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Determination of some metal contents of cow's milk in Akçabaat district of Trabzon by ICP-MS

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Abstract

In this study, amounts of chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), cadmium (Cd), lead (Pb), aluminum (Al), iron (Fe), and zinc (Zn), are indicated in cow's milk provided from sixteen different villages (Aykut, Helvacı, Erikli, Düzköy, Akçakale, Demirkapı, Acısu, Yaylacık, Çiçeklidüz, Esentepe, Arpacılı, Işıklar, Adacık, Osmanbaba, Yeşiltepe, Mersin) in Akçaabat district of Trabzon. The study was done in two different terms of the year. The first term is the winter season (November, December and, January named as Term I), during which animals are fed with dry fodder in the barn. The second term is the summer season (June, July, and August named as Term II), during which animals are fed with fresh feed. A total of 256 samples are analyzed. Levels of metals in the samples are determined with the ICP-MS method. It is found that the amounts of lead and aluminum in cow's milk in winter and summer season are close to each other while the amounts of chromium, cobalt, zinc, nickel, copper, iron, and cadmium are a bit more in the winter season. The amounts of metal in the analyzed kinds of milk are found to be between or under the specified rates given in literature except for nickel in milk from all villages and lead in one village. The amounts of mineral matter in milk in all villages are found under the rates given in the literature.

Keywords: Cow's milk, ICP-MS, metal concentration, Akçabaat district

1. Introduction

Many animal and vegetable-based foods are consumed to provide an adequate and balanced diet. As one of these sources, milk is the only food that starts from birth and contains protein, fat, carbohydrates, minerals, and vitamins in a balanced and sufficient quantity, which is necessary for the body at every stage of human life [1,2]. Milk has an important characteristic as being the main food of the living thing from the very first time in life for humans and animals due to the various nutritional values present in its structure. As long as it is not been subjected to external influences in any way due to its environmental conditions, it has kept its integrity as a completely harmless food [3].

The most important reason that milk is a very valuable source of human and animal nutrition is that it is rich in mineral matter. Especially, it contains quantitatively sufficient the most important reason that milk is a very valuable source of human and animal nutrition is that it is rich in mineral matter. Especially, it contains quantitatively sufficient amounts of matters such as protein and mineral substances that play a preventive role in growth and development. Milk contains enough chlorine, sodium, phosphorus, potassium, magnesium, and calcium, which are the minerals required to be taken from outside for living things. It is also known that the milk contains mineral substances which have toxic effects such as lead, lithium, mercury, cesium, aluminum, and cadmium, which are not required to be taken from outside. However, the concentration of these undesirable minerals in the milk must be very low [4].

It should be noted that there are differences according to the regions where milk is It should be noted that there are differences according to the regions where milk is obtained at the level of minerals naturally present and unwanted in the milk. The main reasons for these differences are animal nutrition, the environmental conditions, and the production process, which have a possible effect of a change on milk and dairies [5].

Heavy metal pollution in milk and dairies can be caused by raw materials from milking due to the

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Figure 1. The satellite image of Akçaabat district of Trabzon

contamination of milking animals or by machinery and equipment in contact with dairies during production and storage. The main elements in metallic impurities in metal containers used for preserving milk during technological processes are metals such as copper, zinc, iron, tin, lead, arsenic, and cadmium [6].

The amount of mineral matter in milk shows regional differences caused by different plants eaten at different places on the soil. Animals feed on soils containing toxic metals. The enrichment of heavy metals in the soil at toxic levels disrupts plant growth and quality. Toxic metals also reach people and animals fed with plants through the food chain [7]. Soil fertility and plant quality are adversely affected by agricultural irrigation, especially with polluted water sources. In addition, soil pollution stemmed from fertilization, industrial wastes, septic tank evacuation, and disinfestation can be caused by the volcanic structure of the environment [8,9]. The concentrations of heavy metal elements in the air, water, and soil environments have been examined by many researchers. Different results have been obtained regionally [10]. Soils are the last storage place for heavy metals. The heavy metals in the soil solution are taken by the soil microorganisms and the roots of the plant or washed into the groundwater. This leads to the deterioration of groundwater quality and to the pollution of the food chain [9,10]. In the literature, it is seen that there are many new studies on this subject [11,12].

In this study, in order to determine the heavy metal content, milk samples were collected from Akçaabat district in Trabzon, Turkey, due to the high production of dairy products, and analyzed with AAS. Thus, it was aimed to examine the factors affecting the change of heavy metal content in milk.

2. Materials and Methods

2.1. Collection of milk samples

Cow's milk samples were collected from 16 different locations (Aykut, Helvacı, Erikli, Düzköy, Akçakale, Demirkapı, Acısu, Yaylacık, Çiçeklidüz, Esentepe, Arpacılı, Işıklar, Adacık, Osmanbaba, Yeşiltepe, and Mersin) in Akçaabat district of Trabzon. The satellite image of the Akçaabat district of Trabzon is given in Fig. 1. The location information of the sample collected places is given in Table 1.

The milk samples were collected from these settlements in two different periods during the winter (1st period= Term I) and summer (2nd period= Term II) periods considering the nutritional status of animals. A total of 128 milk samples were collected in each period. So, a total of 256 samples were analyzed with parallel samples. The first period is the winter period in which the animal is fed with dry food and kept in the barn. The collection of milk and products in this period was completed in November, December, and January. The second period is the period when the animal is fed with green grass and removed to the pasture. This period covers the months of June, July, and August. Samples taken in special storage containers were kept in appropriate temperature conditions and moved to the laboratory environment. The milk samples taken from the mentioned settlements homogeneously into 100 mL PolyTri Flora Ethylene (PTFE) were stored in the freezer at -18 °C until analysis.

Table 1. Location information of the sample taken places in Akçaabat.

Number	Akçaabat District	Location	
		North	East
1	Aykut	40°54′14″	39°27′33″
2	Helvacı	40°59′30″	39°33′30″
3	Erikli	40°54′58″	39°29′47″
4	Düzköy	40°52′27″	39°25′30″
5	Akçakale	41°04′45″	39°30′10″
6	Demirkapı	40°54′49″	39°27′50″
7	Acısu	40°56′23″	39°27′24″
8	Yaylacık	41°00′55″	39°34′46″
9	Çiçeklidüz	40°58′43″	39°33′19″
10	Esentepe	40°55′59″	39°34′41″
11	Arpacılı	40°57′27″	39°28′53″
12	Işıklar	40°53′45″	39°27′52″
13	Adacık	41°02′34″	39°28′38″
14	Osmanbaba	41°00′12″	39°35′31″
15	Yeşiltepe	40°58′17″	39°30′22″
16	Mersin	41°05′16″	39°28′28″

2.2. Preparation method of Milk Samples for Analysis

Frozen milk samples from the -18 °C deep freezer were taken and thoroughly mixed at room temperature. During the preparation phase of the samples in the microwave oven, 2.00 mL was taken from the milk samples and placed in the Teflon sample containers of the device. Milestone Ethos EZ model microwave sample preparation system was used for this study [13]. Upon the samples, 4.00 mL of 65% HNO_3 and 2.00 mL of 30% hydrogen peroxide (H_2O_2) were added. The apparatus was operated at 180 °C and 270 bars. At the end of the digestion process, Teflon containers were kept in the oven for 5-10 minutes and then left to cool for 15-20 minutes in the drawer. Cooled containers were carefully opened. The cover and Teflon cup inner wall were washed with ultra-pure water, and the limpid solutions were quantitatively completed with ultra-pure water to 25 ml volume. With this method, the samples became ready for heavy metal analysis. In addition, 2.00 mL of ultra-pure water, 4.00 mL of nitric acid, and 2.00 mL of hydrogen peroxide were prepared without any samples as a blank solution. The Samples to be analyzed were placed in the sample storage cabinet at room temperature until measurement. Various internal standard solutions of 50 mL containing different elements were prepared.

All these procedures were carried out at Chemistry Research Laboratory in the Faculty of Science at Karadeniz Technical University. The measurement of heavy metal levels was performed by a multi-element reference method in the Agilent 7700e model ICP-MS (Inductive Coupled Plasma Mass Spectroscopy) device at Gümüşhane University Central Research Laboratory. Operation conditions and some analytical characteristics of the method were summarized in Table 2.

Standard deviation values are calculated from the obtained data. The results are given in Tables 3 and 4. Estimated daily intake (EDI) [14] of heavy metals and trace elements, Target hazard quotient (THQ) [15], Carcinogenic risk (CR) values can be calculated from the data given in Table 3 and Table 4 using Equations 1-3 [16].

$$EDI = \frac{Milk\ intake\ (kg/day).Heavy\ metal\ content\ in\ milk\ (\mu g/kg)}{Average\ individual\ weight\ (kg)} \quad (1)$$

$$THQ = \frac{MC.IR.EF.ED.CF}{RfD.BW.ATn}\, 10^{-3} \quad (2)$$

Here, MC is the heavy metal concentration in milk (mg/kg d.w.), IR is the ingestion rate (g/kg d.w.), EF is the exposure frequency (365 days/year), ED is the exposure duration for non-cancer risk as used by USEPA, CF is the conversion factor, RfD is the reference dose of individual metal, BW is an average adult body weight and ATn is the average exposure time for non-carcinogens [14].

$$CR = \frac{Exposure\ dose\ .Risk\ factor\ .Years\ of\ exposure}{70\ years\ (lifetime)} \quad (3)$$

3. Result and Discussion

The results obtained from ICP-MS analyses of milk samples collected in two different periods in the Akçaabat during the winter (Term I) and the summer (Term II) period are given in Table 2 and Table 3, respectively. Graphical representation of the data in these tables is given in Fig. 2.

According to these results, the highest chromium concentration value is 3.02 ± 0.70 µg/L (I. Term) in Çiçeklidüz, and the lowest value is 0.30 ± 0.32 µg/L (I. Term) in Helvacı. The highest value of cobalt was 3.27 ± 1.12 µg/L (Term II) in Yeşiltepe, while Yaylacık (Term I and Term II), Aykut (Term II), Helvacı (Term II), Erikli (Term II), Düzköy (Term II), Akcakale (Term II), Acısu (Term II), Çiçeklidüz (Term II) and Esentepe (Term II) were detected below the boundary of the places. For the nickel, the highest value was obtained in 206.52 ± 9.05 µg/L (Term I) in Mersin, while the lowest value was 104.85 ± 8.64 µg/L (Term II) in Esentepe. The highest value was 108.35 ± 16.15 µg/L (Term II) in Erikli, while the lowest value was 14.86 ± 1.83 µg/L (Term II) in Aykut for copper.

Table 2. Operation conditions and some analytical characteristics of Agilent 7700e model ICP-MS device

Nebulizer	: Low flow quartz concentric 0.2 mL/min
Spray chamber	: Quartz, low-volume, Scott-type double-pass water-cooled
Peristaltic Pump	: Low-pulsation, high-precision 10-roller peristaltic pump with 3 channels
Cell geometry	: Octopole
Sampling cone	: 1 mm diameter orifice, Ni-tipped
Skimmer cone	: 0.4 mm diameter orifice, Ni
RF generator power	: 27 MHz, 1400-1500 W, in steps of 10 W
Reflected power	: <10 W
Plasma gas flow	: 15 L/min
Nebulizer gas flow	: 0.95-1.00 L/min
Auxiliary gas flow	: 0.99 L/min
Expansion stage	: 2.0 mbar
Intermediate stage	: 2.0×10^{-4}-3.0×10^{-4} mbar
Analyzer stage	: 1.0×10^{-6}-2.0×10^{-6} mbar
Octopole bias	: −8 V
Quadrupole bias	: −3 V
Isotope	: ^{27}Al, ^{52}Cr, ^{56}Fe, ^{59}Co, ^{60}Ni, ^{63}Cu, ^{66}Zn, ^{111}Cd, ^{208}Pb
Internal standard	: ^{45}Sc, ^{89}Y, ^{185}Re, ^{209}Bi

	Al	Cr	Fe	Co	Ni	Cu	Zn	Cd	Pb
LOD, µg/L	1.2	0.1	0.5	0.1	0.5	0.8	0.6	0.3	0.5
LOQ, µg/L	4.0	0.4	1.7	0.4	1.7	2.7	2.0	1.1	1.7
RSD, %	2.6	3.3	0.9	1.0	0.9	1.3	1.9	2.1	1.3

LOD: Limit of detection, LOQ: Limit of quantification, RSD: Relative standard deviation

Table 3. Variation of metal concentrations in the Term - I (*N*: 3, *C*: µg/L)

Number	Location	Cr	Co	Ni	Cu	Cd	Pb	Al	Fe	Zn
1	Aykut	3.03 ± 0.42	1.26 ± 0.05	183.0 ± 11.0	93.0 ± 8.5	1.76 ± 0.09	26.0 ± 3.8	260.2 ± 11.7	155.0 ± 11.8	360.5 ± 12.6
2	Helvacı	0.30 ± 0.32	1.35 ± 0.05	174.9 ± 9.6	69.1 ± 7.4	1.76 ± 0.13	27.5 ± 2.8	250.9 ± 17.0	171.4 ± 14.2	361.3 ± 14.7
3	Erikli	1.50 ± 0.76	1.58 ± 0.07	166.5 ± 15.5	85.9 ± 10.2	1.82 ± 0.08	26.1 ± 5.1	251.2 ± 14.2	146.9 ± 14.4	386.4 ± 18.3
4	Düzköy	1.73 ± 0.32	1.04 ± 0.01	189.9 ± 5.4	24.7 ± 1.8	1.62 ± 0.42	21.9 ± 3.1	259.4 ± 9.5	170.52 ± 16.3	355.8 ± 17.2
5	Akçakale	2.17 ± 0.38	1.19 ± 0.13	182.3 ± 10.2	65.5 ± 5.4	1.88 ± 0.02	11.6 ± 1.6	262.7 ± 16.2	169.7 ± 13.7	368.4 ± 17.1
6	Demirkapı	2.00 ± 0.74	1.30 ± 0.12	180.9 ± 6.0	93.8 ± 2.9	1.75 ± 0.13	17.9 ± 3.9	248.4 ± 12.0	153.5 ± 14.6	363.5 ± 12.5
7	Acısu	1.29 ± 0.42	1.25 ± 0.14	160.3 ± 13.5	82.4 ± 6.4	1.70 ± 0.17	17.8 ± 2.6	261.7 ± 13.9	160.0 ± 10.0	347.5 ± 13.9
8	Yaylacık	1.84 ± 1.42	ND	178.6 ± 15.6	34.8 ± 2.8	1.69 ± 0.21	27.2 ± 5.5	276.4 ± 4.9	132.8 ± 9.0	362.8 ± 11.8
9	Çiçeklidüz	3.02 ± 0.70	1.66 ± 2.14	181.3 ± 9.3	53.7 ± 1.6	1.79 ± 0.19	30.5 ± 2.4	256.6 ± 13.8	156.4 ± 9.5	356.1 ± 11.1
10	Esentepe	1.91 ± 0.56	2.99 ± 0.58	185.0 ± 8.6	62.5 ± 4.0	1.82 ± 0.10	13.3 ± 1.3	261.7 ± 9.1	154.7 ± 10.4	366.7 ± 22.0
11	Arpacılı	1.40 ± 0.53	0.62 ± 0.11	166.3 ± 16.7	66.3 ± 3.9	1.90 ± 0.06	25.4 ± 4.9	271.2 ± 12.2	167.2 ± 7.4	362.9 ± 11.0
12	Işıklar	1.39 ± 0.37	1.14 ± 0.31	173.6 ± 17.9	69.8 ± 7.1	1.77 ± 0.22	14.0 ± 2.3	260.7 ± 15.7	146.1 ± 10.7	373.0 ± 15.4
13	Adacık	1.95 ± 0.99	1.62 ± 0.08	183.1 ± 18.9	27.5 ± 9.8	1.91 ± 0.03	25.4 ± 8.1	240.3 ± 8.2	151.7 ± 7.9	369.4 ± 1.6
14	Osmanbaba	1.44 ± 1.28	0.72 ± 0.29	171.7 ± 15.3	75.6 ± 5.7	1.90 ± 0.03	25.0 ± 1.3	279.2 ± 12.1	151.7 ± 15.8	361.6 ± 15.0
15	Yeşiltepe	2.55 ± 0.75	2.50 ± 0.52	190.7 ± 16.8	39.0 ± 9.6	1.84 ± 0.03	25.4 ± 5.9	250.8 ± 10.1	140.1 ± 10.6	358.6 ± 7.1
16	Mersin	2.53 ± 0.51	1.63 ± 0.13	206.5 ± 9.1	98.6 ± 13.8	1.89 ± 0.09	15.4 ± 3.4	243.4 ± 13.1	183.8 ± 9.7	345.4 ± 19.1

The amount of cadmium the highest value was found to be 2.36 ± 0.11 µg/L in Erikli (Term II). The lowest value was 1.32 ± 0.12 µg/L in Esentepe µg/L (Term II). For lead, the highest value was obtained 64.81 ± 3.59 µg/L in Arpacılı (Term II), and the lowest value was obtained 10.82 ± 3.30 g/L in Helvacı (Term II). The highest value alumnium was obtained 295.27 ± 13.87 µg/L in Yeşiltepe (Term II), and the lowest value was obtained 215.25 ± 11.95 µg/L in Demirkapı (Term II). According to results. The highest iron content was found 183.82 ± 9.68 µg/L in Mersin (Term I), and the lowest value was found 132.77 ± 9.01 µg/L in Yaylacık (Term I). Finally, when we look at the zinc values, the highest value was obtained 386.42 ± 18.28 µg/L in Erikli (Term I), and the lowest value was obtained 224.55 ± 13.47 µg/L in Yaylacık (Term II).

When the literature is examined, copper, iron, zinc, lead, cadmium, nickel, and aluminum have been found the determination of metal made in milk in different places [17-20]. The amount of chromium found in cow's milk is given in the range of 5-82 µg/L [19]. It has been observed that the amount of chromium in the milk in Akçaabat villages is far below these values. On the other hand, the average chromium contents of summer and winter periods were found as 1.88 ± 0.66 µg/L (Term I) and 1.29 ± 0.53 µg/L (Term II), respectively. Generally, higher results were found in winter.

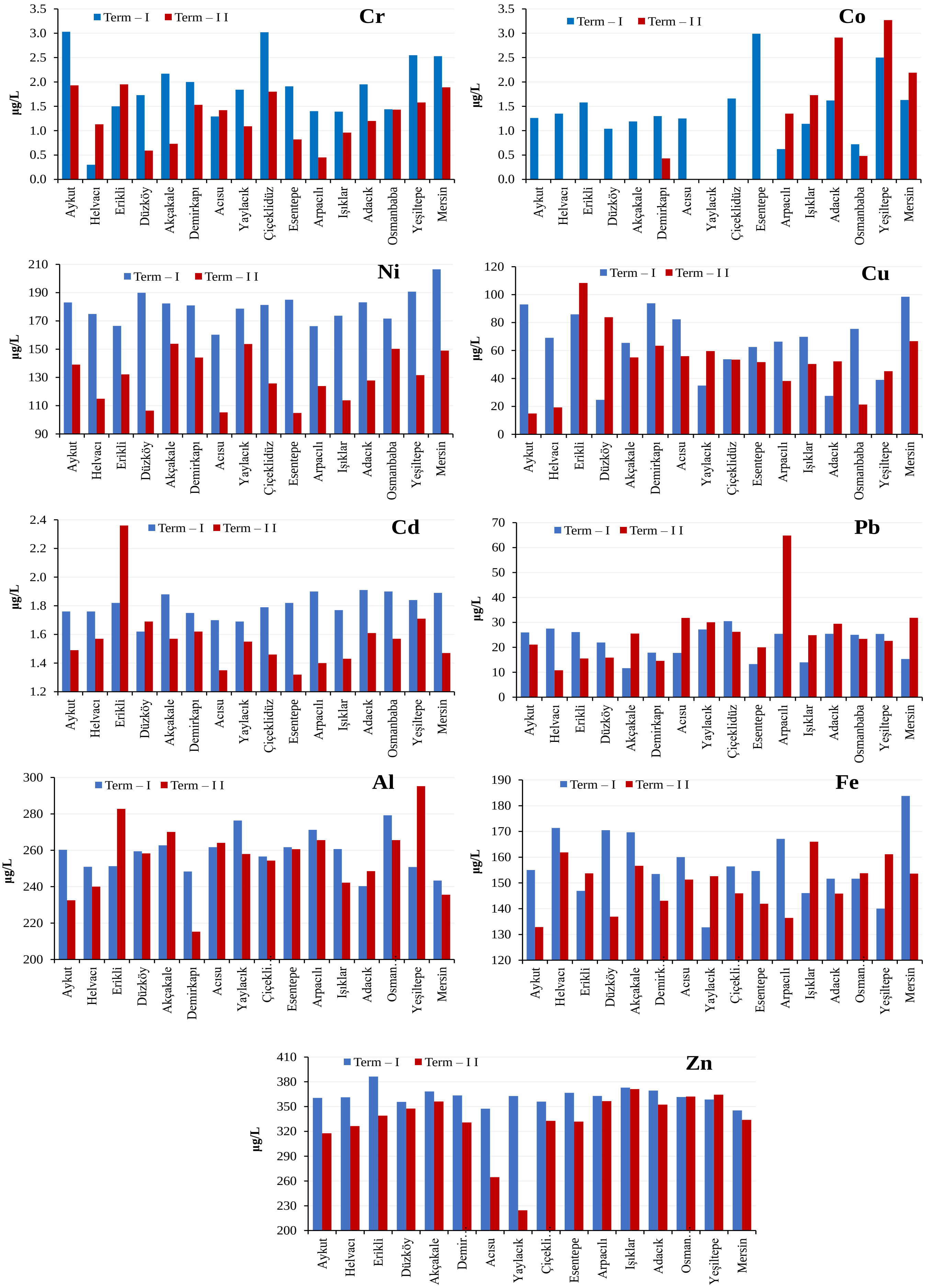

Figure 2. Graphical representation of the variation of metal concentrations

The amount of cobalt in cow's milk ranged from 0-10 μg/L, and this value was an average of 0.8 μg/L in milk [19,21]. It has been observed that the amount of cobalt in the milk from villages in Akçaabat is below or between these values. The mean cobalt levels for summer and winter periods were found as 1.36 ± 0.33 μg/L (Term I) 0.77 ± 0.15 μg/L (Term II). The winter period was about twice as much as the summer period.

The amount of nickel in cow's milk ranges from 0 to 36 μg/L. The amount in the cow's milk is reported to be well above 100 μg/L [5,22]. It has been observed that the amount of nickel in milk from villages in Akçaabat is much higher than these values. It is thought that this may be caused by nickel in drinking and potable water. The average nickel amounts for summer and winter periods were determined as 179.67 ± 12.46 μg/L (Term I) 129.79 ± 10.69 μg/L (Term II), and higher results were found in the winter period.

The amount of copper in the investigated milk ranges between 50-300 μg/L [5,22-23]. It was observed that the amount of copper in milk in Akçaabat villages was slightly below or much below these values. However, the average copper content in the summer and winter periods was found to be 65.14 ± 6.31 μg/L (Term I), 51.84 ± 6.44 μg/L (Term II), and generally higher values were found in the winter period.

The amount of cadmium for milk and dairies was set as 50 μg/L in Australia and 10 μg/L in Denmark, but in the Netherlands and Germany, it was set as 5 μg/L [24]. It has been observed that the amounts of cadmium in the milk in Akçaabat villages are well below the limits set in the Netherlands, Germany, Australia, and Denmark. The mean cadmium content of summer and winter periods was found to be 1.80 ± 0.12 μg/L (Term I), 1.58 ± 0.21 μg/ L (Term II). Higher results were found in winter.

The amount of lead allowed in milk is reported as 24 μg/L [25]. The amount of lead in milk was reported to be an average of 40 μg/L [22,26]. The average lead amounts for summer and winter periods were determined as 21.89 ± 3.61 (Term I) and 25.53 ± 4.3 (Term II), and periodically close values were found. The average lead amounts in the milk we analyzed do not exceed the upper limits given in the studies above. However, the amount of milk in the village Arpacıklı is exceeded the limits. It may be due to the contamination of containers or other reasons.

According to Aysal [19], the amount of aluminum in milk varies between 100-2100 μg/L, and the results of the analysis were found among these values. In summer and winter periods, the average amounts of aluminum in the village milk were 258.43 ± 12,72 μg/L (Term I), 255.54 ± 13.97 μg/L (Term II) and the results were close to each other periodically.

Previous studies showed that the average amount of iron in one liter of milk was 1400 μg [5,26]. The amount of iron in Akçaabat and its villages was found to be very below the stated value. In summer and winter periods, the average amounts of iron in the village milk were 156.96 ± 11.62 μg/L (Term I) and 149.62 ± 10.99 μg/L (Term II) and the results were close to each other periodically.

The average amount of zinc in cow's milk was 3500 μg/L according to Metin [26] and 3700 μg/L according to Yetişmeyen [5]. The amount of zinc in the milk determined in Akçaabat villages was measured below these values. Average values observed for zinc were found as 362.49 ± 13.77 μg/L (Term I) and 332.03 ± 14.90 μg/L (Term II). Higher results were found in winter.

As a result, it was found that average amounts of lead, aluminum, and iron in winter and summer milk are similar. Amounts of chromium, cobalt, zinc, nickel, copper, and cadmium are a little more in wintertime. Studied all elemental contents of cow’s milk samples collected from all villages were found to be below the values given in the literature.

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