International Journal of Chemical, Material and Environmental Research 2016, 3 (3): 56-61

Research article

IJCMER
www.ijcmer.org

INTERNATIONAL JOURNAL OF CHEMICAL, MATERIAL AND ENVIRONMENTAL RESEARCH
An International Open Free Access, Peer Reviewed Research Journal

ISSN 2410-5945
2016, Vol. 3, No. 3:
pg. 56-61

Determination of Lead and Cadmium Content in some Selected Processed Wheat Flour

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(Received: August 09, 2016; Accepted: September 01, 2016)

Abstract

In this study, the concentrations of lead and cadmium in eight brands of processed wheat flour consumed were investigated. The metals were determined by flame atomic absorption spectrometry (FAAS) after pressurized microwave-assisted digestion of the processed wheat samples using a mixture of nitric acid (65% w/w HNO_3), and hydrogen peroxide (30% v/v H_2O_2). The measurements were based on calibrations using fortified analytical solutions. The concentration of lead in the flour samples analyzed was in the range of 0.10 mg/kg to 0.20 mg/kg. However, cadmium concentrations were found to be below 0.005 mg/kg and not detected in some flour samples. Digestion efficiency of microwave assisted digestion combined with ease of operation and robust interface of FAAS is an effective way of detecting the presence of metals such as cadmium and lead in food samples. Calculation of monthly intake of cadmium and daily intake of lead showed that the consumption of average amounts of these foodstuffs does not pose a health risk for the consumers.

Keywords: Lead, cadmium, flour, digestion, atomic absorption spectrometry

1. Introduction

Flour is a fine powder made from ground wheat and starchy foods. It is used mainly in baking bread, which is the most readily available food for many cultures. Although flour can be produced from various plants, wheat remains the main source of flour for human consumption. Wheat of high gluten content are called “white”, or “strong” and that of low gluten are called “soft” or “weak” flour. Different classes of wheat are used to make many different types of food. In the US, soft red wheat grown in Ohio is used for making cookies, doughnut, cakes and other fine pastries. Wheat is the most widely produced cereals in the world and it contributes enormously to human energy intake. Its stability, versatility, acceptance and its geographic distribution make it the suitable route for passing micronutrients to mankind [1]. Naturally, wheat is a good source of vitamins B1 (thiamine), B2 (riboflavin), niacin, B6 (pyridoxine), E, together with iron and zinc [2]. During the milling process, a large portion of these micronutrients is lost because most of them are stored in the outer layers of the wheat grain as shown in Figure 1. To restore the micronutrient lost during milling processes and also for enrichment, wheat is fortified with vitamins B1, B2, niacin, and iron. Calcium, folate, vitamins A and D can also be added.

Heavy metals toxicity in soil varies with the anthropogenic activities, nature and characteristics of the soil, and the time elapsed after its contamination. Soil contamination with toxic metals can lead to bioaccumulation of these metals in plants. The toxic metals accumulation in plants is as a result of soil metals uptake around the plant’s roots and deposition on the leaves of the plants and other external plant tissues [3]. Some plants are able to grow on heavy metal contaminated soil wit-

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Published online at www.ijcmer.org

hout any sign of impairment. Harvested crops from plants growth on soil contaminated with toxic metals are susceptible to the corresponding contaminations. Such crops can easily uptake and accumulate these metals and then transfer the potential hazard to human and animals [5-6]. Based on studies by Zhang *et al*. [7] on human exposure to pollutant elements, food rather than atmospheric air remains the major source of non-occupational toxic metals [8]. In developing countries, the problem of food contamination with lead and cadmium is becoming more serious because these metals are readily available in the environment and are particularly toxic.

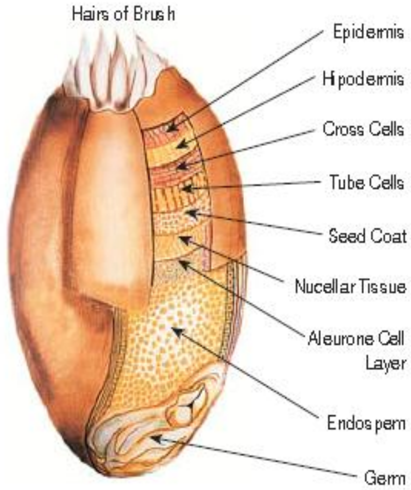

Figure 1. Schematic Diagram of Wheat Grain [4]

Up till date, there has not been any significant mechanism of eliminating heavy metal from human body system; hence their damaging effects on human and animals keep increasing. Metals such as lead and cadmium are both cumulative poisons and highly toxic [6, 9]. Of all the environmental pollutants, lead has the largest toxicological database with more than 10 000 scientific publications [10]. The major source of environmental lead pollution worldwide is leaded automobile fuel which accounts for daily emissions of lead as high as 1000 tons. Generally, in non-smoking population, food and water are the main sources of lead and the effect is more pronounced in children. Its rate of absorption and accumulation (in soft tissues and in bones) is higher in children than in adults. Because of the high sensitivity of the human system to lead, it is assumed that ingestion of lead is harmful at both high and low levels especially for the most sensitive populations (foetuses and young children). Chronic exposure to lead may cause birth defects, mental retardation, psychosis, hyperactivity, shaky hands, muscular weakness, paralysis (beginning in the forearms) and even death. Exposure to high-level concentration may result in intelligent quotient (IQ) and attention span reduction, deficiency in fine and motor control, hyperactivity, inability to concentrate, impaired growth and hearing loss, etc. [11]. In Europe, lead dietary exposure in children ranges between 0.8 and 3.10 (average consumers), and up to 5.51 (high consumers) µg/kg body weight (b.w) per day and in infants between 0.21 and 0.94 µg/kg b.w. per day. Adult exposure ranges from 0.36 to 2.43 µg/kg b.w. per days [12].

Cadmium transfers easily from soil to plant tissues and this makes cadmium a contaminant found predominantly in human foodstuffs thereby making diets a primary source of cadmium exposure in non-smoking and non-occupationally exposed populations. Application of phosphate fertilizers or sewage sludge to the plants in the field can increase cadmium level in some food [13]. More than 80% of dietary cadmium intake comes from cereals, potato, and vegetables with an average daily intake which varies between 8 and 25 µg per day [14]. Toxicity of cadmium became a major concern primarily in 1960 when a painful bone disease "Itai-itai", characterized by renal disease was linked to consumption of cadmium-contaminated rice in an area in Japan [15]. Also in 1993, The International Agency for Research on Cancer classified cadmium as a human carcinogen. Endocrine disruptive nature of cadmium due to its estrogenic properties has been reported [16]. Like lead, cadmium also causes renal dysfunction which leads to osteoporosis a disease characterized by bone demineralization [17]. Reports have shown that the most serious health effect of cadmium toxicity is renal tubular damage which affects both the general population and occupationally exposed workers [18].

The main objective of this study is to determine the concentrations of lead and cadmium in three different brands of processed wheat flour consumed in Malaysia using flame atomic absorption spectroscopy (FAAS). The health risks of dietary exposure to lead and cadmium via processed wheat flour are also discussed.

2. Materials and Methods

2.1. Sampling and Sample Preparation

Eight different brands of widely used processed wheat flour samples were obtained from a supermarket in Taman Melati area of Kuala Lumpur, Malaysia and coded as; AT, TA, TG, TS, TGC, TGB, TB, and TH. Product details and the sample sources were also noted. Two replicate solutions of each sample were prepared by microwave-assisted digestion. Set of digestion blanks were also prepared and subjected to the same microwave procedure. Microwave PTFE vessels were cleaned using 10 mL of concentrated HNO_3, heated for 15 min at 180°C (800W) and then rinsed with deionized water (DIW) before digestion. PTFE evaporation vials were soaked overnight with diluted HNO_3 and then rinsed with DIW.

2.2. Standardization of Instrument

Working standard solutions (100 mg/L) of each element to be determined were prepared by pipetting 5 mL of Cd and Pb stock standard solutions respectively into 100 mL volumetric flasks and then diluted to the mark. For calibration, five Cd and four Pb standard solutions were prepared with the following concentrations: 0.25, 0.5, 1.0, 4.0 and 8.0 mg/L for Cd; and 1.0, 2.5, 5.0 and 8.0 mg/L for Pb. All the solutions were 5% (v/v) in HNO_3. The instrument was standardized with the standard blank (5% v/v HNO_3). The elements were quantified against standard curves prepared on the day of the analysis. The samples were analyzed in triplicate. The correlation coefficient, R^2, obtained for all cases were greater than 0.99. The detection limits (LOD) were calculated as the concentrations of an element that gave the standard deviation of a series of seven consecutive measurements of blank solutions. The detection limits using FAAS were 0.010 mg/L for Cd and 0.002 mg/L for Pb. The detection limit has been defined as the sum of the average value of the blank test and the threefold value of the standard deviation of the blank test (3SD criterion). When the blank value was zero, the standard deviation was calculated measuring samples with analyte concentrations near the detection limits.

2.3. Metal Analysis

The appropriate choice of acid for the wet digestion process of food samples was made [19-21]. All samples were accurately weighted to approximately 0.5 g directly in microwave vessels. 4 mL of high purity HNO_3 was pipetted and added dropwise to the samples in each vessel. Then, 2 mL of H_2O_2 (30% w/w) were added and the vessels were sealed and the pressure relief nut (with safety membrane) was tightened. A total of eight sub-samples were evenly distributed on the microwave turntable. The samples were subjected to a 2-step power (W) with automatic self-programing consisted of a 10 min gradual increase in temperature to 200 °C, a 15 min step at 200°C (1000 W; 106 Pa) and then a ventilated cooling stage. This program was chosen based on recommendations by the manufacturer. After cooling the vessels to room temperature, they were removed to the exhausting hood where the excess pressure was slowly vented. The digested solutions were filtered through 11 μm Whatman filter and quantitatively transferred to 25 mL volumetric flask. All solutions were diluted to 25 mL with DIW and stored in plastic bottles at 4 °C until analysis using FAAS.

2.3.1. Data Quality Control

The accuracy of the data for total lead and cadmium concentration in processed wheat flour has been checked by a parallel analysis of the spiked solution of each sample. 1 mL of 2 mg/L of each standard solution was added to one set of each sample in a 25 mL volumetric flask and made up to the mark. The fortified analytical solution (FAS) was measured three times to obtain the concentration of spiked sample solution and the recovery was calculated. The recoveries of lead and cadmium from the fortified analytical solutions were presented in Table 1. Good recovery range (between 102% and 95%) was recorded for Cd and the recovery of Pb ranges between 55% in TA and 88% in TG. Cd recovery was better than that of Pb.

Table 1. Elemental Concentration in mg/L of Spiked Sample Used for Quality Control

	Pb (mg/L)			Cd (mg/L)		
Sample	**Added**	**Measured**	**Recovery (%)**	**Added**	**Measured**	**Recovery (%)**
AT	0.2	0.115	58	0.2	0.205	102
TA	0.2	0.110	55	0.2	0.194	97
TG	0.2	0.129	65	0.2	0.203	102
TS	0.2	0.101	51	0.2	0.191	96
TGC	0.2	0.155	76	0.2	0.189	95
TGB	0.2	0.176	88	0.2	0.203	102
TB	0.2	0.152	76	0.2	0.193	97
TH	0.2	0.176	88	0.2	0.205	102

n = 3

3. Results and Discussions

Heavy metals such as lead and cadmium affect both the nutritional value of processed wheat and the health of the consuming population. Maximum permissible levels of toxic metals in human food are always put under national and international regulations on food quality. Therefore, controlling the concentration of trace metals in food is very important. Eight samples of processed wheat flour were analyzed to estimate their concentrations of Pb and Cd. Table 2 shows the concentrations of lead and cadmium in the various processed wheat analysed. Four (50%) of the eight samples analysed were cadmium free. Cadmium content of all the four samples was less than 0.005 mg/kg. The lead was detected in all the samples. TS has the highest concentration of lead (0.25 mg/kg) while TB has the lowest (0.12 mg/kg).

Table 2. Concentration of Lead and Cadmium in some Processed Wheat

	Pb		Cd	
Sample	**Conc (mg/Kg)**	**RDS % (n = 3)**	**Conc (mg/Kg)**	**RSD % (n = 3)**
AT	0.20	3.71	< 0.005	1.21
TA	0.15	4.91	< 0.005	1.45
TG	0.22	4.88	< 0.005	1.17
TS	0.25	7.10	< 0.005	1.45
TGC	0.18	5.24	ND	-
TGB	0.15	3.14	ND	-
TB	0.12	4.12	ND	-
TH	0.10	4.56	ND	-

ND - not detected

The content of Pb in the samples was found in the range of 0.25-0.10 mg/kg with the mean content of 0.171 mg/kg. The main factor that affects the high content of lead in grain is absorption through air pollution as a result of the pollution from the highway traffics which can affect the irrigation water or farm soil of the plants. 90% of the lead content of plant material is as a result of atmospheric deposition [22]. In a similar study, high Pb concentration was detected in flour samples and the source of contamination was related to the milling plates in both dry and wet condition [23]. Cadmium content of wheat grain or flour is influenced by several factors such as annual variation, cultivar differences, genetical and regional variations. Regional differences found in cadmium level are mainly affected by cadmium deposition via the atmosphere, soil fertilization and soil properties such as soil type, pH, origin and soil cadmium content [22].

Another important factor that influences the content of lead and cadmium in wheat is its nature at the time of consumption. Previous studies have suggested that lead and cadmium concentrations of wheat decrease linearly from raw materials sources to processed products. This is determined by the nature and complexity of the technological processes involved. It was revealed that there is a decrease in both lead and cadmium content of wheat with a decrease in extraction degree [24]. Wheat bran has the highest concentration and the lowest concentration was found in the processed flour. In fact, Cd concentration in the wheat bran is almost three times higher than those in the flour. These observations showed that the outer part of the wheat grains which is more exposed to the environment present a higher risk of contamination with lead and cadmium. This observation could allude to the very low concentration of cadmium and averagely low concentration lead found in these samples.

3.1. Weekly and Monthly Intake Estimate of Pb and Cd

According to World Health Organization (WHO) guidelines, a provisional tolerable monthly intake (PTMI) of cadmium is set at 25 μg/kg body weight [25], and a provisional tolerable weekly intake (PTWI) of 0.025 mg/kg body weight was set for lead by Joint FAO/WHO Expert Committee on Food [26]. Table 3 shows the estimation of each lead and cadmium intake through consumption of the studied flour samples. An adult with a body weight of 65 kg will have 1.63 mg as his tolerable monthly intake of Cd and the same amount as the tolerable weekly intake of Pb. Based on these results, if he consume an average of 1.5 kg of any of the processed wheat per week, containing the obtained mean level 0.171 mg/kg of Pb

Table 3. Estimation of Lead and Cadmium Intake through Consumption of the Studied Flour Samples

Sample	**Pb (mg/Kg)**	**Intake/week (mg)**	**Cd (mg/Kg)**	**Intake/week (mg)**
AT	0.20	0.30	< 0.005	< 0.0075
TA	0.15	0.23	< 0.005	< 0.0075
TG	0.22	0.33	< 0.005	< 0.0075
TS	0.25	0.38	< 0.005	< 0.0075
TGC	0.18	0.27	ND	-
TGB	0.15	0.23	ND	-
TB	0.12	0.18	ND	-
TH	0.10	0.15	ND	-
Mean Level	0.171	0.257	< 0.005	< 0.0075

and < 0.005 mg/kg of Cd, the average contribution of the processed wheat to his metal intake are 0.257 mg (Pb) and <0.0075 mg (Cd).

Although cadmium accumulates in the kidney and liver with a half-life of several decades if processed wheat flours analysed are the only source of cadmium into the body it may not pose any health risk to the consumers. In the case of lead, although the obtained concentration (0.257 mg) is high compared to Cd, this value represents 16% of the recommended value. Thus, this concentration is not high enough to pose a health risk. This result indicates that concentrations of lead and cadmium in the processed wheat analyses in relation to the estimated provisional tolerable monthly intake (in the case of cadmium) and weekly intake (in the case of lead) are below the set limit by Joint FAO/WHO Expert Committee on Food Additives. Therefore, the consumption of average amounts of this food stuff does not pose a health risk for the consumer.

4. Conclusions

The determination of elements using FAAS with microwave digestion is advantageous mainly because of high digestion efficiency of microwave assisted digestion in terms of accuracy and reliability of sample preparation. Also, FAAS is a very fast and compact instrumental technique with relatively few interferences and robust interface. The concentrations of the analyzed elements are generally below the safe limits. These results give valuable information on the amount of lead and cadmium in some selected processed wheat consume in Malaysia. This results can also be adopted to evaluate the chemical contents of varieties of processed grains to check the possible risk involve in their public consumption. It should be noted that the influence of soil contamination of cadmium, and its absorption by plants, half of the sample analysed are cadmium free and others contain very small amount of cadmium. The concentrations of lead in all the eight samples are very minimal. There may be some species of wheat varieties with little or no capability to absorb cadmium and with the ability to absorb less lead content if any. It could, therefore, be of scientific interest to know the species of such grains and the characteristics of the soil of the areas concerning lead and cadmium contents and factors associated with the transfer of the metals in the soil to cereal grains where the sample cereals were harvested. Unfortunately, the harvest location was not known and no data was available on the geochemistry of the soil where the grains were harvested. It could be concluded that the obtained estimated daily intake for lead and cadmium are below the reported limit by the Joint FAO/WHO Expert Committee on Food Additives, 2010 who set a limit for lead and cadmium intake based on body weight (60 kg-70 kg) of an average adult. Therefore, the consumption of average amount could not affect the health of the consumers.

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