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Metal Contents in Some Brands of Biscuits Consumed in Southern Nigeria

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ABSTRACT

The concentrations of zinc, iron, chromium, calcium, magnesium, nickel, lead, copper, cobalt and cadmium were determined in six major classes of biscuits in Nigerian market after acid digestion by flame atomic absorption spectrophotometry. The mean concentrations of metals in various biscuit classes ranged from 29.06-49.27, 45.02-109.00, 0.39-0.72, 0.01-2.90, 205.64-395.27, 118.10-121.30, 2.15-4.88, <0.001-1.07, 0.53-5.04, nd-1.30, 0.03-0.05 $\mu g\ g^{-1}$ for Zn, Fe, Cr, Mn, Ca, Mg, Ni, Pb, Cu. Co and Cd, respectively. The estimated daily intake in $\mu g\ kg^{-1}$ body weight is below the prescribed Tolerable Daily Intake (TDI) values for each metal except for nickel.

Key words: Essential elements, toxic element, tolerable daily intake, biscuit

INTRODUCTION

The study of metal composition of foods is of great significance because some of these metals are essential or toxic (Onianwa *et al*., 1999; Gopalani *et al*., 2007; Iwegbue, 2011). The essential metals (e.g., Mn, Cu, Zn, Co, etc) own their essentiality being constituents of enzymes and other important proteins involved in key metabolic pathways, hence, deficient supply results into metabolic dysfunction causing diseases (Iwegbue, 2011). The toxic metals (e.g., Hg, Cd and Pb) have no known biological function and exhibit toxicological problems even at trace concentrations. Biscuits are the most favourite food items of children and one often presented to them as token of love and affection from their parents and relatives. Consumption of biscuit is not limited to children alone; adults do consume biscuits as well.

The common basic ingredients of biscuits are flour, fats, sugar, aerating, chemicals and milk or water. Most types are prepared from one of the following classes of dough, soft sweet (digestive), hard sweet (e.g., Rich tea) and laminated (e.g., cream crackers) (Pearson, 1976).

Ingestion of foods is an obvious means of exposure to metals, not only because many metals are natural components of food stuffs but also because of contamination during process (Ashraf, 2006). The contamination of food occurs at any point of the production chain to point of consumption. Obviously, concentrations of metals in food items at the point of consumption are necessary for estimation of actual amount of metals exposure to humans from ingestion of these food items. In developing countries like Nigeria, there is paucity of data on metal composition of foods and extent of the exposure of humans to chemical hazards in foods is still patchy. For many developing countries, data on contamination of foods and exposure through foods are not collected or may be incomplete or collected in a way that make it difficult for inter-country comparison. To assess the risk to human's health arising from the presence of metals in foods, the actual dietary intake of metal should be estimated and compared with the corresponding toxicological reference intake, the Provisional Tolerable Weekly Intake (PTWI). Estimation of actual dietary intake of metals is essential for risk assessment and can be used to determine whether there may be a relationship between the observed adverse effects in human and exposure to particular contaminant (Anonymous, 2009). In view of this fact, the present study presents an account of concentrations of Ca, Mg, Cd, Cu, Co, Cr, Ni, Fe and Zn in some brands of biscuits in Nigeria.

MATERIALS AND METHODS

Samples of biscuits were collected from various brands within the six major classes of biscuits namely (1) short cake (2) short bread (3) cookies (4) cream crackers (5) digestives and (6) cabin. A total of 10-18 different brands were collected within each major group. The samples collected covered both locally produced and imported biscuits. The choice of the samples was carefully made to reflect the various brands consumed by different income classes and influenced availability as of the time of the study.

All reagents used were of analytical grades. Working standards of Cd, Cu, Cr, Co, Fe Ni, Pb, Ca, Mg, Mn and Zn were prepared by diluting stock solution (Merck, Darmstadt, Germany) of 1000 mg L^{-1} in 0.25 mol L^{-1} HNO_3.

The samples of the biscuit were dried at 60°C for 1 h in the oven. The dried materials were powdered using a porcelain crucible. The digestion of samples for determination of metals (Ca, Mg, Ca Cu, Cr, Co, Ni, Mn, Fe and Zn) was done using a mixture of nitric acid and perchloric acids. Biscuit samples (2.00 g) placed in digestion tubes were pre-digested using 10 mL of concentrated HNO_3 at 120°C until the liquor was clear, next, 10 mL HNO_3 and 2 mL of $HClO_4$ were added and temperature was maintained at 135°C for 2 h until the liquor becomes colourless. The digested aliquots were filtered through a Whatman No. 1 filter paper and diluted to 25 mL with 0.25 mol L^{-1} HNO_3. In all metal determinations, analytical blanks were prepared in a similar manner omitting the sample.

The concentrations of metals in sample solutions were determined using flame atomic absorption spectrophotometry (GBC scientific equipment SENS AA, Melbourne, Australia). Blanks and calibration standards were also analyzed in a similar manner.

All glassware was soaked in a solution of 10% nitric acid for 48 h followed by thorough rinsing with deionized water. Instrument readings were corrected with blank readings. All samples were analysed in triplicate. The relative standard deviation between triplicate analyses is $< 5\%$. The analytical procedure was checked using spikes. The spike recoveries for the metals in this study were 96.6% for Cu, 89.9% for Cd, 95.7% for Ni, 103.7% for Fe, 98.4% for Zn, 91.3% for Pb, 97.2% for Mn, 92.8% for Cr, 89.7% for Co, 106.6% for Ca and 94.7% for Mn. The detection limit (μg g^{-1}) set at three times the standard deviation of the blanks, that are as, Cu (0.05), Cd (0.001), Ni (0.02), Fe (0.02), Zn (0.05), Pb (0.001), Mn (0.001), Cr (0.002) and Co (0.001). The estimated limits of quantification for the studied metals (μg g^{-1}) were Cu (0.05), Cd (0.005), Ni (0.10), Fe (0.10), Zn (0.3), Pb (0.05), Mn (0.10), Cr (0.005) and Co (0.05). Two-ways analysis of variance (ANOVA) and student's t-test were used to determine whether the concentrations of the metals varied significantly within and between brands, with values less than 0.05 ($p<0.05$) considered statistically significant. The statistical calculations were performed with SPSS 11.5 Version.

RESULTS AND DISCUSSION

The mean concentrations of metals in various types of biscuits are shown in Table 1. The values in parentheses refer to concentration ranges. The concentrations of calcium and magnesium in the samples were higher than any other elements studied. The highest mean value of calcium was

Table 1: Mean concentrations (μg g^{-1}) of metals in biscuits types in Nigerian market

Sample type	n	Zn	Fe	Cr	Mn	Ca	Mg	Ni	Pb	Cu	Co	Cd
Digestives	16	21.8±9.1	46.3±32.1	0.4±0.2	1.45±2.04	395.3±226.5	121.3±0.8	2.8±1.5	0.6±0.1	3.3±1.0	0.07±0.08	0.04±0.01
		(11.3-33.3)	(5.7-81.7)	(0.2-0.8)	(0.0-4.34)	(196.9-696.0)	(120.7-122.0)	(0.9-4.6)	<0.001-1.9	(2.1-4.6)	(001-0.18)	(0.03-0.05)
Cookies	10	42.7±32.5	33.9±9.2	0.7±0.5	0.01±0.00	258.0±176.3	120.8±17.6	4.9±1.3	<0.001	2.7±0.5	<0.001	0.03±0.02
		(14.2-73.3)	(26.3-47.2)	(0.2-1.2)	(0.01.0.01)	(104.5-503.6)	(116.4-122.0)	(3.9-6.5)		(2.4-3.5)		(0.02-0.05)
Cream crackers	18	36.5±25.8	109.0±120.4	0.6±0.3	2.90±2.25	346.7±106.9	120.7±11	3.7±1.6	0.4±0.6	2.6±2.1	0.68±0.79	0.05±0.02
		(11.0-77.6)	(3.50-364.2)	(0.3-1.2)	(0.01-5.23)	(234.6-503.6)	(118.4-122.0)	(1.2-6.1)	(0.01-0.9)	(0.01-4.8)	(<0.001-1.91)	(0.02-0.07)
Short cake	17	29.1±25.0	68.0±23.2	0.4±0.3	0.01±0.0	337.1±150.3	118.1±2.7	2.5±2.5	1.1±1.2	5.0±3.9	1.30±1.51	0.03±0.01
		(7.9-64.2)	(29.5-94.2)	(0.2-0.8)	(0.01-0.01)	(124.6-455.0)	(114.3-121.2)	(0.5-6.1)	(0.2-2.8)	(0.01-7.5)	(<0.001-2.84)	(0.02-0.07)
Short bread	16	49.3±13.51	83.8±21.9	0.64±0.36	0.01±0.00	363.6±168.0	119.1±4.0	2.2±1.2	0.4±0.5	0.5±0.7	<0.001	0.04±0.01
		(33.9-59.2)	(63.9-107.2)	(0.14-0.98)	(0.01.0.01)	(195.4-531.5)	(114.5-116.8)	(0.8-2.0)	(0.01-1.1)	(0.11-1.3)	<0.001	(0.03-0.05)
Cabin	10	34.9±17.1	45.0±32.3	0.6±0.4	1.03±1.77	205.6±201.5	119.0±3.8	3.01±0.37	0.54±0.92	1.43±2.22	0.10±0.16	0.03±0.01
		(15.2-44.9)	(1.4-9.3)	(0.1-1.0)	(0.01-0.98)	(87.3-438.3)	(115.4-128.4)	(2.623.34)	(0.01-1.60)	(<0.001-3.99)	(<0.001-029)	(0.02-0.04)

observed in the digestive brands (395.3 μg g^{-1}), while the lowest mean level was observed in the cabin brands (205.64 μg g^{-1}). Within each group, there is a significant difference ($p<0.05$) in levels of calcium. Similarly, the highest level of magnesium was observed in the digestive and with lowest levels in cabin and short cake brands. There is no significant difference ($p<0.05$) with the mean levels of magnesium in the various biscuit types are compared. Calcium is essential for bone formation in children. Sehecie and Dragojevic (2005) reported that calcium and magnesium contents in different kinds of biscuit in Critic ranged from 204.3 to 879.2 μg g^{-1} and from 172.9 to 595.3 μg g^{-1}, respectively. The concentrations of Ca and Mg found in the biscuit samples were lower than levels reported by Sehecie and Dragojevic (2005).

The concentrations of zinc varied from 21.8 μg g^{-1} in the digestive brand to 49.3 μg g^{-1} in the short cake. The permissible levels of zinc in grains and beans are 50 and 100 μg g^{-1}, respectively (USDA, 2003). The levels of zinc found in the various biscuit types were less than these limits. Saracoglu *et al.* (2004) reported zinc levels ranging between 3.1-16.1 μg g^{-1} in biscuits in Turkey. Gopalani *et al.* (2007) reported zinc concentrations ranging from not detected to 13.4 μg g^{-1} in biscuits from India. Similarly, Salama and Radwan (2005) reported zinc levels ranging between 2.347-4.749 μg g^{-1} in biscuits from Egyptian market. The concentrations of zinc found in the present study were higher than values reported by these researchers.

The mean concentrations of iron the biscuit samples ranged from 33.9 to 109.0 μg g^{-1}. The cream crackers have significant higher concentrations of iron compared to any other type studied. The lowest iron concentration was observed in the cookies. Gopalani *et al.* (2007) reported iron concentration ranging from not detected to 36.2 μg g^{-1}. The mean concentrations of iron in different biscuit types in this study were higher than upper limits of reported by Gopalani *et al.* (2007) except for the cookies. The concentrations of chromium in the different biscuit types ranged between 0.39 and 0.72 μg g^{-1}. The highest chromium level was observed in the cookies. Gopalani *et al.* (2007) reported chromium concentrations ranging from not detected to 0.55 μg g^{-1} in biscuit from India. Chromium is an essential trace element and the biologically usable form of chromium plays essential roles in glucose metabolism. It has been estimated that human requires nearly 1 μg day^{-1}. The sources of contamination by these metals are mainly from raw materials used, manufacturing processes, leaching of these metals from vessel in which they are stored. Processing of biscuit is done in containers from which nickel contamination is possible in addition to contamination of catalyst used in preparation of hydrogenated vegetable oil (Dahiya *et al.*, 2005). In addition to this, Ni in the biscuit arose from cocoa additives which is known to contain elevated concentrations of nickel (Milacic and Kralj, 2003). The concentrations of nickel in the various biscuit types ranged from 2.1-4.9 μg g^{-1}. The highest mean concentration nickel was observed in the cookies. The levels of nickel found in this study were comparable to 5.7-9.85 μg g^{-1} ranges reported by Gopalani *et al.* (2007).

The concentrations of manganese in the different biscuit type ranges from 0.01-2.9 μg g^{-1}. The highest level of manganese was observed in the cream crackers (2.9 μg g^{-1}). The concentrations of manganese in different brands of biscuits showed significant difference within and between the different biscuits types except for the cookies, short cake and cream crackers. Gopalani *et al.* (2007) reported manganese levels ranging between 2.053-8.24 μg g^{-1} in biscuits available in Nagpur city, India. The concentrations reported in this study are slightly lower than that of Gopalani *et al.* (2007).

The concentrations of lead in different biscuit types spanned between 0.39 and 2.92 μg g^{-1}. The highest concentration was found in the cream cracker. The permissible limit for lead in cereal and legumes is 0.2 μg g^{-1}(CAC, 2003). The concentrations of lead in these products exceeded the permissible limits for Pb in cereal products except for the cookies.

The concentrations of copper ranged between 0.5 μg g^{-1} in short breads to 5.0 μg g^{-1} in the short cake types. Gopalani *et al.* (2007) reported copper levels ranging from not detected to 4.699 μg g^{-1} in potato chips and biscuits available in Nagpur City, India. Similarly, Salama and Radwan (2005) recorded mean concentrations of copper in sweet and salted biscuits in Egypt as 0.787 and 1.386 μg g^{-1}, respectively. Saracoglu *et al.* (2004) reported mean concentrations of copper in 20 different kinds of hard biscuit produced in Turkey as <1.0-4.2 μg g^{-1}. The observed concentrations of copper in the present study are slightly lower than values reported by these researchers. The permissible limit for copper in all foods is 10 μg g^{-1} (CAC, 2003).

The average cobalt concentrations in the various biscuit types ranged between 0.01 to 1.30 μg g^{-1}. The highest mean concentration of cobalt was observed in the short cake types. The levels of cobalt observed in this study were similar to the range reported for potato chips and biscuits in Indian (Gopalani *et al.*, 2007).

Cadmium contents of the various biscuit types ranged from 0.03-0.05 μg g^{-1}. The highest mean level was found in the cream crackers type. The permissible level of cadmium in foods is 0.05 μg g^{-1} (FAO/WHO, 2001). The mean levels of cadmium in this study were below the permissible limits. However, some individual samples contained cadmium at levels above the permissible limit. Salama and Radwan (2005) reported cadmium contents ranging from 0.0 13-0.122 μg g^{-1} in biscuits from Egypt. Similarly, Karavoltsos *et al.* (2002) reported cadmium contents in biscuits from Greece as 0.0126-0.0143 μg g^{-1}. Gopalani *et al.* (2007) found cadmium at levels below the limit of detection in biscuits from India. The levels of cadmium observed in this study are comparable to levels for found in biscuits in some other countries in the world (Karavoltsos *et al.*, 2002; Salama and Radwan, 2005).

Table 2 presents the results of estimated dietary intakes of metals.

The estimated dietary intake based on consumption of 40 g of biscuit per day for a 15 kg child ranged from 584.3-1054.1 μg kg^{-1} b.wt. $day^{-1}$for calcium while 314.7-323.5 μg kg^{-1} b.wt. day^{-1} were recorded for magnesium. The recommended dietary allowance for calcium is set at 700 mg, 1000 mg and 1300 mg for 1-3 years, 4-8 years and 9-18 years, respectively. The upper tolerable intake of calcium is set at 2500 mg and 3000 mg for 1-8 years and 9-18 years, respectively (Institute of Medicine, 2010). The estimated maximum daily intake of Ca in this study constitutes approximately 0.007% of the recommended dietary allowance for Ca.

The estimated daily intake of zinc from consumption of biscuit ranged from 58.0 to 131.4 μg kg^{-1} b.wt. day^{-1} which constitutes about 5.8-13.1% of Joint Expert Committee of Food Additives Provisional Maximal Tolerable Intake of 1000 μg kg^{-1} b.wt. day^{-1} (WHO, 1982). The

Table 2: Estimated daily intake of metal based on consumption of 40 g of biscuit per day (μg/kg b.wt./day)

Sample type	Zn	Fe	Cr	Mn	Ca	Mg	Ni	Pb	Cu	Co	Cd
Digestives	58.00	123.4	1.0	3.90	1054.1	323.5	7.4	1.6	8.9	0.2	0.11
Cookies	113.80	90.5	1.9	0.03	688.0	322.9	13.0	0.0	7.3	0.0	0.08
Cream crackers	97.30	209.7	1.7	7.70	924.5	321.9	13.0	1.1	6.9	1.8	0.13
Short cake	78.70	181.3	1.1	0.03	898.9	314.7	6.6	2.9	13.4	3.5	0.08
Short bread	131.40	223.5	1.7	0.03	969.6	317.6	5.7	1.0	1.4	0.0	0.11
Cabin	93.00	120.1	1.7	2.80	548.3	317.1	8.0	1.4	3.8	0.3	0.08

intake values of intake of iron in this study ranged from 90.5-223.5 μg kg^{-1} b.wt. day^{-1}. The estimated dietary intake of iron in this study ranged from which is far below the recommended dietary allowance value of 10-19 mg day^{-1} person (WHO, 1993). The EVM guidance level for total dietary intake of trivalent chromium is 150 μg kg^{-1} bw. day^{-1} (EVM, 2003). The estimated daily intake of chromium spanned between 1.0 and 1.9 μg kg^{-1} bw. day^{-1} which constitutes about 0.69-1.28% of the EVM guidance level.

The Tolerable Daily Intake (TDI) of nickel is 5 μg kg^{-1} b.wt. day^{-1} (WHO, 1996). The estimated daily intake of nickel in the present study ranged from 5.8-13.0 μg kg^{-1} b.wt. day^{-1}. The estimated daily intake exceeded the tolerable daily intakes of nickel. Similar intake values have been reported for chocolates and candies in Nigeria (Iwegbue, 2011). There is need to exercise some caution in the consumption of these products. Higher daily intakes of Ni have been reported in literature for confectionery. For example, Dahiya *et al.* (2005) reported that the dietary contribution of nickel from chocolates in India as 8.68-55.26 μg day^{-1}. Higher dietary contribution of nickel ranging from 200 to 900 μg day^{-1} has been reported (Myron *et al.*, 1978; Nielson and Flyvholm, 1984; Larsen *et al.*, 2002). Krishnamurti and Pushpa (1991) reported nickel intake as high as 240-3900 μg kg^{-1} in Indian foods.

The National Research Council of Canada (NRC) has recommended safe and adequate daily intake levels of manganese that ranged from 0.3-1 mg day^{-1} for children up to 1 year, 1-2 mg day^{-1} for children up to age 10 and 2-5 mg for children up to 10 and older (Institute of Medicine, 2001). The estimated daily intakes of manganese in this study spanned from 0.03 to 7.7 μg kg^{-1} b.wt. day^{-1} which is far below the recommended adequate daily intake levels.

The Provisional Tolerable Weekly Intake (PTWI) of lead has been set at 25 μg kg^{-1} body weight for children (WHO, 1993) which is equivalent to 3.6 μg kg^{-1} body weight/day. The estimated tolerable daily intake of lead in this study ranged from 0.0-2.9 μg kg^{-1} b.wt. day^{-1}. The upper limit of estimated daily intake exceeded the provisional tolerable intakes of lead. Assuming 40% absorption rate for Pb (http://www.atsdr.cdc.gov/). By consuming 40 g of any of the biscuit types with a lead content of <0.001 to 1.1 μg g^{-1}, a child weighing 15 kg would be acquiring approximately 0.0-32.6 % of his or her PTDI from this source.

The JECFA Provisional Maximal Tolerable Daily Intake (PMTDI) of copper is 500 μg kg^{-1} b.wt. day^{-1} while the Safe Upper Level (SUL) of 160 μg kg^{-1} b.wt. day^{-1} has been recommended by the Expert Group on Vitamins and Minerals (EVM, 2003).The estimated daily intake of copper from consumption of any types of the biscuit ranged 1.4-13.4 μg kg^{-1} b.wt. day^{-1} which is approximately 0.3-2.7% of the PMTDI and 0.83-8.3% of safe upper level.

Normal daily intake of cobalt is reported to be in the range of 2.5 to 3.0 mg day^{-1} poisoning occurs within the range of greater than 23-30 mg cobalt daily (Hokin *et al.*, 2004). The estimated daily intake of cobalt in this study ranged between 0.0-3.5 μg kg^{-1} b.wt. day^{-1} which is by far less than the normal daily intake. The provisional tolerable intake for cadmium has been established at 7 μg kg^{-1} of body weight (WHO, 2001). For a child with body weight of 15 kg, the PTDI for consumption of any of the biscuit types ranged from 0.08-0.13 μg kg^{-1} b.wt. day^{-1} which contributes approximately 8-13% of the PTMDI for cadmium from this source.

CONCLUSION

The metals are present in biscuit samples at concentrations below permissible limits except for nickel and lead in some types. The estimated tolerable daily intakes of metals were below the provisional tolerable intakes for the metals except for nickel and lead. There is need for caution in the consumption of these metals since nickel can exacerbate dermatitis/eczema and while lead can cause reduction of IQ in humans.

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