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Determination of minerals in infant milk formulae by energy dispersive X-ray fluorescence spectrometry

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ABSTRACT

Commercial milk formulae hold a major role in the diet of many infants in developed countries and assessing their mineral content is important in order to ensure compliance with the manufacturer claims and with regulation guidelines. In this study, the concentrations of K, Ca, Fe, Zn, Br, Rb and Sr were determined by Energy-Dispersive X-Ray Fluorescence (EDXRF) spectrometry in 28 infant milk formulae widely distributed in the Greek market. The concentration ranges (in mg kg^{-1}) measured for K (2180–7600), Ca (3240–7480), Fe (29–98) and Zn (38–84) were within the limits recommended by the European legislation, with only a few exceptions. Although measured values were generally consistent with those labelled by the manufacturers, discrepancies over 50% to 100% were observed in some cases and were analyzed by multivariate statistical methods. The findings indicate the need for close monitoring to guarantee good manufacturing practices and constant quality of infant formulae. The daily intakes of minerals from consumption of different types of milk formulae were also estimated and the overall ranges were (0.23–0.85) g d^{-1} for K, (348–736) mg d^{-1} for Ca, (3.2–10.5) mg d^{-1} for Fe and (4.1–9.0) mg d^{-1} for Zn. These values conform to currently recommended safety limits.

1. Introduction

Infancy is probably the most demanding period of humans' life from the nutritional point of view, since the body weight typically doubles during the first 4 to 6 months of life and triples by the end of the first year. Providing an appropriate diet for a growing infant is critical in terms of healthy growth and development, given that low intake or reduced bio-availability of nutrients may lead to deficiencies and body function impairment. The WHO/UNICEF Global Strategy for Infant and Young Child Feeding recommends exclusive breastfeeding for the first six months of life, followed by continued breastfeeding for up to two years of age or beyond, as complementary foods are progressively introduced in the infant's diet (WHO, 2003). Extensive research has demonstrated multiple and compelling benefits of breastfeeding for the infant, the mother, the community and the environment. There is mounting evidence that breastfeeding of infants provides advantages with regard to general health, growth and development, while significantly reducing the risk for a number of acute and chronic diseases, such as gastroenteritis, acute otitis media, lower and upper respiratory tract infections, urinary tract infection, atopic dermatitis, sudden infant death syndrome, childhood leukaemia, type 1 and type 2 diabetes and obesity in adult life (Kramer and Kakuma, 2004; Ip et al., 2007; American Academy of Pediatrics, 2012).

Despite the strong endorsement of breastfeeding by major health organizations, current rates are far from optimal, worldwide. Less than 40% of infants are exclusively breastfed at the age of 6 months in developing countries (WHO, 2009), while merely 13% of US infants (CDC, 2010) and 13% of EU infants (Bagci Bosi et al., 2016) are exclusively breastfed at this age. In Greece, a large national study, conducted by the Institute of Child Health and the National School of Public Health, reported that exclusive breastfeeding rates were 41% during the first day of life and declined to 21, 11 and 0.8% by the end of 1, 3 and 6 months of age, respectively (Gaki et al., 2009). These facts point out that infant formulae are widely used as a substitute for human milk and hold a major role in infants' diet during the first year of life. Concerns about the nutritional adequacy of commercial products have triggered scientific research addressing, among others, the mineral content of infant milk formulae and their compliance with reference values. Minerals are essential for biological processes, since they are involved in significant body functions, e.g. bone mineralisation, enzymatic reactions, secretion of hormones, as well as protection of cells and lipids in biological membranes (Schlenker and William, 2003).

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A list of elemental analysis techniques has been implemented for mineral determination in milk and infant milk formulae, including inductively coupled plasma (ICP) mass spectrometry, ICP-optical emission and ICP-atomic emission spectrometry (McKinstry et al., 1999; Hua et al., 2000; Ikem et al., 2002; Sola-Larrañaga and Navarro-Blasco, 2006; Melø et al., 2008; Al Khalifa and Ahmad, 2010; Lesniewicz et al., 2010, Ljung et al., 2011; Zand et al., 2011; Pandelova et al., 2012; Pereira et al., 2013; Bu-Hamdi et al., 2016; Bargellini et al., 2018; Chajduk et al., 2018; Pacquette et al., 2018), atomic absorption spectrometry (Tripathi et al., 1999; Sola-Larrañaga and Navarro-Blasco, 2006; Saracoglu et al., 2007; Moraes et al., 2009; Moreno-Rojas et al., 2015; Machado et al., 2016; Ahmed et al., 2017), instrumental neutron activation analysis (Santos et al., 2008; Chajduk et al., 2018), radioisotope-induced energy-dispersive X-ray fluorescence (EDXRF) spectrometry (Khan et al., 1989; Ekinci et al., 2005; Mohammed et al., 2006), X-ray tube-induced EDXRF spectrometry (Alvarez and Mazo-Gray, 1990; Perring and Monard, 2010; Rossmann et al., 2016) and wavelength-dispersive X-ray fluorescence spectrometry (Perring and Andrey, 2004; Perring and Blanc, 2008; Pashkova, 2009; Fernandes et al., 2015).

To our knowledge, there are no published data on the mineral content of infant milk formulae available in the Greek market. The aim of the present study was to determine the mineral composition of commercial infant milk formulae using Energy-Dispersive X-Ray Fluorescence (EDXRF) spectrometry, to compare the measured values with the corresponding values labelled by manufacturers and with those described in the European legislation and to evaluate the commercial infant formulae considering nutritional requirements and daily permissible doses.

2. Materials and methods

2.1. Collection and preparation of samples

Twenty eight infant milk-based powder formulae from ten different brands marketed through Greek pharmacies, were randomly selected and analyzed for their mineral content. Standard ($n = 13$) and specialized ($n = 15$) formulae were assayed, which were further distinguished to starter (0–6 months of age, $n = 12$), follow-on (6–12 months of age, $n = 7$) and from birth-to-first-year ($n = 9$). All products were manufactured from cow's milk proteins or protein hydrolysates. A detailed description of the analyzed formula powders is presented in Table 1. For confidentiality reasons, the commercial names of the sampled milk powder formulations shall not be disclosed. Milk powders were contained in metal cans and were manually homogenized by agitation, prior to sampling, in order to get a representative sample from each product. Two powder samples of each milk formulation were examined.

Sample pellets, 1.25 cm in diameter, were prepared by mixing 0.3 g of milk powder with microcrystalline cellulose at a ratio of 10%w/w and pressing at 8 tonnes using a manual hydraulic press (Specac, UK). No special sample treatment or drying was applied prior to pelletizing.

2.2. EDXRF measurements

Elemental analyses were carried out using a home-built Energy-Dispersive X-Ray Fluorescence (EDXRF) spectrometry arrangement. Photons emitted from an 20 mCi annular ^{109}Cd source were used for sample excitation. The source has a radius of 21 mm and is housed in a cylindrical, aluminum container, fixed coaxially above a Canberra SL80175 Si(Li) detector (Canberra Industries, Inc., Meriden, Connecticut, USA) (5 mm crystal thickness, 80 mm^2 area), with a 25 μm-thick Be window and an energy resolution of 171 eV for the 5.9 keV Mn K_α line. Samples were placed on top of the assembly at a distance of 12 mm from the source plane. The overall experimental setup (Fig. 1) is operated in open air. The Si(Li) detector is coupled with NIM standard high voltage power supply and amplifier modules. An ORTEC Trump 8k ISA card multichannel analyzer (ORTEC, Oak Ridge, USA) and the ORTEC MAESTRO-32 emulation software package were used for data acquisition. Spectral analysis was carried out using the WinQxas software package developed by the International Atomic Energy Agency (IAEA, Vienna, Austria). Qualitative analysis revealed a similar elemental pattern among all samples and K, Ca, Fe, Zn, Br, Rb and Sr were the elements detected (Fig. 2).

Table 1
Labelling information on products included in the present study.

Brand ID	Product ID	Age (mo)	Sample information
C1	M1	0-6	Basic formula
	M2	0+	Anti-constipation formula
	M3	0-12	Formula to ease baby's stomach discomfort
	M4	0-6	Formula with partially-hydrolyzed cow's milk protein for babies with allergy or intolerance to cow's milk
	M5	0-6	Formula to prevent allergy to cow's milk, for babies with family history of allergy
C2	M1	0-6	Organic formula
	M2	6+	Organic formula
C3	M1	0-6	Basic formula
	M2	6-12	Basic formula
	M3	0+	Lactose free formula
C4	M1	6-12	Basic formula
	M2	0+	Anti-reflux formula
C5	M1	0+	Formula for Crohn's disease
	M2	0-6	Basic formula
C6	M1	0-6	Basic formula
	M2	6-12	Basic formula
	M3	0-12	Anti-colic formula
	M4	0+	Anti-reflux formula
	M5	0-6	Anti-constipation formula
	M6	0-12	Formula for allergy to cow's milk protein/ anti-reflux
	M7	0-6	Formula enriched with prebiotics (GOS) DHA & ARA and nucleotides
	M8	6-12	Formula enriched with prebiotics (GOS) DHA & ARA and nucleotides
C7	M1	6-12	Basic formula
C8	M1	0+	Formula with extensively-hydrolyzed cow's milk protein
	M2	0-6	Anti-reflux, anti-constipation formula
C9	M1	0-6	Basic formula
	M2	6-12	Basic formula
C10	M1	0-6	Organic formula

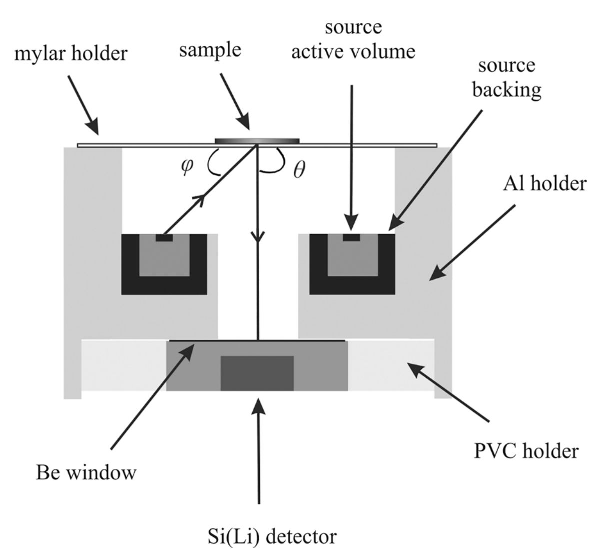

Fig. 1. Schematic drawing showing the vertical cross-section of the radioisotope-induced EDXRF spectrometry setup.