   | 0.02±0.004    | 0.1±0.012    | 0.02±0.003    |
| K                | 1146±138     | 1370±164      | 1081±130     | 920±138       |
| Li               | 0.007±0.0015 | 0.008±0.0016  | 0.009±0.0017 | 0.004±0.0009  |
| Mg               | 77.48±7.75   | 78.41±7.84    | 68.17±6.82   | 53.4±5.34     |
| Mn               | 0.09±0.013   | 0.1±0.014     | 0.14±0.017   | 0.07±0.011    |
| Na               | 355±36       | 326±33        | 321±32       | 234±23        |
| Ni               | 0.21±0.025   | 0.15±0.019    | 0.17±0.02    | 0.06±0.009    |
| P                | 672±101      | 559±84        | 567±85       | 513±77        |
| Se               | 0.01±0.002   | 0.008±0.0015  | 0.008±0.0015 | 0.009±0.0017  |
| Si               | 7.68±1.92    | 10.03±2.01    | 11.04±2.21   | 8.85±2.21     |
| Sn               | 0.01±0.002   | 0.008±0.0016  | 0.01±0.002   | 0.008±0.0015  |
| Sr               | 0.47±0.056   | 0.41±0.049    | 0.45±0.054   | 0.36±0.043    |
| V                | 0.004±0.0009 | 0.004±0.0008  | 0.006±0.0011 | 0.001±0.0003  |
Figure 4. Transition of main macronutrients from cream to sour cream (Test Group I).

Figure 5. Transition of main macronutrients from cream to sour cream (Test Group II).

Diagrams 4 and 5 show that processing of milk in sour cream production slightly reduced the content of mineral substances may be due to the skim milk starter culture added in the amount of 3-5%. In terms of the main macroelements, the cream from Test Group I milk surpassed its comparison samples.

The concentrations of heavy metals in cream and sour cream samples are presented in table 8. The transition of zinc and copper into processed milk products is shown in figures 6 and 7.

Table 8. Concentrations of heavy metals in cream and sour cream.

| Chemical element | MAC | Contents of heavy metals (μg/g) | cream | sour cream |
|------------------|-----|---------------------------------|-------|-----------|
|                  |     | Test Group I | Test Group II | Test Group I | Test Group II |
| Zn               | 5.0 | 2.53±0.25 | 1.63±0.16 | 2.29±0.23 | 1.79±0.18 |
| As               | 0.05 | 0.002±0.0004 | 0.003±0.0005 | 0.003±0.0007 | 0.001±0.0003 |
| Cd               | 0.01 | 0.0009±0.00027 | 0.001±0.0003 | 0.002±0.0004 | 0.001±0.0002 |
| Cu               | 1.0 | 0.09±0.013 | 0.15±0.018 | 0.13±0.015 | 0.08±0.012 |
| Hg               | 0.005 | <0.00054 | <0.00054 | <0.00054 | <0.00054 |
| Pb               | 0.05 | 0.02±0.002 | 0.003±0.004 | 0.02±0.003 | 0.009±0.0018 |
Figure 6. Transition of zinc and copper into processed milk products in Group I.

Table 8 shows that all heavy metals concentrations did not exceed the MAC, therefore, cream and sour cream made from raw milk of both Test Groups were safe.

Figure 7. Transition of zinc and copper into processed milk products in Group II.

Figures 6 and 7 show that zinc largely transited from raw milk to cottage cheese due to the concentration of proteins during processing, and zinc to a greater extent migrated into curd whey.

4. Conclusions
Having systematized the data obtained, it was possible to infer the safety of raw milk in both Test Groups. The macroelement analysis showed that raw milk in Test Group I at the breeding factory Donskoye, Kalachevsky District, Volgograd Region, surpassed milk in Test Group II. The contents of the most important elements, such as calcium, potassium, magnesium, and phosphorus, in milk of Test Group I were higher, so we can conclude that the stall keeping of cows with optimized diets is preferable.

The research study established that the mineral content of the processed milk products in both Test Groups was safe and complete. Therefore, we can infer good quality of these products and proper technological keeping of lactating cows. The data obtained and their analysis indicated that milk products in Test Group I (cattle at the breeding plant “Donskoye”, Kalachevsky district of the Volgograd
region) had higher concentrations of the main macro- and microelements than milk products in Test Group II (cattle of Kirov Farm, Staropoltavsky district, Volgograd region). This might be due to the fact that the composition of microflora in forestomachs of the ruminants was gradually changing during transition from winter to summer rations on pastures; so the productivity and quality of milk temporarily decreased. The consequence of this is that the stall keeping is preferable for high-quality dairy raw material. However, proper feeding, optimal balance of nutrients, and careful observing dietary norms, the difference in the quantitative content of chemical elements in milk processed products under the considered conditions of keeping will be minimal, and the concentrations of these substances will be included in the averaged ranges of values.

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