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ARAŞTIRMA MAKALESİ /**RESEARCH ARTICLE**

A SURVEY STUDY FOR THE METAL ANALYSIS BY ICP-OES IN BABY FOODS

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ABSTRACT

This study describes the survey results of baby foods regarding metal analysis in twenty six samples. Metal content or contaminants (Fe, Cu, Zn, Pb, Hg and As) were examined by measuring inductively coupled plasma-optical emission spectrometry (ICP-OES). Prior to analysis, the digestion procedure was applied to the samples and their results were evaluated by considering the official regulations. It is remarked that some metal levels are lower than the daily intake doses. Besides, some metal amounts exceed the limits suggested by European Union Commission Regulation (EC, 466/2001) such as maximum level for Pb (0.02 mg/kg baby food).

Keywords: Baby food, ICP-OES analysis, Metal analysis.

ICP-OES İLE BEBEK MAMALARINDA METAL ANALİZİ ÜZERİNE İNCELEME

ÖZ

Bu çalışma 26 bebek maması örneğindeki metal analizi araştırma sonuçlarını göstermektedir. Metal içerikleri ya da kirleticileri (Fe, Cu, Zn, Pb, Hg ve As) indüktif eşleşmiş plazma-optik emisyon spektrometresi (ICP-OES) ile ölçülerek incelenmiştir. Örneklere analiz öncesi parçalama işlemi uygulanmış ve sonuçlar resmi düzenlemelere göre değerlendirilmiştir. Bazı metal düzeylerinin günlük alınması gereken dozlardan daha düşük olduğu görülmüştür. Ayrıca, Pb gibi bazı metal miktarları Avrupa Birliği Komisyonu (EC, 466/2001) tarafından önerilen limitleri (0.02 mg/kg bebek maması) aşmıştır.

Anahtar Kelimeler: Bebek maması, ICP-OES analizi, Metal analizi.

1. INTRODUCTION

Baby foods have special functions to play in diets of infants because they are major source of nutrients (Ikem et al., 2002; Bermejo et al., 2000; FDA, 1997) and a unique source of food during the first months of their life (Rodriguez et al., 2000).

Some elements such as iron and zinc are essential elements in infant formulas because of their biological function in the human body. Recommended daily intake for these essential trace elements are 18 mg for Fe and 15 mg for Zn. Despite the benefits of infant formulas as a major source of food for infants, the presence of contaminants, such as heavy metals, pesticides and others in infants' formula may carry health risks for children. These pollutants may arise from raw materials used in production, poor quality production processes, adulteration of infant foods and bad practices by mothers as

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regards infant formulation preparation and handling. In this case, the presence of toxic elements such as Hg, As and Pb in baby food may create significant health problems for infants (Doner and Ege, 2004; FDA, 2002).

Babies (0-6 months old) may be met more risk because they feed only baby food (Ikem et al., 2002). So, the regulations have very big importance (Turkish Standard, 1996).

The aim of this study is to examine metal contents and contaminants (Fe, Cu, Zn, Pb, Hg and As) by inductively coupled plasma-optical emission spectrometry (ICP-OES). The detected metals were inspected regarding to official regulations.

2. EXPERIMENTAL

2.1. Apparatus

A Perkin-Elmer DV 4300 inductively coupled plasma-optical emission spectrophotometer (ICP-OES) (Shelton, USA) was used to analyze the elements in digested samples. Measurements were performed at 238.204 nm, 327.393 nm, 206.200 nm, 220.353 nm, 253.652 nm, and 188.979 nm for Fe, Cu, Zn, Pb, Hg and As, successively. FIAS Perkin Elmer Mercury/Hydride Chemifold was used for Hg and As determinations.

A model of Berghof-Speed Wave, MWS-3 microwave oven (Eningen, Germany) for digestion a Labofuge 200 model centrifuging (Heraeus Instruments GmbH, Hanau, Germany) was used for centrifugation, a Sonorex Ultrasonic Bath (Bandelin, Berlin, Germany) degassing all of the solutions after centrifugation. A Daigger model Vortex Genie 2 G 560-E (Scientific Industries Inc., Bohemia, USA) model vortex for dissolving and mixing baby food samples were used.

High quality pure water was obtained by using a Model of Water Pro PS Labconco Corp. (Kansan City, Missouri).

2.2. Chemicals

Hg (SRM 3133), As (SRM 3103a), Pb (SRM 3128), Fe (SRM 3126a), Zn (SRM 3168), Cu (SRM 3114) element standards were supplied by NIST (North Kingstown, RI, USA).

Baby food samples belonging to one brand were supplied in a local market and they were coded.

2.3. Procedures

All working standard solutions were daily prepared. Use of glassware was reduced to a minimum and plastic (polypropylene) vessels of the type commonly used for preparing and storing the solutions to decrease the risk of possible contaminations. All the glassware and plastic ware were nitric acid-washed and rinsed with ultra pure water.

2.3.1. Microwave-Oven Digestion and ICP-OES Analysis of Baby Foods

Prior to the analysis, each baby food was digested in an acid solution of (HNO_3, 65% (v/v) and HCl 37% (v/v)) using the microwave digestion system. Approximately 400 mg sample was weighed, transferred to the digestion vessel and 10.0 ml of acid mixture (HCl-HNO3, 3:1, v/v) was added. The mixture was carefully shaken, it was left at least 20 min, and the vessel was sealed and placed into the microwave oven. Then, it was digested under the optimized microwave digestion program. The procedures were duplicated for all samples. The digestion program is demonstrated in Table 1.

Table1. Optimum microwave digestion program

Step	1	2	3	4
T (^{0}C)	100	140	160	190
Ramp(min.)	10	5	5	10
Time (min.)	10	10	25	15

Amount of various metals in the digested samples were determined by using the ICP instrument, in the presented conditions which are in Table 2.

3. RESULTS AND DISCUSSION

3.1 ICP-OES Analysis

3.1.1 Some Element Levels in Baby Food

All baby foods were digested in an acid solution of (HNO_3, 65% (v/v) and HCl 37% (v/v)) using the microwave digestion system as explained in the experimental section. The microwave digestion procedure was free from contamination risk and has low time and chemical consumption. After digestion of the samples, the metals Fe, Cu, Hg, Zn, Pb and As were determined by ICP-OES and the results are shown in Table 3.

Turkish Standard, TS 11983, Baby Food I-Infant Formula of Turkish Standard requires the metals to be in the limits of Fe, Cu and Zn. But, it does not permit to be contaminated by Hg and As. European Communities Commission Regulation (No 466/2001) states maximum Pb level is 0.02 mg/kg infant formula (EC, 466/2001). The brief explanations for the metal limits are presented in Table 4.

In the evaluation of the results and considering, the Turkish standard mentioned above, Cu contents for three types of baby food (code no 1, 3, and 20) are higher than the permissible limits. Cu content, in some types of baby food are lower than the limits baby food (code no 2, 4, 5, 21, 24, 25, 26). Zn and Fe content of some samples in baby food (code no 14, 5, 10, 17, 19, 21, 22, 23, 25, 26) and (code no 7, 10, 13, 20, 23) are lower than the limits, respectively. Moreover, 162.35 µg Pb/100kcal (7.34 mg Pb/kg baby food) in the baby food (code no 12) and 12.70 µg Pb/100kcal (0.06 mg Pb/kg baby food) in the baby food (code no14) were observed. These values exceed European Union Commission Regulation (EC, 466/2001) maximum level for Pb (0.02 mg/kg baby food) (EC, 466/2001). As a good result, As was not detected in any sample, at all.

4. CONCLUSION

A survey study was realized by measuring certain metal contents in baby foods. Although some metals are needed for human body, especially heavy metals are very harmful for the organism. The subjected metals were analyzed by using ICP-OES that it is more precise and accurate technique. Some remarkable results were obtained for the analysis of baby food. Some values are in the permissible limits in the point of the view some metals (Cu, Zn and Fe). But, some of them exceed the limits.

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Table 2. Instrument operating conditions applied for metals determination by ICP-OES

Parameters	
View mode	Axial
View height	15 mm
Gas	Argon
Shear gas	air
Gas: plasma	16 l/min
Gas flow: auxiliary	0.2 l/min
Source equilibration time	15 s
Pump flow rate	1 ml/min
Detector	Segmented charged coupled (SCD)
RF power	1450 W
Nebulizer	0.55 l/min
Sample aspiration rate	1 ml/min
Read Peak	Height for hydride system/peak area for others
Number of replicates	3
Background correction	Manual point correction
Read delay	50 s

Table 3. Certain element contents (Fe, Cu, Zn, Pb, Hg and As) of baby food samples by ICP-OESa).

(Baby food)	mgFe/100kcal	µgCu/100kcal	mgZn/100kcal	µgPb/100kcal	µgHg/100kcal
1	0.9	107.65	0.4	–	–
2	0.9	–	0.5	–	–
3	0.8	87.58	0.5	–	–
4	0.5	–	0.3	–	–
5	0.6	–	0.3	–	–
6	2.3	28.34	0.6	–	–
7	0.3	44.76	0.5	–	–
8	0.5	15.74	0.4	–	–
9	1.2	70.12	0.5	–	–
10	0.4	62.86	0.3	–	–
11	0.5	15.69	0.4	–	–
12	1.1	60.88	0.6	162.35 [a)]	–
13	0.0014	23.75	0.6	–	–
14	0.8	18.45	0.5	12.70 [a)]	–
15	1.2	22.56	0.5	–	–
16	1.0	121.11	0.5	–	–
17	0.7	64.93	0.4	–	–
18	0.7	13.15	0.6	–	–
19	1.2	24.48	0.4	–	–
20	0.05	117.88	1.2	–	–
21	0.9	–	0.3	–	–
22	1.0	25.15	0.4	–	–
23	0.06	30.26	0.3	–	–
24	0.8	15.66	0.7	–	–
25	0.7	–	0.3	–	–
26	1.1	–	0.2	–	–

(–) is designate under the detection limit and their limits are 4.6 ppb, 9.7 ppb, 5.9 ppb, 42 ppb, 61 ppb and 100ppb for Fe, Cu, Zn, Pb and Hg, respectively.
a) 162.35 µg Pb/100kcal and 12.70 µg Pb/100kcal values are equivalent to 7.34 mg Pb/kg baby food and 0.06 mg Pb/kg baby food, respectively.

Table 4. Limits for some elements in baby foods[a)]

Nutrient	Limit/100 kcal	
	Minimum	Maximum
Fe[b)]	0.5 mg	15 mg
Fe[c)]	1 mg	2 mg
Cu	20 µg	80 µg
Zn	0.5 mg	indefinite
Pb[d)]	no definition	no definition
Hg	not permitted	not permitted
As	not permitted	not permitted

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