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Analysis of Infant Foods using Energy Dispersive X-Ray Fluorescence Spectrometry

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The present work aims at the determination of detection limits of the various infant foods from Turkey using energy dispersive X-ray fluorescence spectrometer (EDXRF). The X-ray spectra were collected using a Si(Li) detector coupled with Canberra DSA-1000 desktop spectrum analyzer and a ^{55}Fe annular radioactive source. The relative errors for the detection limit measurements of ^{55}Fe radioactive source were found within 2-6 %. From the results, it can be concluded that the infant foods are rich in elements such as chlorine, potassium and calcium. Finally, the EDXRF technique was found to be sensitive and readily applicable for the determination of detection limit in various infant foods.

Key Words: Energy dispersive X-ray fluorescence, Infant food, Elemental concentration, Detection limit.

INTRODUCTION

The first year of life is the most critical for a child, particularly from a nutritional standpoint. Throughout the first year of a baby's life, amazing changes take place. During this period an infant experiences the most rapid growth and development period in its lifetime. Aside from these obvious external physical changes, important growth also takes place in an infant's vital organs. In fact, the brain, heart and kidneys double in size by a baby's first birthday. By 18 months of age, most of the brain cells have been formed. Therefore, appropriate amounts of essential nutrients such as protein, carbohydrate, fat, vitamins and minerals are necessary to ensure and sustain this rapid yet normal rate of growth and development. All professional and international health organizations are in agreement that human milk is the best nutriment for baby. But some of the babies can not feed with mother milk because of different reasons such as mother's health (the mother is infected with HIV or tuberculosis), the baby is unable to breastfeed (the child has a birth defect or inborn error of metabolism), family pressures (family members encourage use of infant formula), under-education, financial pressure, societal structure and dietary concerns. Therefore scientists and physicians began scientific investigations of breast milk substitutes. The best alternative feeding or supplement to breastfeeding is commercially prepared, iron-fortified infant formula. The aim of the commercial infant food manufacturers is to prepare an infant formula with a composition similar to that of mother milk. Like breast milk, formula also provides the proper nutrients at appropriate levels necessary for a baby to sustain a rapid rate of growth and development and will not stress the infant's delicate and developing organ systems. Various analytical methods have been reported in the literature for the determination of element concentrations in infant food materials. The trace elements in different types of baby foods consumed in Turkey were analyzed by flame and graphite furnace atomic absorption spectrometry[1]. A simple method for determination of analytes in milk-based products was developed[2]. In addition, EDXRF method was used for determination of iron in infant cereals[3]. The selenium content in human milk and Spanish infant formula were also determined[4]. Selenium in infant milk was analyzed by inductively coupled plasma spectrometry[5]. A method was used to determine iodine in human milk and infant formulae[6]. Besides, phosphorus in honey, milk and infant formulas was analyzed by the electro thermal atomic absorption spectrometry[7]. Iron, copper and zinc determination in 35 infant formula samples were performed by atomic absorption spectrometry[8]. Furthermore, detection limit and the uncertainty estimation in analytical X-ray spectrometry (XRS) results were described[9]. The available analytical methods used for elemental analysis except for X-ray fluorescence analysis technique generally use samples need to be dissolved with a digestion step. This sample preparation procedure can be tedious and time consuming. Also, some inconveniences that occur due to the incomplete element extraction and incomplete solubility may lead to some systematic errors. Such inconveniences can be overcome by

determination of analyte concentrations through direct methods, in which the samples are analyzed in their solid state, without digestion step[10]. In this work, we presented a procedure for simple analysis of infant foods using EDXRF. The application of energy dispersive X-ray fluorescent analysis (EDXRF) for the determination of elements released from five different dental luting cements such as zinc polycarboxylate (Carbchem), zinc carboxylate (Adhesor Carbofine), glass ionomer (Meron), resin cement (Duo-cement kit) and carboxylate (Durelon) in artificial saliva is described[11].

EXPERIMENTAL

In this study, five infant foods considered as subjects have been analyzed by using EDXRF technique. A high resolution Si(Li) detector (full with half maximum = 160 eV at 5.9 keV) coupled to a Canberra DSA-1000 desktop spectrum analyzer was utilized to collect the X-ray spectra and 5.9 keV photons emitted from ^{55}Fe were used to excite the characteristic X-rays of elements present in the samples. The main advantage of radioisotope excitation over X-ray tube excitation is in the mono-energetic character of radioisotope-emitted X-rays, inexpensiveness and commercially availability. For the X-ray excitations, the spectral distribution relationship between scattered and background radiation intensities is more complex as a result of the bremsstrahlung continuum.

The samples were collected and kept dried in absence of light at room temperature according to manufacturers' recommendations. After drying, the samples were sieved to a mesh size of 200 μm. The amount of these samples was 0.1 g. A 2 ton hydraulic press was used to compress the sample powder into a thin pellet of 30 mm diameter. The advantage of making these pellets is that inter-element enhancement effects in the sample are minimized. The effects of the matrix composition on the measured analyte-line intensity are known as matrix-, inter-element-, self-absorption- and absorption-enhancement effects. Whatever absorption-enhancement effects a specified analyte-matrix system may be subject to, they are most severe at and above infinite thickness, decrease in severity as thickness decreases below infinite thickness and substantially disappear in thin samples[12].

To determine the contributions of the background and scattering from sample holder, measurements without sample were performed. As discussed in detail in the following section, to increase the accuracy of determined detection limit of the prepared samples, the counting time for each sample was chosen in accordance with the area under the resultant peak which was higher than 10^4 counts and the samples were irradiated for 8 h by the annular sources.

The detection limit is a measure of the minimum quantity that can be detected by an instrument or technique under the given experimental condition (sample geometry, sample matrix, analysis time, *etc.*). Detection limit has been defined in terms of standard counting error of the background intensity. If σ_P and σ_B are the counting errors of the individual peak and background respectively, then the standard counting error for net count can be expressed as:

$$\sigma = \sqrt{\sigma_P^2 + \sigma_B^2} \quad (1)$$

At the limit of detection one can assume $\sigma_P \approx \sigma_B$. Therefore:

$$\sigma = \sqrt{2}\sigma_B \quad (2)$$

For 95 % confidence level, the counting error would be

$$2\sigma = 3\sqrt{N_B} \quad (3)$$

By taking into account time T and slope m of fluorescence intensity *vs.* analyte concentration curve, one can write the expression for minimum detectable concentration for an analyte element as:

$$C_{DL} = \frac{3}{m}\sqrt{\frac{N_B}{T}} \qquad m = I_A / C_A$$

or

$$C_{DL} = \frac{3\sqrt{I_B}}{\left(\frac{I_A}{C_A}\right)} \quad (4)$$

where I_B = background intensity (N_B/T); I_B = net area intensity; C_A = mass concentration of analyte; N_B = background counts in a given time T; m = I_A/C_A *i.e.*, slope of analyte counts-concentration curve; C_{DL} = minimum detectable concentration of analyte. From eqn. 4, it is clear that, for a specifie counting time, the minimum detectable concentration of analyte or detection limit depends mainly on two parameters: the background intensity I_B and the net area intensity per unit analyte mass (I_A/C_A)[13].

RESULTS AND DISCUSSION

Values of detection limit are presented in Table-1. There are high concentration of chlorine (11.46 ± 0.4 ppm) and general potassium-enrichment (5.36 ± 0.19) in the sample 4. There is also calcium-enrichment (5.27 ± 0.32) in the sample 3. The corresponding spectrum obtained from sample 1 using ^{55}Fe annular radioactive sources has been presented in Fig. 1. The X-ray lines of two elements overlap in the spectra. In order to obtain the intensity of each X-ray line, spectra were unfolded by using the intensity ratios of the X-ray lines corresponding to pure elements excited in the same geometry. These pure element samples were prepared in a similar way as the infant food. As an example, KK_β line overlaps strongly with the $C_a K_\alpha$ line. In this case, $C_a K_\alpha$ was determined by subtracting the KK_β intensity as measured with a pure elemental sample of K. Since extra elements weren't visible in the spectrum by the annular ^{241}Am annular radioactive source this spectrum has not been given.

TABLE-1
DETECTION LIMIT VALUES (ppm) OF THE ELEMENTS OBTAINED FROM DIFFERENT INFANT FOODS

Samples	DL ± RE		
	Cl	K	Ca
S1	8.13 ± 0.16	4.75 ± 0.10	5.06 ± 0.10
S2	7.18 ± 0.29	4.48 ± 0.18	4.86 ± 0.19
S3	8.07 ± 0.48	3.05 ± 0.18	5.27 ± 0.32
S4	11.46 ± 0.40	5.36 ± 0.19	4.43 ± 0.16
S5	9.28 ± 0.42	4.04 ± 0.18	2.81 ± 0.13

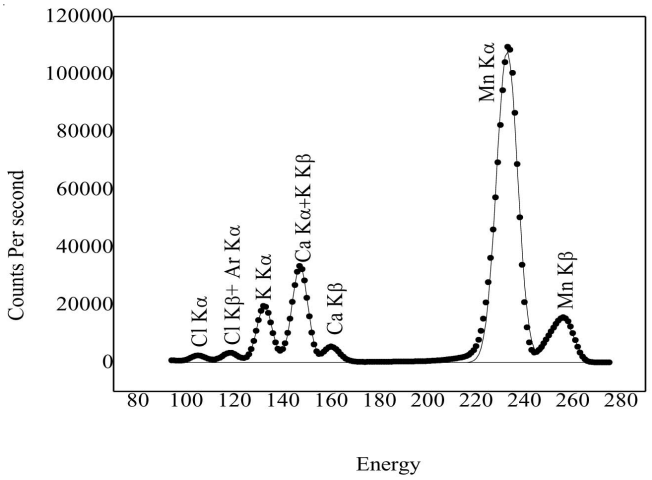

Fig .1. Typical X-ray spectra of the elements present in sample 1

Errors are unavoidable concomitant of every measurement. The errors arise from net area intensity, sample preparation, statistical error or geometry reproducibility. To calculate the relative error in detection limit measurement, ten successive measurements were made for each measurement time. The relative error contributed to detection limit measurement for ^{55}Fe radioactive annular source has been found to vary from 2 to 6 %.

Starting from birth, good nutrition helps build the foundation of a healthy life. Trace elements play an essential role in the biological and physiological possess of the human organism. Both deficiencies and excesses of these elements may result in a number of disorders in the human body.

Potassium is necessary for muscle contraction (especially cardiac fiber), transmission of the nerve impulses, synthesis of some proteins and as an enzymatic cofactor. Problems associated with low potassium levels include high blood pressure, congestive heart failure, cardiac, arrhythmias, palpitations, muscle weakness hyperthyroid, elevated blood sugar, mental apathy, depression, fatigue and general weakness, while severe potassium loss can cause death. Excessively high potassium levels result in acute or chronic cystitis (bladder infections) and right-sided ovarian cysts and testicular cancer.

Some of the studies claim that high chlorine levels increase the risk of bladder cancer and incidence of Hodgkin's disease, colorectal, esophageal and breast cancer. According to these claims, women with breast cancer have 50-60 % higher levels of organochlorines (chlorination by products) in their breast tissue compared to women without breast cancer. Chlorine has also been associated with declining sperm counts, male infertility.

Calcium is now the most promoted nutrient by proponents of conventional, nutritional and alternative medicine-yet at the same time, the assumed need is based purely on the speculation that the body's calcium intake is well below its requirements. Of the approximately 1 g of calcium in the average 70 kg adult body, almost 98% is found in bone, 1 % in teeth and the rest is found in blood, extra cellular fluids and within cells where it is a co-factor for a number of enzymes. Calcium promotes blood clotting by activating the protein fibrin and along with magnesium helps to regulate the heart beat, muscle tone, muscle contraction and nerve conduction. Chronic calcium deficiency is associated with some forms of hypertension, prostate and colorectal cancer, some types of kidney stones, miscarriage, birth (heart) defects in children when mother is periodontal diseases, sleep disturbances, mental health, depressive disorders and cardiovascular or hemorrhagic diseases. Elevated calcium levels are associated with arthritic and vascular degeneration, calcification of soft tissue, hypertension and stroke, gastrointestinal disturbances, mood and depressive disorders, chronic fatigue, increased alkalinity and general mineral imbalances. High calcium levels interfere with vitamin D and subsequently inhibit the vitamin's cancer-protective effect unless extra amounts of vitamin D are supplemented[14].

In conclusion, EDXRF method is found to be very well suited for the direct analysis of infant foods samples. This analysis can be helpful for quality control of these products.

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