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YJFCA 2517 1–5

Journal of Food Composition and Analysis xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

Journal of Food Composition and Analysis

journal homepage: www.elsevier.com/locate/jfca

Original Research Article

Content of macro- and microelements and evaluation of the intake of different dairy products consumed in Croatia

Q1 Nina Bilandžić *, Marija Sedak, Maja Đokić, Đurđica Božić, Antonija Vrbić

Laboratory for Residue Control, Department for Veterinary Public Health, Croatian Veterinary Institute, Savska cesta 143, HR-10000 Zagreb, Croatia

ARTICLE INFO

Article history:
Received 30 April 2014
Received in revised form 20 January 2015
Accepted 21 January 2015
Available online xxx

Keywords:
Macroelements
Microelements
Food composition
Dairy products from Croatia
Milk
Cheese
Butter
Inductively coupled plasma-optical emission spectrometry

ABSTRACT

Macromineral and microelement contents were measured by inductively coupled plasma-optical emission spectrometry in 51 dairy products (milk, four types of cheese and butter). Significant differences in the concentrations of Ca, K, Na, Mg, Zn and Cu were observed ($p < 0.05$, all) between dairy products. Higher levels of Ca, Na, Mg and Se were measured in hard cheese than in semi-hard, fresh and cream cheese samples. The highest mean levels of elements were measured in products (mg kg^{-1}): milk: K 2070; hard fat cheese: Ca 10,700, Na 10,300, Mg 440, Zn 91.3, Se 0.78; cream cheese: Cu 3.12, Fe 3.9; butter: Se 0.79. The estimated daily intakes (EDIs) calculated for milk show contribution to the recommended dietary allowance (RDA) for the elements (%): Ca 42, K 13.2, Mg 10.3–13.3, Zn 11.4–15.7, Se 13.1. The highest contribution of elements to the RDA values was determined for hard fat cheese (%): Na 7.93, Zn 16.6–22.8, Se 28.4. However, a contribution to the RDA of less than 1% was determined for K, Mg and Fe in cheeses. The contribution of Cu to the RDA value ranged in cheese samples between 2.78 and 6.93%, though this was less than 1% in milk.

1. Introduction

Q2 Due to the extensive use and nutritional importance of dairy products, knowledge of their macromineral and trace element composition is important, from both a nutritional and toxicological/safety point of view. The macro and microelement concentrations in milk and in dairy products depend on factors such as the genetic characteristics of lactating animals, environmental conditions, types of pasture, lactation stage, and manufacturing procedures and ingredients in the processing of these products (Demirozu-Erdinc and Saldamli, 2000; Merdivan et al., 2004; Suhaj and Koreňovská, 2008; Sola-Larrañaga and Navarro-Blasco, 2009). For example, the ultra filtration processes utilized in the dairy industry retain more Ca than the traditional filtering of coagulate through cheesecloth (Reykdal et al., 2011). During cheese production, high acidity is developed and Ca and Mg salts become more soluble and are therefore easily removed with the whey during rinsing (Suhaj and Koreňovská, 2008). On the other hand, the Na content in cheese depends on the addition of sodium chloride salt as an ingredient during processing. During cheese ripening, the pH gradient effects may cause changes in the concentration of certain elements in the final product (Moreno-Rojas et al., 1994; Pastorino et al., 2012).

Homemade cottage cheese is the most widespread product in traditional Croatian lowland cheese-making (Kirin, 2009). On the other hand, the main dairy industries in Croatia produce traditional and innovative dairy products and therefore contribute to characterization of the quality and adequacy of the consumer diet in Croatia. Over the past decade, mineral and trace element contents in cow cheese have been reported in different countries: Brazil (Cichoseki et al., 2002; Kira and Maihara, 2007), Iceland (Reykdal et al., 2011), Italy (Licata et al., 2004), France (Arnich et al., 2012; Chekri et al., 2012; Maas et al., 2011; Millour et al., 2012; Noël et al., 2012), Saudi Arabia (Aly et al., 2010), Spain (Prieto et al., 2002; González-Martín et al., 2011; González-Weller et al., 2013) and Turkey (Yüzbaşı et al., 2003; Merdivan et al., 2004; Mendil, 2006; Bakircioglu et al., 2011).

Data on minerals and trace elements in dairy foods from Croatia have been limited. The objectives of this study were: (I) to determine the macromineral and microelement concentrations in two dairy products and four kinds of cheese in Croatia, (II) to check the difference between the results of this study with previously reported data in dairy products around the world, and (III) to compare the estimated intakes with the reference nutritional values for each element.

* Corresponding author. Tel.: +385 1 612 3601; fax: +385 1 612 3636.
E-mail address: bilandzic@veinst.hr (N. Bilandžić).

http://dx.doi.org/10.1016/j.jfca.2015.01.003
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2. Materials and methods

2.1. Sample collection

A total of 51 dairy products produced by the four largest and other small dairy factories in Croatia were collected during summer 2013. The four basic dairy products were randomly collected from large and small marketplaces in the capital of Croatia, Zagreb and the neighbouring small towns: 4 hard fat cheeses, 13 semi-hard fat cheeses, 5 cream cheeses and 4 butters. Also, low-fat fresh cottage cheese ($n = 9$) and milk ($n = 16$) produced by four major and other small dairy factories were collected.

Following collection, samples were placed into clean polyethylene bottles and bags, labelled and stored at −18 °C until analysis.

2.2. Reagents

Analytical grade reagents used for sample dissolution, HNO_3 (65%, v/v) and H_2O_2 (30%, v/v) were purchased from Kemika (Zagreb, Croatia). All solutions were prepared and diluted with ultra-pure water (18 MΩ cm) generated by the system NIRO VV UV UF 20 (Nirosta d.o.o. Water Technologies, Osijek, Croatia).

Standard stock solutions containing 1000 mg L^{-1} of Ca (998 ± 5 mg L^{-1}), K (999 ± 5 mg L^{-1}), Na (1000 ± 4 mg L^{-1}), Mg (1003 ± 6 mg L^{-1}), Cu (1002 ± 5 mg L^{-1}), Zn (998 ± 4 mg L^{-1}) and Fe (1002 ± 5 mg L^{-1}) were purchased from Inorganic Ventures (Christiansburg, VA, USA). Standard solutions containing 1000 mg L^{-1} 1 of Se (990 ± 2 mg L^{-1}) was supplied from Sigma–Aldrich (Buchs, Switzerland). The stock solution and working standards were diluted in HNO_3 (0.5%, v/v).

2.3. Element analysis

Samples of dairy products (2 g) were weighed in a PFA digestion vessel and 1 mL of H_2O_2 and 6 mL of HNO_3 were added. A high-pressure laboratory microwave oven Multiwave 3000 (Anton Paar, Ostfildern, Germany) was employed to perform the acid digestion of samples. The digestion program was: step 1 power 800 W, ramped 15 min, 800 W for 15 min; step II power 0 W for 15 min. Digested samples were diluted with ultra-pure water to a volume of 50 mL. Analytical batches contained a blank sample and two spiked samples. All element concentrations were determined on a wet weight basis. An Optima 8000 (Perkin Elmer, Waltham, MA, USA) inductively coupled plasma optical emission spectrometer (ICP-OES) was used for element determination. The instrumental experimental conditions are summarized in Table 1.

Table 1
Instrumental conditions for measurements with inductively coupled plasma optical emission spectrometer.

Analyte	Ca, K, Na, Mg	Cu, Fe, Zn, Se
Parameter	Intensity	Intensity
Plasma viewing mode	Radial	Axial
Read time	1–5 s	1–5 s
Measurement replicates	3	3
RF incident power	1000 W	1300 W
Plasma argon flow rate	8 L min^{-1}	15 L min^{-1}
Nebulizer argon flow rate	0.85 L min^{-1}	0.55 L min^{-1}
Auxiliary argon flow rate	0.2 L min^{-1}	0.2 L min^{-1}
Sample uptake rate	1.5 mL min^{-1}	1.5 mL min^{-1}
Inner diameter of the torch injector	2.0 mm	2.0 mm
Nebulizer type	Concentric glass (Meinhard)	Concentric glass (Meinhard)
Spray chamber type	Glass cyclonic spray chamber	Glass cyclonic spray chamber

Table 2
Concentrations of elements (mean ± SD) in certified reference material (CRM).

Element	Unit	Certified value	Determined value ($n = 5$)	Recovery (%)
Ca	g kg^{-1}	13.49 ± 0.10	13.3 ± 0.34	98.6
K	g kg^{-1}	17.68 ± 0.19	17.4 ± 1.2	98.4
Na	g kg^{-1}	4.37 ± 0.031	4.20 ± 0.21	96.1
Mg	g kg^{-1}	1.263 ± 0.024	1.18 ± 0.081	93.4
Zn	mg kg^{-1}	49.0 ± 0.6	48.5 ± 1.3	98.9
Fe	mg kg^{-1}	2.32 ± 0.23	2.28 ± 0.15	98.6
Cu	mg kg^{-1}	0.602 ± 0.019	0.588 ± 0.016	97.7
Se	μg kg^{-1}	(129)	128 ± 6.5	99.2

Calculation of detection limits levels were performed according to three times the standard deviation of ten blanks. The limits of detection were (mg kg^{-1}): Ca 0.01, Na 0.01, K 0.025, Mg 0.02, Cu 0.01, Fe 0.005, Zn 0.005 and Se 0.005.

In order to check the accuracy of the analytical procedure, the certified reference material of skim milk powder (CRM 063R, Institute for Reference Materials and Measurements, Geel, Belgium) was analysed. Good agreement was obtained between certified and analysed values for the elements measured (Table 2). The average elemental recovery ranged between 93.4 and 99.2%.

2.4. Calculation of estimated daily intake

The estimated daily intake (EDI) was calculated by the equation:

$$\text{EDI}\,(\text{mg day}^{-1}) = (\text{element concentration; mg kg}^{-1}) \\ \text{per}\,(\text{meal size or daily intake of milk and cheese; kg day}^{-1}).$$

The average consumption of milk and cheese in Croatia was set as 0.3 L day^{-1} and 0.020 kg day^{-1} per adult (Antonić Degač et al., 2007). The values of EDI were used to calculate contributions of each element to the reference nutritional (RDA, Recommended Dietary Allowance, for females and males; UL, Tolerable Upper Intake Level).

2.5. Data analysis

Statistical analysis was conducted by Statistica 6.1 software (StatSoft® Inc., Tulsa, OK, USA). Concentrations were expressed as mean ± SD. One-way ANOVA was used to test for statistical differences in element levels between dairy products. Results were considered significant at $p < 0.05$.

3. Results and discussion

The concentrations of macro and microelements measured in milk, butter and four kinds of cheese commercially produced and consumed in Croatia are presented in Table 3.

3.1. Macrominerals

Inadequate dietary intakes of the macroelements Ca, Mg, Na and K, either insufficient or excessive, can cause serious health issues such as hypertension, osteoporosis and cardiovascular disease (Park, 2009). These minerals are involved in physiological processes in the body, the most important of which are muscle contraction, nerve conductance, pH maintenance, osmotic pressure and energy production (Institute of Medicine, 2005).

Milk samples were rich in the minerals Ca, Na, Mg and especially K, with levels 2.2–4.1 times higher than the tested cheese samples. In comparison to the results of studies from other countries, Ca and Mg were similar, though Na and K levels were