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Elemental Analysis of Formula Milk and Full-Cream Powder Milk in Bangladesh

R. Khatun[1,*], S. Akter[1], A. N. Monika[2], M. M. Rahman[3], M. M. Ahasan[2]

[1]Medical Physics Division, Atomic Energy Centre, Dhaka, Bangladesh
[2]Institute of Nuclear Medical Physics, AERE, Savar, Dhaka, Bangladesh
[3]Accelerator Facilities Division, Atomic Energy Centre, Dhaka, Bangladesh

Abstract An accelerator- based elemental study of powder milk, i.e. formula milk and full-cream powder milk was performed using proton induced X-ray emission (PIXE) technique. Concentration levels of various trace elements in twelve (12) different brands of milk samples including formula milk and full-cream powder milk that were commonly consumed in Bangladesh, were determined to reveal what types of nutrients might be passed from powder milk to babies. The experiments have been done with the 3 MV horizontal type (KN 3000) positive ion Van de Graaff accelerator at the Atomic Energy Centre, Dhaka (AECD). Full-cream powder milk samples were characterized by higher concentrations of S, Cl, K, Ca and Br compare to that in formula milk samples by overlapping concentration ranges of their elements. The concentration of elements examined in this work falls within or nearer the values reported in the literature except for Fe, Zn and Br as indicated earlier. Special interest of this research was to search for certain toxic elements which include Cd, Hg, Pb etc. because of their negative roles in enhancing infant mortality rates, but none of them was detected in both the cases. The Standard Reference Material 1633b (Coal Fly Ash) and IAEA Milk Powder (A-11) were also examined, and the measured values of trace elements were compared with the given certified values and found to be compatible.

Keywords Formula milk, Full-cream Powder milk, Trace elements, PIXE

1. Introduction

The dietary habits depend on so many factors like socio-economic status, regional customs, traditions, religion and so on. Milk is the first food for human being that serves as the main source of essential nutrients required for the biological functions and growth during early stages of life [1]. Trace elements play the important roles in the functioning of many proteins, hormones, enzymes, and other large molecules. However, some essential metals become *"toxic"* when their concentration is increased, especially the levels exceeding by 40-to-200-fold [2]. If their intakes via food chain cross the permissible levels [3], toxicity may be a problem. The severity of toxicity depends on nature and concentration of the elements, body resistance, and antagonistic effects of other chemical contaminants [4].

Milk and milk products are important components of diet for human beings, specially for growing children and is an important dietary source of minerals also. The concentration level of trace elements in both kinds of powder milk may vary with the local geochemical conditions, food chain, etc. All the efforts should be made so that children can take a well balanced nutritious food. United Nations Sub-committee on Nutrition demands that it is impossible to meet 100% recommended dietary allowances (RDA) of micronutrients from dietary sources alone [5]. Hence, nutritional supplements are mandatory to improve physical growth and mental development and to prevent occurrence of common day-to-day infections. Powder milk, i.e. formula milk and full-cream powder milk may play the important role as a supplement to meet the requirements. Elemental analysis of the powder milk is important for many reasons. The assessment of essential and heavy metal toxicity will provide sufficient knowledge to understand the nutritional status of milk powder and their effects on baby health. In the developed countries, food quality analysis is a routine work and is done to ensure the health and quality of their daily life [6].

The development of modern technology and increasing industrial activities may introduce the pollution of food, water, air, soil and so on. Hence, it is imperative to know whether the milks commonly used as substitutes or supplements of human milk for babies, have the adequate amount of essential trace elements and also the concentration of elements with harmful effects is below safety limits. Trace elements in milk are evidently of prime interest from the nutritional point of view, and also from the view-point of their effect on milk processing and dairy technology. It has

* Corresponding author:
rajadakhatunhasi@gmail.com (R. Khatun)
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long been known that trace elements in powder milk may be significantly altered through handling of the milk or through various technological processes. Additionally, the relevant companies have embarked on advertisement campaigns concerning the nutritional quality of their products. In many cases, the manufacturers of these instant milk powders advocate their products as having been fortified with several mineral elements. However, the information of individual mineral element is often not indicated on the product labels of this milk powder [7,8].

Commercial dried milk and infant formula are deliberately fortified with essential elements such as iron, zinc, and copper to ensure proper nutrients provision [9]. The assessment of heavy metal exposure in the infants' diets is very essential so as to have a clear picture of the intakes, utilization, and retention of essential as well as toxic nutrients. Infants and young children may absorb 50% of dietary toxic metals as compared with only 10% by adults [10]. Heavy metals are dangerous because they tend to bioaccumulate in a biological organism faster than being metabolized or excreted [11].

As far as infant foods are concerned, the main issue is whether formula products consumed by babies, contain sufficient amount of essential nutrients. Such formulations should contain all the essential trace elements at least in those levels that are present in human milk because milk formulas are used as substitute of human milk [12]. Hence the elemental analysis of formula milk and full-cream powder milk is necessary for adjudging its suitability and to obtain information on the quantities of trace elements present in powder milk, i.e. formula milk and full-cream powder milk in adequate level or not. The aim of the present study was to analyze the infant formula milk and in full cream powder milk samples collected from different local markets of Bangladesh and to determine the levels of essential and toxic trace elements present in them to obtain the nutritional judgment about them.

2. Methodology

2.1. Sample Collection and Preparation

The commercially available powder milk samples of twelve different brands were collected from different supermarkets in Dhaka city. Among them, five are formula milk and the other seven are full-cream powder milk. The samples were stored in airtight plastic containers at ambient temperature until the completion of analysis. Before pelleting, the powder milk samples were freeze dried at about -85℃ temperature and 133×10^{-3} mbar pressure to attain a constant mass. This milk powder was made into pellets of about 7mm dia with a hand press pellet maker and labeled and kept in desiccators. Freeze drying is a convenient technique, which reduces the background in the spectrum and improves the sensitivity [13]. At every stage, minimum sample handling was ensured to avoid contamination.

2.2. Method of Analysis

Proton induced X-ray emission (PIXE) technique was employed to analyze the powder milk samples to study which kind of nutrients are present in them. The main feature of PIXE method is to ionize an atom to produce characteristic X-ray and to detect them by the Si(Li) detector. The PIXE analysis provides high Z elements such as S, Cl, K, Ca, Fe, Cu, Zn, Ga, Br, Ru, Pb etc. and gives the accurate concentration of the most of the elements present in the samples with high accuracy [14].

MAESTRO software was used to save the X-ray emission spectrum and GUPIX with DAN32 interfacing software for analyzing PIXE spectra of our desired purpose [15]. The peak of the pulse height spectra gives information of an element and its concentration if it corresponds to the full - energy absorption peak of that element. The peak location is a measure of the photon energy and the peak area represents the number of photons. Measurements were done using internal beam technique and the characteristic X-rays emitted from the sample upon bombardment with 2.5 MeV proton beam were detected using a 30 mm^2 Si(Li) detector and associated electronic setup for routine analysis. The detector resolution was 165 eV at 5.9 keV. The count rate was reduced using a 0.147 μm thick Mylar absorber. The dead time of the measurements was generally less than 5%. A 200 $\mu g/cm^2$ gold diffuser foil was used to homogenize the beam onto the target samples and current was in the range of 5-10 nA.

3. Results and Discussions

Now-a-days many important advances in analytical techniques for trace element analyses in biological samples have occurred. These analyses are still commonly subject to significant errors due to mass interferences, high risk of contamination, and lack of suitable reference materials. This study investigated a wide variety of element concentrations of powder milk, using a highly sensitive analytical method which enables determination of very low concentrations of elements with high precision.

The Minimum Detection Limits (MDL) for this method usually depends on the matrix constituents of the sample. It is, therefore, necessary to keep attention of the MDLs to obtain quality results. Before doing any experiment, it is essential to validate the method accurately and is also important to demonstrate the quality of the data that are being used for decision making. It is necessary to present the data with the confidence that they really reflect the human health and thus environment [16-17] to convince the government or industries to take appropriate steps. However, sometimes it is difficult to find the standards of similar matrix description to the field of respective samples for quality assurance practice. In such cases, validation and good knowledge of the fundamentals of the techniques are not necessarily sufficient to obtain good results; careful attention to details must be needed on the part of the analyst to ensure the quality of results.

3.1. Method of Validation

The validity of the method has been checked by analysing two standard samples: Standard Reference Material 1633b (Coal Fly Ash) from National Institute of Standard and Technology (NIST) and IAEA Milk Powder (A-11) from IAEA. Measurements were taken several times and the obtained values are presented in Table 1.

Almost all values found agreed with the certified values, indicating that the instrument performed well under the given conditions at the time of sample analysis. The average results obtained for both the cases agreed within ± 5% of the certified values, which conforms the validity of our method. The wide variation of concentration of the elements may be due to the different sample matrix. In addition, the accuracy and precision of the method was sufficient for the experiment. Figure 1 represents the PIXE spectra for standard reference material.

Table 1. Elemental concentrations of the Standard Reference Material

Elements	Coal Fly Ash		IAEA Milk Powder (A-11)	
	Certified value (ppm)	Measured Value (ppm)	Certified value (ppm)	Measured Value (ppm)
S	2075 ± 11	2059 ± 45	--	--
K	19500 ± 300	19163 ± 432	17200±1000	16845±1224
Ca	15100 ± 600	14962 ± 500	12900±800	13156±1421
Cl	--	--	9080±1740	8935±1512
Fe	77800 ± 2300	77575 ± 1500	3.65±0.76	4.53±1.4
Cu	112.8	118±8.45	0.84±0.165	1.23±0.32
Zn	210	220 ±10	38.9±2.3	36.12±5.23
Br	140	145 ±5	14	13.67±3.4
Rb	--	--	30.8±6.3	32±7.5

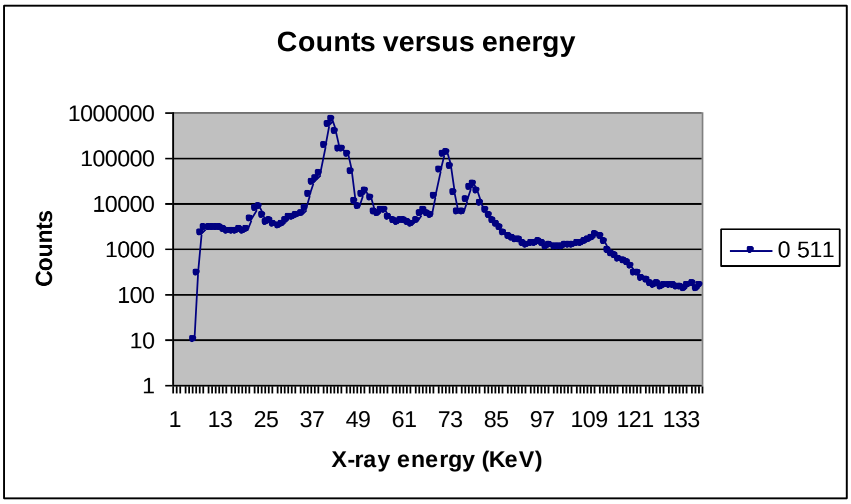

Figure 1. PIXE spectra for standard reference material

3.2. Experimental Details

Milk is a valuable source of essential minerals but some toxic elements may be accidently added during handling, processing, and remixing of milk. The information on food consumption by the children is also essential for assessing the food needs for them at national/regional level. No realistic policies on food production, procurement and distribution can be formulated without a thorough knowledge of food habits of the people. Healthy children do provide a solid foundation to the society in order to ensure optimal human resource development of a country [18]. Thus the powder milk and milk products may be used as supplements and can play an important role in human nutrition, i. e. child nutrition. Table 2 and Table 3 represent the obtained and manufacturer labeled concentrations of K, Ca, Fe, Cu, Zn and Cl in the milk samples. Table 4 shows the concentration ranges of elements in full-cream powdered milk and in formula milk with some literature values.

The percent - wise distributions of full-cream powder milk constituents (Figure 1A) are: Ca 28%, K 34%, Cl 28%, S 10%; Cu, Zn, Br, Rb and Fe contribute 0%. The distributions shown in this way (Figure 1B) for the formula milk are: Ca 29%, K 37%, Cl 24%, S 10%; and for other elements like Cu, Zn, Br, Rb and Fe this is zero percent. The comparison of average elemental concentrations of the milk samples are shown in Figure 2 through bar diagram. This diagram shows that K is maximum for full-cream powder milk and Ca is maximum for formula milk samples.

Table 2. Experimental and labeled concentration of K, Ca, and Fe

Sample ID	K (ppm)		Ca (ppm)		Fe (ppm)	
	Expt.	Labeled	Expt.	Labeled	Expt.	Labeled
PM-1	12133	12500	9423	9500	25.5	--
PM-2	13640	--	11674	--	33.3	--
PM-3	13603	--	12496	--	39.3	--
PM-4	9758	--	8159	9500	18.8	20
PM-5	11175	11000	8580	8600	57.3	100
PM-6	13555	--	9256	9300	57.8	--
PM-7	11177	--	9280	9300	24.9	--
PM-8	8957	10800	7630	7800	56.3	70
PM-9	10816	9843	7267	8500	73.5	75
PM-10	6320	6450	5491	5500	62.4	65.2
PM-11	6290	6350	5450	5550	65.3	73
PM-12	9300	9200	8100	7800	55.5	60

Table 3. Experimental and labeled concentration of Cl, Cu and Zn

Sample ID	Cl (ppm)		Cu (ppm)		Zn (ppm)	
	Expt.	Labeled	Expt.	Labeled	Expt.	Labeled
PM-1	9743	--	32.8	34	4.2	--
PM-2	8682	--	42.1	--	3.9	--
PM-3	9846	--	48.4	--	2.2	--
PM-4	10456	--	56.2	--	3.3	--
PM-5	11064	--	39.8	45	5.7	--
PM-6	9945	--	46	--	2.2	--
PM-7	8909	--	27.2	--	10.6	--
PM-8	6201	6300	8.3	10	6.4	7.5
PM-9	7463	7500	28.2	30	3.4	--
PM-10	4244	3700	45.2	52.6	3.2	3.3
PM-11	4010	4200	32.7	47	4.5	4.7
PM-12	6345	6400	26.8	30	6.9	7.5

Table 4. Concentration ranges of the powder milk samples (in ppm) with some reference study

Element	Full-cream milk		Formula milk		Literature values (Ref)
	Mean Con.	Range	Mean Con.	Range	
S	3598	3085-4465	2350	1934-2826	2100-3600 [21]
Cl	9817	8682-11064	5653	4010-7463	7900-10900 [21]
K	12151	9758-13640	8337	6290-10816	9700-13000 [21]
Ca	9907	8159-12496	6788	5450-8100	8400-9800 [21]
Fe	38.57	18.8-57.8	62.6	55.5-73.5	1.2-5.3 [8]
Cu	4.65	2.2-10.6	4.88	3.2-6.9	0.15-4.5 [9]
Zn	43.28	27.2-56.2	28.24	8.3-45.2	28.0-39.6 [8]
Br	17.67	15.7-28.6	7.92	5.3-12.7	0.1-1.5 [22]
Rb	5.3	13.2-19.3	7.64	10.3-12.9	8.9-22.2 [8]

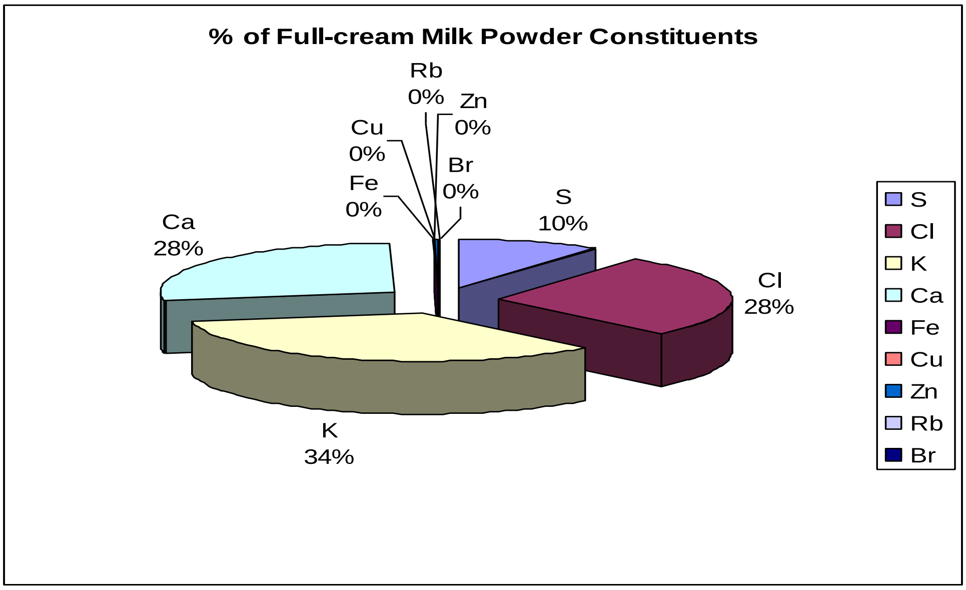

Figure 1A. Elemental concentration of full-cream Powder Milk

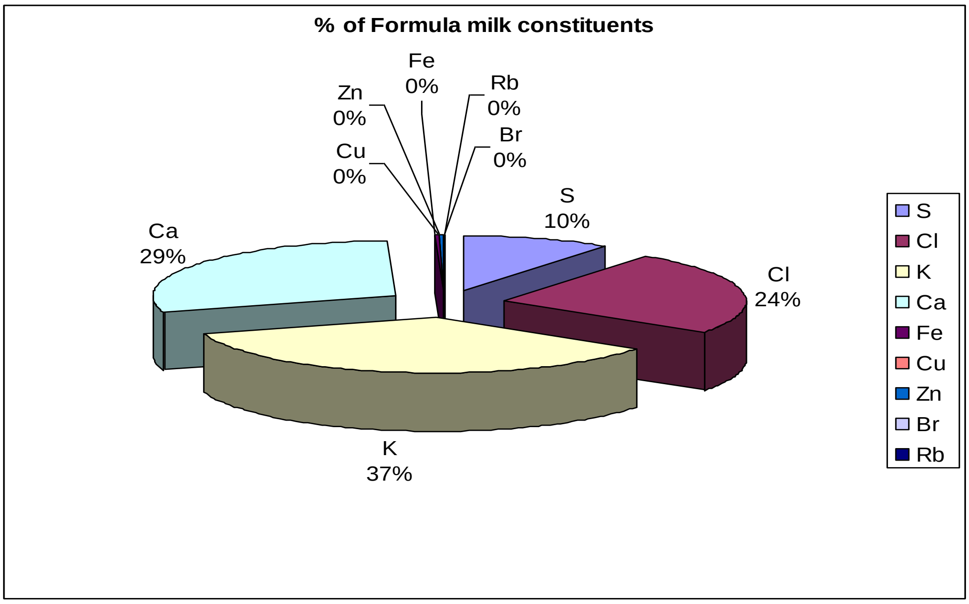

Figure 1B. Elemental concentration of formula milk

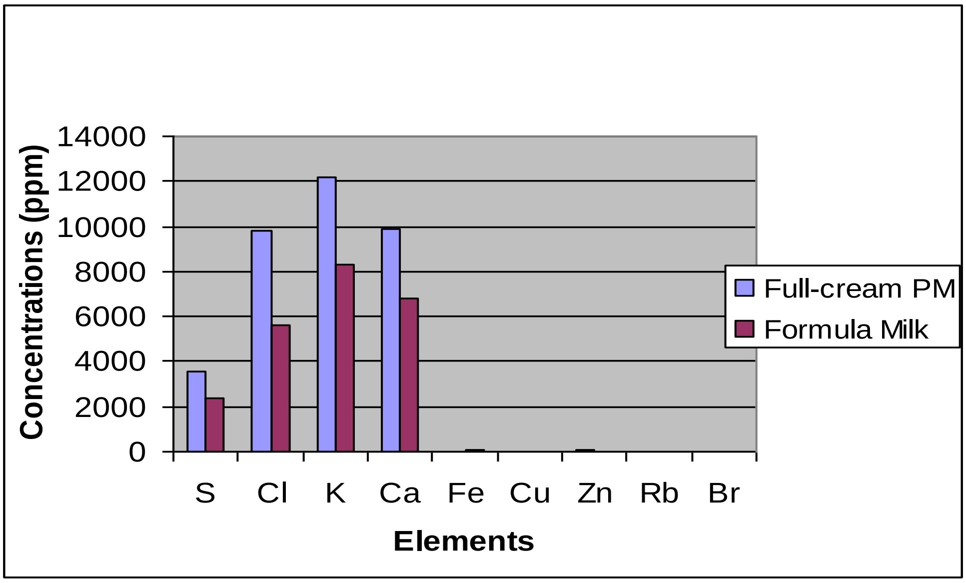

Figure 2. Comparison of powder milk constituents

Trace elements are required for the human body, but when our results were compared with the acceptable values for infants, some of them exceeded the adequate intake values. Most of the elements found in this study meet their labeled concentrations given by the manufacturer (Table 2 and Table 3). But some manufacturers did not specify the mineral compositions at the label, specially for full-cream powder milk and this is little disturbing for customer to verify the nutritional content. As a result, infants are likely to be exposed to higher metal levels through infant formula than through breast milk [19]. In this study the elements S, Cl, K, Ca, Fe, Cu, Zn, Br and Rb were found with some variation. Among all the obtained constituents, the concentration level of Potassium (K) is the maximum and the concentration level of Copper (Cu) is the minimum for both the cases (Table 4). The concentration limits of full-cream powdered milk samples observed in this study were greater than the formula milk constituents, trend that was confirmed by earlier literature values [15], except for Fe and Cu. The exception of Fe and Cu may be due to the fortification, because these two elements are very essential to infants for various metabolic activities. Considering that infants who are not breast-fed are specially dependent on formula diets, and that infants are potentially more susceptible, toxic metal contamination and essential metal limits should be regularly monitored during manufacturing [20].

4. Conclusions

The results of this study provide valuable information of various elements present in different powdered milk (formula and full cream) samples available in the market. It was observed that the determined values were within the acceptable limits. The present study demonstrates the levels of essential elements S, Cl, K, Ca, Fe, Cu, Zn, Br and Rb were found in the analyzed samples with a large variation interval. Full-cream powder milk samples are characterized by higher concentrations of S, Cl, K, Ca and Br, when compared to that in formula milk samples by overlapping concentration ranges of their elements. The concentration of elements examined in this work falls within or nearer the values reported in the literature except for Fe, Zn and Br as indicated earlier. The level of K is the maximum and Cu is the minimum for all the cases of milk samples. The Fe level concentration in full-cream powdered milk is found lower than in formula milk samples and this may be due to the fortification of formula milk. The milk samples contained considerable amounts of calcium, while potassium levels were well above the required limits. Copper levels were slightly lower than the permissible allowances. The concentrations of zinc in the milk samples were up to the mark for both the cases. Infant milk formulae had better iron levels as compared to other milk samples. Moreover, since infants are potentially more susceptible to metals, infant foods should be monitored regularly and checked for contamination by toxic metals as well as for levels of essential trace elements. Hence, the outcome of this research work will provide sufficient knowledge to understand the nutritional status of milk powder and their effects on human health.

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