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Determination of Key Chemical Elements by Energy Dispersive X-Ray Fluorescence Analysis in Commercially Available Infant and Toddler Formulas consumed in UK

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Abstract

We have developed and validated an energy dispersive X-Ray fluorescence (EDXRF) method for the rapid determination of concentrations of key chemical elements (Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn) in milk-based products. To demonstrate the utility of the EDXRF method for the chemical element determination in milk product, concentrations of chemical elements in two infant formulas (infant first milk and toddler milk) widely marketed in the UK were investigated. The infant first milk formula was found to have significantly high levels of Cu, Fe, Mg, Se and particularly high concentrations of Ca and P, which were nearly six and twenty times higher than the manufacturer's label declaration, respectively. By contrast, analysis of the chemical elements concentrations in the toddler milk formula was generally close to the values declared by the manufacturer with the exception of Cu and Mg, which were about three and two times higher than declared on the label, respectively. We discuss the obtained results in the light of specific nutritional needs of infants and data reported in literature.

Keywords: Infant formula products; Chemical element concentrations; Energy-dispersive X-Ray fluorescent analysis

Abbreviations: EDXRF: Energy-Dispersive X-Ray Fluorescent Analysis; AAS: Atomic Absorption Spectrometry; ICP-OES or ICP-AES: Inductively Coupled Plasma Optical/Atomic Emission Spectrometry; ICP-MS: Inductively Coupled Plasma Mass Spectrometry

Introduction

Milk and dairy products are the unique source of nutrients, including chemical elements, and play an essential role in growth, development and health maintenance of human body, particularly during the first months and years of life [1]. Breastfeeding ensures the best possible health as well as the best developmental and psychosocial outcomes for the infants [1,2]. Human breast milk provides an adequate intake of all nutrients (e.g., protein, lipids, and carbohydrates) and micronutrients, including major and trace elements, and naturally serves as a nutritional reference for development of infant formulas.

About 50 years ago a postulate of chemical element homeostasis in human body tissues and fluids in normal environmental and health conditions was formulated [3]. It was suggested that the homeostatic levels of chemical elements depend on many factors such as gender, age, race, diet, lifestyle, climate, biogeochemical and environmental characteristics of habitation, occupational exposure to certain chemical compounds and so on. It also was supposed that all chemical elements are actively or inactively/indirectly involved in normal metabolism of human tissues and are not just the building material. Chemical elements have essential physiological functions such as maintenance and regulation of cell function and signalling, gene regulation, activation or inhibition of enzymatic reactions, neurotransmission, and regulation of membrane function. Essential or toxic (mutagenic, carcinogenic) properties of chemical elements depend on tissue-specific need or tolerance, respectively. Excessive accumulation, deficiency or an imbalance of the chemical elements may disturb the cell functions and may result in cellular degeneration or death [3]. Because chemical elements play the vital role in human health as well as in normal growth and development, there is a growing interest in their determination in food, and especially in breast milk and infant formulas.

On the one hand, up-to-date analytical technologies are very important tool for the extensive research on the wide list of chemical elements in human milk in normal environmental and health conditions as well as changes of normal levels of chemical element concentrations during breastfeeding. Such studies need to clarify the ideal chemical element concentrations in breast milk which have to serve as reference/ideal levels to develop and produce infant formulas. On the other hand, from a nutritional point of view, there are hundreds of infant formulas produced by numerous manufactures in different countries and marketed everywhere. Thus, conventional analytical measurements are essential to monitor the most important and potentially toxic chemical elements to control the quality of infant formulas, both in manufacturing and trade. Such methods and measurements have to ensure the high accuracy of analytical results, to be simple, fast, and cheap as well as to guarantee quality to the end users.

Current methods applied for measurement of chemical element concentrations in milk and infant formulas include a number of methods, such as flame photometry (Na and K) [4], flame atomic absorption spectrometry - AAS (Ca, Cu, Fe, K, Mg, Mn, Na, and Zn) [4-7], graphite furnace atomic absorption spectroscopy (Al, Cd, Co, Cr, Fe, Mn, Mo, and Pb) [7-11], differential pulse anodic stripping voltammetric technique (Zn, Cd and Pb) [5,12], stripping potentiometry (Cd, Pb, and Cu) [13], direct potentiometric method.(F and Cl) [6,14], capillary zone electrophoresis (Ca, K, Mg, and Na) [15], laser-induced breakdown spectroscopy (Ca, Fe, Mg, Na) [16] inductively coupled plasma optical/atomic emission spectrometry - ICP-OES or ICP-AES (Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, P, Se, and Zn) [17-22], inductively coupled plasma mass spectrometry - ICP-MS (Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Hg, Mn, Mo, Ni, Pb, Sb, Sn, Tl, V, and Zn) [2,21,23-28], instrumental neutron activation analysis INAA (Ca, Cl, Co, Cr, Cs, Fe, Hg, K, Mg, Mn, Na, P, Rb, Sb, Sc, Se, and Zn) [18,29-32], and energy dispersive X-ray fluorescence technique - EDXRF (Ca, Cl, Fe, K, P, S, and Zn) [33-35].

Among these methods the EDXRF is one of the simplest, fast, reliable, efficient, and available techniques. There are many different kinds of EDXRF devises on the market and technical possibilities of this method improve rapidly. Therefore, the present study had four aims.

The first and main aim was to investigate capabilities of a new spectrometer Shimadzu EDX-7000 for the spectroscopic determination of chemical element contents in milk products and development of adequate techniques for a precise determination of available chemical element contents in milk products using this device. The second aim was to test chemical element contents in the randomly chosen infant and toddler formula distributed in UK supermarkets. The third aim was to calculate selected ratios of chemical element contents in infant and toddler formula milks, and the last aim was to compare measured chemical element contents in infant formula with published data.

Materials and Methods

Sample collection

Two brands of liquid (ready-made) infant formula samples (infant first milk and toddler milk) were purchased from one of major supermarket chain in suburbs of Milton Keynes (UK). Both infant formulas were milk-based types. For an estimation of precision and accuracy of the method, a European reference material ERM®-BD151 (skimmed milk powder) was analyzed. The ERM®-BD151 was purchased from LGC Standards (UK).

Sample preparation

To evaluate of Ca, Cl, K, P, and S concentration in the infant formula samples measurements were performed using "thick layer" method [36]. Therefore 250 μL of each infant formula sample was poured into a receptacle (for liquid samples) sealed with 3 μm polypropylene film (Figure 1). For determination of the mass fraction of the same elements in the reference material, skimmed milk powder ERM®-BD151 was diluted in deionized water in ratio 1:6 and 250 μL of obtained suspension was poured into a receptacle for liquid samples.

To evaluate of Br, Cu, Fe, Mg, Se, and Zn concentration in infant formula samples and ERM®-BD151 measurements were performed using "thin layer" method [36-38]. 50 μL of each infant formula sample or ERM®-BD151 suspension sample was dropped onto a filter paper disk mounted on an adhesive tape, dried at 80°C for 10 minutes and spanned on a frame (Figure 2).

The measurements of Ca, Cl, K, P, and S concentration in infant formula samples were made in triplicate. For estimation of Br, Cu, Fe, Mg, Se, and Zn concentration in infant formula six sub-samples and for the ERM®-BD151 ten sub-samples were prepared and measured.

EDXRF analysis of samples

Chemical element contents in infant formula samples and ERM®-BD151 sub-samples were determined using "Fundamental Parameters" and "Peak Fitting" options. The EDXRF measurements were performed on a Shimadzu EDX-7000 spectrometer in two ways at conditions/settings presented in table 1.

Statistical analysis

The descriptive statistics that include means (M), standard deviations SD), relative standard deviations (RSD), standard error of mean (SEM), minimum (Min), and maximum (Max),of the data were computed using Microsoft Excel 2002.

Results and Discussion

The mass fractions of eleven elements (Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn) that cover the range of 8 elements (Ca, Cl, Cu, Fe, K, Mg, P, and Zn) with certified values in ERM®-BD151 (skimmed milk powder) were determined. The result was generally in agreement with ERM®-BD151 certified values with the exception of Cl and Mg. The founded mean level of the Cl mass fraction in ERM®-BD151 was 22.5% lower while the determined mean level of the Mg mass fraction was 29.4% higher than the corresponding certified values. Generally good agreement with the certified data of reference material indicate an acceptable accuracy of the results obtained in the study of chemical element concentrations in the infant first milk and toddler milk presented in tables 2 and 3.

Values for Cl, K, and Zn concentrations obtained by EDXRF in infant first milk (Table 2) were similar to those declared by the formula producer (differences averaged from -10% to -22%). The measured Ca, Cu, Fe, Mg, P, and Se concentrations were 520%, 270%, 96%, 67%, 1871%, and 140% higher, respectively, than those reported by the infant first milk producer. The EDXRF results for Ca and P were particularly surprising because they were in 6.2 and 19.7 times higher in comparison with the product's label data.

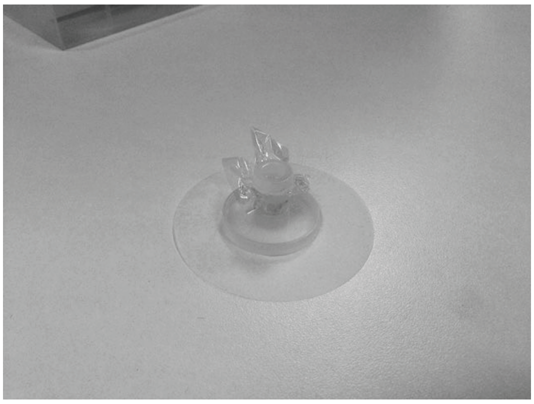

Figure 1: A receptacle for liquid sample used for a measurement in conditions of "thick layer" by EDXRF spectrometer Shimadzu EDX-7000.

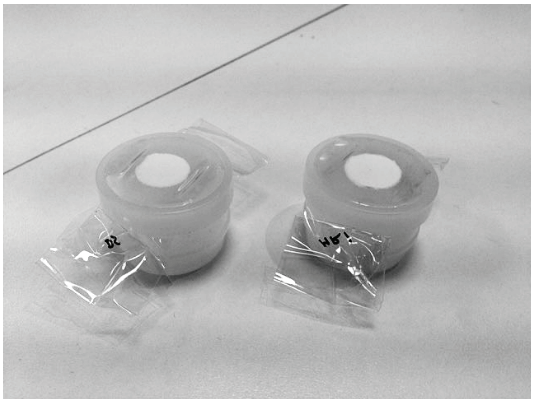

Figure 2: Solid residual samples prepared for a measurement in conditions of "thin layer" by EDXRF spectrometer Shimadzu EDX-7000.

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Parameter	Measurement in "Thick layer" (Liquid samples)	Measurement in "Thin layer" (Liquid samples after drying)
Elements	Ca, Cl, K, P, and S	Br, Cu, Fe, Mg, Se, and Zn
Sample volume	250 µL	50 µL
Filter	2	no filter/4
Measuring time	300 sec	300 Sec/300 Sec
Atmosphere	He	He
Drying	-	80°C 10 Min

Table 1: Instrument operating conditions applied for chemical element determination by EDXRF spectrometer EDX-7000 (Shimadzu)

Element Ratio	This work result						Label M_l	Difference *Δ, %	Ratio M_m/M_l
	n	M_m	SD	SEM	Min	Max			
Br	6	0.56	0.06	0.02	0.52	0.63	-	-	-
Ca	3	3100	29	17	3072	3131	500	520	6.2
Cl	3	319	3.0	1.9	315	320	410	- 22	0.78
Cu	6	1.48	0.04	0.02	1.4	1.5	0.4	270	3.7
Fe	6	10.8	1.1	0.5	9.9	12.3	5.5	96	1.96
K	3	609	4.5	2.6	605	614	680	- 10	0.90
Mg	6	83.5	8.8	3.6	73.1	92.7	50	67	1.67
P	3	5518	52	30	5482	5577	280	1871	19.7
S	3	203	8.1	4.7	194	210	-	-	-
Se	6	0.036	0.014	0.006	0.027	0.055	0.015	140	2.4
Zn	6	4.32	0.15	0.06	4.10	4.50	5.0	- 13.6	0.86
P/S	3	27.2	1.0	0.6	26.1	28.2	-	-	-
Ca/Cl	3	9.73	0.19	0.11	9.59	9.94	1.22	698	7.98
Ca/K	3	5.09	0.09	0.05	5.00	5.17	0.74	588	6.88
Ca/Mg	3	39.4	5.2	3.0	33.4	42.8	10	294	3.94
Ca/P	3	0.562	0.002	0.001	0.560	0.564	1.79	-69	0.31
Mg/Zn	6	19.3	1.7	0.7	17.4	21.6	10	93	1.93
Fe/Zn	6	2.50	0.35	0.14	2.24	2.98	1.1	127	2.27
Fe/Cu	6	7.26	0.94	0.38	6.60	8.71	13.8	- 47	0.53
Zn/Cu	6	2.91	0.07	0.03	2.80	3.00	12.5	-77	0.23

Table 2: EDXRF data of the Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn concentrations (mg/L) and selected chemical element concentration ratios in infant first milk compared to product's label
n: number of sub-samples; M_m: arithmetic mean of measured data; SD: Standard deviation of mean; SEM: Standard error of mean; Min: Minimum; Max: Maximum; M_l: Arithmetic mean of product's label; Difference $*\Delta=[(M_m-M_l)/M_l] \times 100\%$.

Element Ratio	This work result						Label M_l	Difference *Δ, %	Ratio M_m/M_l
	n	M_m	SD	SEM	Min	Max			
Br	6	0.926	0.036	0.015	0.875	0.962	-	-	-
Ca	3	961	22	13	936	978	780	23	1.23
Cl	3	420	4.0	2.3	418	425	550	- 24	0.76
Cu	6	1.65	0.05	0.02	1.60	1.70	0.50	230	3.3
Fe	6	15.1	0.12	0.05	14.9	15.2	12.0	26	1.26
K	3	892	15	8.6	877	907	900	-0.9	0.99
Mg	6	131	8.6	3.5	121	140	65	102	2.02
P	3	620	9.1	5.2	610	628	500	24	1.24
S	3	161	5.0	2.9	156	165	-	-	-
Se	6	≤0.027	-	-	-	-	0.015	-	-
Zn	6	7.37	0.16	0.07	7.20	7.60	9.3	- 21	0.79
P/S	3	3.85	0.17	0.10	3.69	4.03	-	-	-
Ca/Cl	3	2.29	0.05	0.03	2.24	2.34	1.42	61	1.61
Ca/K	3	1.08	0.009	0.005	1.07	1.09	0.87	24	1.24
Ca/Mg	3	7.60	0.53	0.31	7.01	8.03	12.0	- 37	0.63
Ca/P	3	1.55	0.05	0.03	1.50	1.60	1.56	-0.6	0.99
Mg/Zn	6	17.8	1.1	0.4	16.3	18.6	6.99	155	2.55
Fe/Zn	6	2.05	0.04	0.02	2.00	2.11	1.29	59	1.59
Fe/Cu	6	9.14	0.31	0.13	8.77	9.50	24.0	-62	0.38
Zn/Cu	6	4.47	0.14	0.06	4.29	4.69	18.6	- 76	0.24

Table 3: EDXRF data of the Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn concentrations (mg/L) and selected chemical element concentration ratios in toddler milk compared to product's label
n: number of sub-samples; M_m: arithmetic mean of measured data; SD: Standard deviation of mean; SEM: Standard error of mean; Min: Minimum; Max: Maximum; M_l: Arithmetic mean of product's label; Difference $*\Delta=[(M_m-M_l)/M_l] \times 100\%$.

In general, EDXRF results for the chemical element concentrations of toddler milk were consistent with data reported on the label with the only exception of Cu and Mg (Table 3). Values for Ca, Cl, Fe, K, P, and Zn concentrations in toddler milk were similar to those declared by the producer (differences averaged from -24% for Cl to 26% for Fe), while concentrations of Cu and Mg were in 3.3 and 2.0 times higher, respectively, than product's label data.

Because the values of many chemical element concentrations reported by the infant first milk producer did not agree with our results obtained by EDXRF it was decided to compare available data on chemical element contents in the infant formulas published for the last 20 years [5,6,14,17,22,39-43]. For proper comparison the element levels in all powder formulas were converted by us from dry mass basis to concentration in mg/L. If the specified feeding tables supplied by the powder infant formula manufacturers were not presented in the cited paper, the concentration in mg/L were calculated using the median of published data for water content in different infant formulas 85.7% (or dilution 1:6) [42].

Table 4 depicts median, minimum and maximum value of means of the Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn concentrations (mg/L) in formula milk for infants according to data from the literature in comparison with our results. The concentrations of Fe, K, Mg, and Zn in the infant first milk formula investigated in this study were similar to medians of reported mean values for the infant formulas. Also concentrations of Ca and Se were found to be within the reported ranges of means (Table 4). Our result for the Cu concentration was 2 times higher than the upper reported mean of this trace element concentration in infant milk formulas. Only one study was found, where the Cl content in infant formulas was measured by a potentiometric method using digestion-dissolution samples [14]. Our result for the Cl concentration was 5 times higher than mean reported in the cited study. The largest and most surprising difference was shown for P. In the infant first milk formula P concentration was 6 times higher than the upper reported mean of this chemical element concentration in infant milk formulas. No data was found about the Br and S concentrations in infant milk formulas.

Chemical elements are known to compete with each other for pathways of absorption and transport [3]. For example, high intakes of Fe and Zn can interfere with Cu absorption [44]. It therefore seems prudent to express chemical element concentrations of the infant formulas in ratios that can be regarded as biologically sound. Within reason, the ratios for these elements in human milk can serve as guidelines for issuing of recommendations. The selected ratios of chemical element concentrations such as P/S, Ca/Cl, Ca/K, Ca/Mg, Ca/P, Mg/Zn, Fe/Zn, Fe/Cu/, and Zn/Cu were calculated for each measured sample of the infant first milk and toddler milk formulas and some main statistical characteristics of obtained results were presented in tables 2 and 3, respectively. The same ratios were also calculated using information given on product labels about mean chemical element concentrations (tables 2 and 3).

The mean values of ratios obtained from EDXRF analysis of the infant first milk very differ from results obtained from product's labels. For example, mean values of Mg/Zn, Fe/Zn, Ca/Mg, Ca/K, and Ca/Cl ratios obtained by EDXRF were nearly 2 to 8 times higher while mean values of Fe/Cu, Ca/P, and Zn/Cu nearly 2 to 4 times lower than values calculated from product's labels data. The mean values of Ca/P and Ca/K ratios obtained from EDXRF analysis of the toddler milk were similar to values calculated from product's labels data, while Fe/Zn, Ca/Cl, and Mg/Zn ratios were nearly 1.6 to 2.6 times higher and Ca/Mg, Fe/Cu, and Zn/Cu Zn ratios were nearly 1.6 to 4 times lower. When chemical element ratios in the infant first milk and toddler milk formulas were compared with data of literature different results were obtained. For example, the Zn/Cu ratio in the infant first milk and toddler milk formulas determined in this work (7.3 and 9.1, respectively) agreed well with the range of reported data for infant formulas manufactured in the United States (7.6-9.5) [45]. The Ca/P ratio in the infant first milk (0.56) was lower while in the toddler milk (1.5) it was equal to the ratio (1.3-1.5) one presented in cited study [45]. The Fe/Zn ratio in the infant first milk and toddler milk formulas obtained in this work (2.50 and 2.05, respectively) was at the upper limit of the range reported for infant formulas manufactured in the United States (0.33-1.41) [45].

Due to rapid growth and development, requirements for essential chemical elements particularly put infants at risk of deficiencies or excess of certain chemical elements. It is therefore essential that infant formulas contain chemical elements in amounts that satisfy their nutritional requirements without leading to adverse effects. In spite of existing standardization of infant formulas, which began from 1980s in the United States, and definite allowable Min and Max levels [46], chemical element concentrations in manufactured products vary very widely (Table 4). For example, the Max/Min ratios of Ca and Zn concentrations in infant formulas observed by us were nearly two orders of magnitude. There are two reasons for such great variation of reported data. One reason may be a carelessness of the infant formula's producers. The second reason, which should be taken into account, may originate in the analytical uncertainties. Majority of the official analytical methods currently used and validated for the determination of major and trace elements in infant formulas are based on AAS, ICP-AES, and ICP-MS techniques after sample digestion [46]. The most frequently used digestion procedures are the traditional dry ashing and high-pressure wet digestion that allow destruction of organic matter of the sample. Sample digestion is a critical step in elemental analysis and due to the risk of contamination and analytes loss contributes to the systematic uncontrolled analysis errors [3,18,21,47-51]. Therefore, sample-nondestructive technique like EDXRF is good alternatives for multielemental determination in infant formula and milk products.

In recent review of chemical element analytical techniques and related current official methods from various international organizations including AOAC INTERNATIONAL, the International Organization for Standardization (ISO), the International Dairy Federation (IDF), and the European Committee for Standardization (CEN) it was shown that the list of controlled essential chemical elements in infant formulas and milk products includes Ca, Mg, Na, K, P, Cl, Fe, Zn, Cu, Cr, Mo, Se, and I [46].

Element	Published data [Reference]			This work result M ± SD
	Median of means (n^a)	Minimum of means M or M ± SD, (n^b)	Maximum of means M or M ± SD, (n^b)	
Br	-	-	-	0.56 ± 0.06
Ca	548 (9)	12.5 ± 0.3 (3) [39]	4019 ± 286 (32) [5]	3100 ± 29
Cl	61 (1)	61 (-) [14]	61 (-) [14]	319 ± 3
Cu	0.33 (8)	<0.00014 (-) [40]	0.75 ± 0.05 (10) [22]	1.48 ± 0.04
Fe	9.3 (8)	0.63 (-) [40]	22.3 (23) [6]	10.8 ± 1.1
K	674 (4)	347 ± 1 (3) [39]	1538 (20) [41]	609 ± 5
Mg	65 (8)	6.05 ± 0.14 (3) [39]	245 (23) [6]	83.5 ± 8.8
P	600 (3)	203 ± 1 (5) [42]	893 (20) [41]	5518 ± 52
S	-	-	-	203 ± 8
Se	0.0122 (4)	0.0057 ± 0.0001 (2) [43]	0.035 (-) [17]	0.036 ± 0.014
Zn	5.11 (12)	0.26 (-) [40]	22.3 (23) [6]	4.32 ± 0.15

Table 4: Median, minimum and maximum value of means of the Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn concentrations (mg/L) in formula milk for infants according to data from the literature in comparison with our results *M:* arithmetic mean, *SD:* Standard deviation; "–"no data; [a]Number of all references, [b]Number of samples.

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The detection limits reached by current EDXRF technologies used in new spectrometer EDX-7000 (Shimadzu) allow determine the concentrations of key chemical elements of this list such as Ca, Mg, K, P, Cl, Fe, Zn, Cu, and Se (Table 5). In addition, this spectrometer allows determine in infant formulas and milk products the concentrations of two very essential chemical elements - Br and S.

The precision (repeatability) of the EDXRF method developed in this work was investigated by analysis of ten ERM®-BD151 sub-samples in two runs with different assistants/operators. The precision of results for the infant first milk and the toddler milk formulas were estimated using three sub-samples for the Ca, Cl, K, P, and S concentration measurement and six sub-samples for the Br, Cu, Fe, Mg, Se, and Zn concentration measurement. Obtained results and their comparison with repeatability of routinely used methods (mainly AAS, ICP-OES/ICP-AES and ICP-MS) are given in table 6. For most of the elements, repeatability, characterized by the relative standard deviations (RSD), was found to be lower than 5% (Ca, Cl, Cu, K, P, and S), close to 5% (Zn), or nearly 10% (Br, Fe and Mg). The RSD value exceeded 10% only for Se. These results of RSD are very similar with medians of RSD values reported in the studies of chemical element concentration of the infant formulas using routine alternative methods (Table 6).

Element	This work result (n=10)						Certificate	Difference
	M_m	SD	SEM	Min	Max	DL	M_c ± SD	Δ, %
Ca	13528	347	110	13204	14195	11	13900 ± 700	- 2.7
Cl	7596	144	46	7435	7835	14	9800 ± 1200	- 22.5
Cu	4.96	0.24	0.05	4.8	5.4	0.6	5.00 ± 0.23	0.8
Fe	53.4	5.0	1.22	46.2	67.8	0.6	53.0 ± 4.0	0.8
K	15209	265	83	14897	15724	13	17000 ± 800	- 10.5
Mg	1630	152	54	1361	1812	39	1260 ± 70	29.4
P	10982	352	111	10577	11639	55	11000 ± 600	- 0.2
Zn	45.7	2.5	0.55	42.0	49.2	0.6	44.9 ± 2.3	1.8

Table 5: EDXRF data of chemical element mass fractions (mg/kg, dry mass basis) in the European reference material ERM®-BD151 (skimmed milk powder) compared to certified values
n: number of sub-samples; M_m: arithmetic mean of measured data; SD: Standard deviation of mean; SEM: Standard error of mean; Min: Minimum; Max: Maximum; DL: Detection limit; M_c: Arithmetic mean of certificate; $\Delta=[(M_m - M_c)/M_c] \times 100\%$.

Element	Published data [5,7,14,21,22,25-27,31,33-35,41-43]			This work result		
	Median (n)	Min	Max	ERM®-BD151	First milk	Toddler milk
Br	9 (1)	9	9	-	10.7	3.9
Ca	5 (11)	1	7	2.6	0.9	2.3
Cl	9 (3)	2	10	1.9	0.9	1.0
Cu	7 (6)	3	16	4.8	2.7	3.0
Fe	10 (10)	1	45	9.4	10.2	0.8
K	4 (8)	1	7	1.7	0.7	1.7
Mg	5 (6)	1	8	9.3	10.5	6.6
P	6 (4)	1	9	3.2	0.9	1.5
S	7 (2)	4	10	-	4.0	3.1
Se	7 (5)	1	44	-	38.8	-
Zn	6 (10)	2	10	5.5	3.5	2.2

Table 6: Median, minimum and maximum value of repeatability (RSD, %) for the Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn contents in infant formula milks according to data from the literature in comparison with our results for infant formula milks and reference material ERM®-BD151
RSD: Relative standard deviation; Min: minimum; Max: maximum; n: number of all references

Conclusion

An EDXRF method for the rapid determination of key chemical element (Br, Ca, Cl, Cu, Fe, K, Mg, P, S, Se, and Zn) concentrations in milk-based products has been developed and validated. This work demonstrates the feasibility of the developed EDXRF method as a nondestructive, fast, safe, less cost technique for qualitative evaluation of chemical elements in infant formulas and other milk products. Undoubtedly the EDXRF technique will be gaining popularity because it is the fast, uncomplicated, inexpensive method, and does not require highly skilled staff to carry out a measurement. Moreover, the EDXRF technique is very prone for standardization of infant formulas and milk products quality control because it progressively integrates the green inorganic analysis trends such as sustainability, simplicity, miniaturization, and automation as well as reduced energy consumption and excludes use solvents and toxic reagents.

The infant first milk formulas investigated in this study had higher levels of Cu, Fe, Mg, Se and particularly high Ca and P concentration, which were nearly 6 and 20 times higher than the manufacturer's label declaration, respectively. The analysis results for the toddler milk were generally close to the values declared by the manufacturers except for the Cu and Mg concentration, which were nearly 3 and 2 times higher.

Further studies are needed in UK, in the line started here, to control the composition of infant formulas and other milk products for children as the source of chemical elements needed for good health and growth of infants and toddlers. The developed EDXRF method is a suitable tool for this purpose.

Conflict of Interest Statement

The authors declare that no conflict of interest exists.

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