

# Elemental Evaluation of Cereals Commonly Used in Nigeria Using Neutron Activation Analysis

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## Abstract

The concentrations of twenty elements in ten cereals types commonly consumed in Nigeria were determined using Neutron Activation Analysis (NAA). The elements are Al, As, Ba, Br, Ca, Cl, Co, Fe, K, La, Mg, Mn, Na, Rb, Sc, Sm, Sr, Th, V, Zn and the cereals are beans, guinea corn (red and white), maize (white and yellow), millet, rice (basmati, foreign, and local) and wheat. The results obtained for these elements were compared with World Health Organization (WHO) permissible limits. This showed that the concentrations of all the heavy metals studied (Co, Fe, La, Mn, Sm, Th, V, Zn) and some of the mineral elements (Ca, K, Mg, Na) in the cereal samples were below WHO permissible limits except wheat that showed a higher value in Mg (2.309 %) compared with the WHO permissible limit of 0.139 %. The levels of the twenty elements in the cereals did not exceed their permissible levels and are therefore safe for human consumption.

**Keywords:** Neutron Activation Analysis; heavy metals; mineral elements; cereals

## INTRODUCTION

Cereals are small, hard, dry seeds, with or without attached hulls or fruit layers, harvested for human or animal consumption (Babcock & German, 1982). Cereals are edible seeds and are released from the plant when they fully mature. Cereals constitute one of the classes of grains, which can be divided into three groups; cereals (maize, millet, rice, wheat, etc.), pulses (beans, cowpeas, peas, etc.), and oil seeds (linseed, soybeans, sunflower, etc.). Because cereals are

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small, hard and dry, they can be stored, measured, and transported more readily than any other kinds of food crops such as fresh fruits, roots and tubers. The development of cereal agriculture allowed excess food to be produced and stored easily that could have led to the creation of the first permanent settlements and the division of society into classes (Wessel, 1984). Cereals are edible seeds and, as such, would eventually be released from the plant when fully mature.

Cereals such as maize, millet, guinea corn, rice, and wheat having several species are widely cultivated in Nigeria especially in the northern states of Nigeria (Jones, 1995). The main sources of all the essential elements, heavy metals in these cereals are the soil, pollutants from the environment and the use of fertilizer and pesticides such as fungicides and insecticides. The cereals; guinea corn, maize, millet, rice, and wheat forms about 40-60 % of the daily dietary intake in not only the northern states but also in other states of Nigeria (Jones, 1995). They are therefore representative of the daily dietary intake in Nigeria.

There is a growing concern about the physiological and behavioral effects of trace metals in the human population. For example, cadmium contamination in agricultural products was reported in Thailand (Rosentrater & Evers, 2017). Contamination by these metals is an important environmental and health issue as they are toxic even at low concentrations (Chukwuma, 1997). Food composition data is necessary when estimating a population's intake of nutrients and dietary exposure to toxins and indeed, assessing diet quality (Sramkova, Gregova, & Sturdik, 2009) Therefore, metal analysis of foods and fruits is an important aspects of food quality assurance (Elbagermi, Edwards, & Alajtal, 2012; Ismail *et al.*, 2011).

Heavy metals may enter the human body via food, water, air, or absorption through the skin in agricultural, industrial, or residential settings (Dupler, 2001). Coarse cereal and legumes grains being a major source of carbohydrates, protein and fat, form the major part of human diets and are the most staple food consumed and cultivated primarily for human food, livestock feed, seed and as a raw material for starch, bio-fuel and food processing industries. At present, cereals have attracted more attention to common diet because they contain beneficial components. Among these products, millet, maize, sorghum, wheat, cowpea and rice are the most popular agricultural products consumed for medical purpose and maintaining good health (Khan *et al.*, 2008 & Zhou *et al.*, 2007). According to statistics from the Food and Agricultural Organization (FAO) of the United Nations, the average annual global production of sorghum in 1979 was 68.7 million tons and over 52 million hectares were planted each year with numerous varieties of sorghum and millet (Faruruwa, Birnin-Yauri, & Dangogo, 2013). The two crops are the most important and most common plants cultivated in the far northern part of Nigeria (the area characterized with low rainfall). Heavy metals are mobile and easily taken up by the plants in the environment (Khairiah *et al.*, 2004).

Recent research suggests that inputs of calcium, 40 tonnes of cadmium and 767 tonnes of lead annually enter agricultural soils in England and Wales (Alloway *et al.*, 2000). In that work, approximately 52 % of cadmium and 76 % of lead was from atmospheric deposition, with a further 5 % and 15 % of cadmium and lead coming from sewage sludge applications. However, the total amounts of atmospheric deposition for the whole country can be misleading, as they are based on small amounts per unit area multiplied by a very large land area. Metals uptake by plants may pose risks to human beings when such plants are grown on or near contaminated areas. Metal accumulation in plant depends on plant species, genetics, types of soil and metal, soil conditions, weather and environment. Other factors are stage of maturity and supply route to the market, (Ismail *et al.*, 2011).

Nowadays, more land are being contaminated with toxic elements owing to the use of sludge or municipal compost, pesticides, fertilizers and emissions from industrial and municipal waste incinerators, car exhausts, residues from metalliferous mines, and smelting industries (Gupta et al., 2008).

Since toxic elements in the environment can be harmful to human health through the food chain, public concern about the environmental impact has grown in recent decades. Cereal particularly rice, wheat, maize, millet and sorghum are among the main dietary food for supplying trace elements and nutrients. The distribution and concentration of several toxic elements in these cereals has become a source of concern to public health (Yamusa et al., 2013). The entry of cadmium into the food chain is of concern as it can cause adverse health problems in humans such as renal dysfunction, bone disease, lung edema, liver damage, anemia, and hypertension and has recently been associated with brittle bones (Wang et al., 2005). Due to this, cadmium is one of a very small group of metals for which the Food and Agricultural Organization (FAO)/World Health Organization (WHO)(1995; 2007) have set a limit for the provisional daily intake by humans as 70 µg cadmium/day. For example, in the 1980's the US adult population was reported to receive about 20 % of the FAO/WHO (1995; 2007) allowable daily intake of cadmium from the consumption of cereal products (Adams et al, 2001). In contrast, during the same period in the early 1980's, cereal products accounted for about 30 to 40 % of the daily allowable cadmium intake in the European Community (Hutton, 1982).

In a previous study, Buruchara (2006) investigated the cadmium content of British wheat grain in samples collected for the Home Grown Cereals Authority (HGCA) wheat quality assessments in 1982, 1992 and 1993. Mean wheat cadmium concentrations in samples from 1992 and 1993 were similar (0.042 and 0.038 mg kg<sup>-1</sup> dry weight respectively) but were slightly lower than the mean concentration in the 1982 samples (0.052 mg kg<sup>-1</sup>).

Beans, Maize, millet, rice, sorghum, and wheat are staple cereals and major sources of micronutrients for rural dwellers of Northeast Nigeria. Research has shown that certain heavy metals are nutritionally essential for a healthy life in very small quantities such as (Cu, Fe, Mn and Zn). Heavy metals become toxic, when they are not metabolized by the body but gather in the soft tissues. Heavy metals rank high amongst the chief contaminants of leafy vegetables and medicinal plants (Yamada, 2013) because heavy metals are non-biodegradable so they cause serious health hazards in human beings and animals. The main source of metal accumulation in human beings is the plants and vegetables consumed by them that are grown in polluted areas.

According to Food and Agricultural Organization FAO (2007), cereals have long been regarded as valuable sources of essential nutrients. They provide energy, protein, minerals, and vitamins in the human diet. Global production of cereals grains has been approximately 2000 million tonnes over recent years (Simmons et al., 2005). They are cultivated primarily for human food, livestock feed, seed and as a source material for starch, biofuel and other industries. This study is very necessary to assist in national planning for the analysis of national food supplies particularly from regions in Nigeria that could be prone to deficiencies or excesses of the nutritionally important elements. It will increase our knowledge on the metal contamination of cereals as indication of the effects of environmental pollution.

The aim of this research is to determine the concentration of heavy metals in the grain (millet, guinea corn, maize, rice) and legumes (beans) commonly consumed in Nigeria. This study would assess the concentration of the cereals consumed for selected heavy metals, minerals and trace elements. The studies will further assess the extent of exposure to heavy metals, minerals and trace elements through food intake and the potential health implications as well as identify the foods (Cereals) that are rich in metals. Finally, this study would determine the

adverse effects of toxic / contaminated cereals to the consumers and compare the results with the Recommended Daily Allowance (RDA) and Upper Tolerable Intake (UTI) level of the metals so as to provide suggestions for actions to reduce any contamination.

## **MATERIALS AND METHODS**

The Nigeria Research Reactor-1 (NIRR-1) is specifically designed for use in neutron activation analysis (NAA). Thus, there is the need for a complete and careful characterization of the neutron flux parameters in the irradiation channels in order to optimize its utilization for NAA through relative, absolute and single comparator methods. Stable neutron flux characteristics are exhibited by low-power research reactors, such as, the NIRR-1, the Canadian SLOWPOKE and the Chinese MNSR, which are suitable for NAA via the k<sub>0</sub>-standardized method (Akaho & Nyarko, 2002; Acharya & Chatt, 2003). A detailed description of the NIRR-1 and the irradiation facility has been published by Jonah (2005).

The association facility for radioactivity measurements after irradiation is a gamma-ray data acquisition system. It consists of a horizontal dip-stick High-purity Germanium (HPGe) detector with a relative efficiency of 10 % at 1332.5 keV gamma ray line, the MAESTRO emulation software compatible with the ADCAM multi-channel analyzer (MCA) card, associated electronic modules all made by EG and ORTEC and a personal computer. The efficiency curves of the detector geometries have been determined by standard gamma-ray sources in the energy range of 59.5-2254 keV and were extended to 4000 keV by a semi-empirical method (Jonah, 2005). For data processing, the gamma ray spectrum analysis software WINSPAN 2004, software developed at CIAE Beijing, China is used,. On the basis of the well-known activation equation, the software requires that calibration factors be pre-determined by a multi-elements standard reference material for elements of interest using adopted irradiation and counting regimes. In addition to NAA calculations, WINSPAN 2004 performs peak analysis, remote control of the multi-channel analyzer (MCA) and other auxiliary functions such as efficiency calibration, and nuclear data generation.

The following materials and apparatus were used for the preparation of samples: spatula, forceps, hand dryer, analytical weighing balance (Metler AC 100), agate mortar and pestle, oven, polythene bags and rabbit capsules.

Ten different cereal samples; basmati rice, beans, foreign rice, guinea corn red, guinea corn white, local rice, white maize, yellow maize, millet and wheat were bought in some Nigeria markets. Polythene bags of about 3 cm x 4 cm sizes were used to package the samples and were all sealed. All the samples were crushed into grain size of about 125 µm using an agate mortar and pestle and mass of 0.25 - 0.30 g was weighed for irradiation. Prior to using the polythene films, they were soaked in HNO<sub>3</sub> solution for three days in order to remove all impurities and later on washed with deionized water. Then, they were dried in the oven at 60 oC for 12 hours. Gloves were used in order to minimize contamination of samples during preparation. The samples were then packaged and carefully placed inside the capsule vial using the forceps. Cotton wool was added on top before sealing with a cello tape. This was done in order to avoid loss of samples in the irradiation channel. For short lived irradiation, only one sample was placed in a vial while for long lived irradiation, multiple samples were packaged since the maximum temperature inside the irradiation sites does not exceed 50 oC. Both the capsules and spacers are made of high-density polythene materials having high radiation resistance properties.

Sample irradiation involved the exposure of the sample to nuclear radiation (fission reaction). When the samples are bombarded with neutrons, the elements get excited and become

radioactive, releasing their characteristic radiations (i. e. emitting gamma rays with energies unique to each of the radioactive element). The basic premise of detection and counting is that the decay rate (R) of the source or activity of the sample is proportional to the number (N) of radioactive nuclides present, the proportionality constant being the decay constant,  $\lambda$  (Njinga et al., 2015). Thus:

$$R = \frac{\delta N}{\delta t} = \lambda N \tag{1}$$

Given the count for the number of events C, detected by the detector in a fixed period of time  $\delta t$ , it can be estimated that the decay rate is as follows.

$$R = \frac{C}{\varepsilon \delta t} \tag{2}$$

Where  $\varepsilon$  is the efficiency of counting taking into account the source, detector geometry, the intrinsic detection efficiency of the particular radiation emitted. Considering that each atom in the sample will have a certain probability P that the atom will decay during the period of measurement.

$$P = (1 - e^{-\lambda \delta t}) \tag{3}$$

Then, the overall probability of detection as opposed to decay is  $P\varepsilon$  and the expected count is

$$E(C) = P\varepsilon N \tag{4}$$

If the measured count (C) is an estimate of the expected count, then equation (1) can be used to employed to calculate the rate of decay as.

$$R = \lambda N = \frac{\lambda C}{(1 - e^{-\lambda \delta t})\varepsilon} \tag{5}$$

In this work, the certified reference material NIST 1547 (Peach leaves) was used to determine the calibration factors for all the elements being the closest matrix-matching reference material for the samples. For the determination of calibration factors, sample of standards of approximately 2.50 g were weighed and wrapped in polyethylene films.

## RESULTS AND DISCUSSION

The results of the analysis of twelve elements contained in the ten samples are presented in the bar-chart (Figure 1) below:

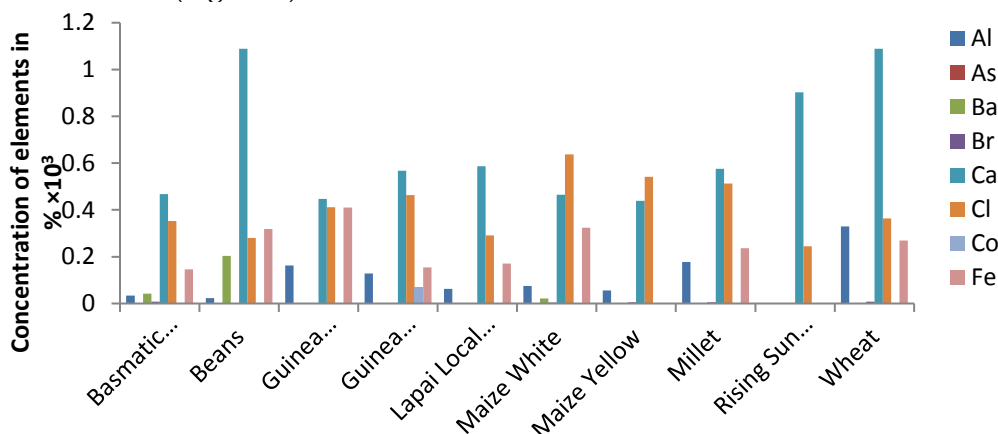


Figure 1: The elemental Concentration in various samples (Al-Fe) in %

From Figure 1, it was observed that among all the elements in the ten samples, Calcium (Ca) recorded the highest concentration in Beans ( $1.089 \pm 0.53$  %) and Wheat ( $1.089 \pm 0.123$  %) and was followed by Rising Sun Rice (RSR) ( $0.902 \pm 0.111$  %). White Maize ( $0.637 \pm 0.16$  %) recorded the highest level of Cl in all the cereal samples and was followed by Yellow Maize ( $0.541 \pm 15$  %). Barium (Ba) was below the detection limits in almost all cereal samples except in Basmati Rice ( $0.0422 \pm 10.2$  %), Beans ( $0.0204 \pm 4$  %), and White Maize ( $0.0214 \pm 1$  %).

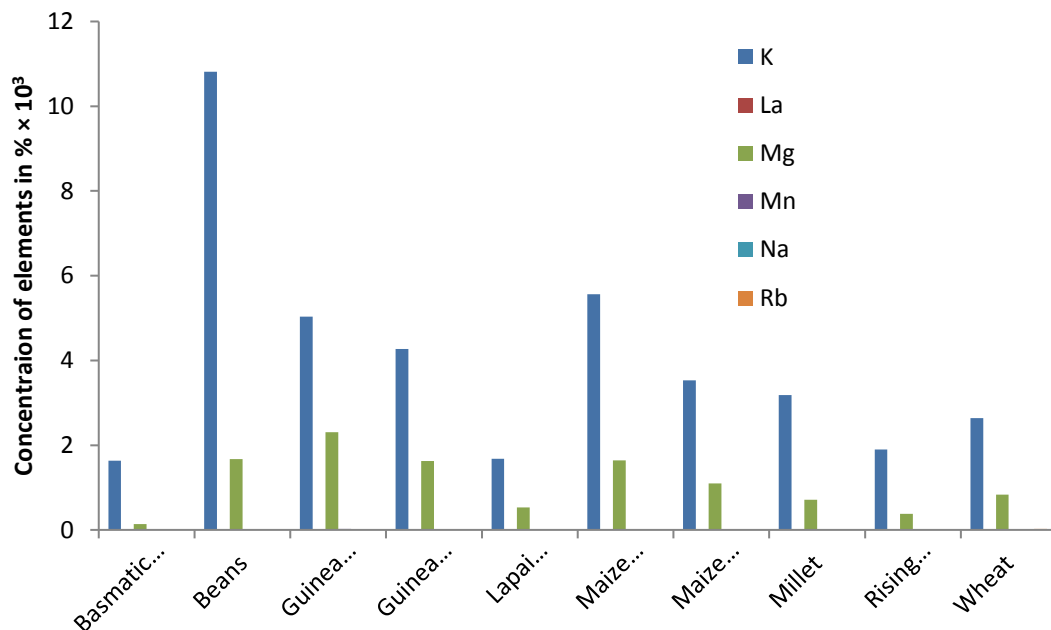


Figure 2: The elemental Concentration in various samples (K-Rb) in %

In Figure 2, K has a relative high concentration over all other elements and Beans recorded the highest concentration ( $10.810 \pm 1.84$  %) among other samples analyzed and was followed by White maize ( $5.559 \pm 1.33$  %) and Red guinea corn ( $5.034 \pm 1.31$  %). Basmati rice recorded the lowest K concentration ( $1.630 \pm 0.42$  %).

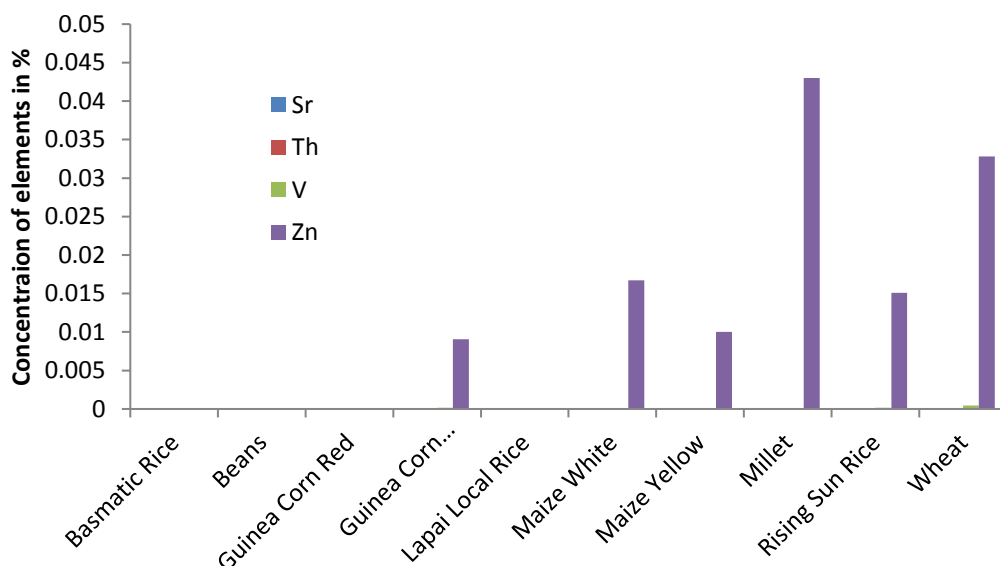


Figure 3: The elemental concentrations in various samples (Sr-Zn) in %

In Figure 3, Sr and Th were below detection limits in all the ten cereal samples analyzed, Vanadium (V) was also below detection limit in all the cereal samples except in the White

Guinea Corn ( $0.00017 \pm 0.03$  %), Rising Sun Rice ( $0.00015 \pm 0.00004$  %) and Wheat ( $0.00044 \pm 0.0009$  %). Millet recorded the highest concentration of Zn ( $0.043 \pm 0.004$  %).

**Table 1:** Samples and elements with highest values compared to WHO permissible limits (%)

Elements	Samples with highest values	This study (%)	WHO permissible Limits (%)
Mg	Red guinea corn	2.309	0.139
Ca	Wheat	1.089	6.3066
Fe	Wheat	0.269	8.16
Mn	Wheat	0.0278	0.54
V	Wheat	0.0004	0.013
Zn	Millet	0.0430	0.16

From Table 1, only Red guinea corn had the highest value in Mg (2.309 %), which is above the WHO limits of 0.139 % for Mg.

**Table 2:** Essential and trace elements in the ten samples compared to WHO permissible limits in human beings (%)

Samples		BR	BS	GCR	GCW	LLR	MW	MY	MT	RSR	WT
Mg	This work	0.135	1.67	2.309	1.623	0.528	1.638	1.095	0.71	0.376	0.829
	WHO	0.139	0.139	0.139	0.139	0.139	0.139	0.139	0.139	0.139	0.139
Ca	This work	0.467	1.089	0.447	0.568	0.587	0.637	0.439	0.575	0.902	1.089
	WHO	6.306	6.306	6.306	6.306	6.306	6.306	6.306	6.306	6.306	6.306
Na	This work	0.011	0.00736	0.0053	0.011	0.019	0.006	0.004	0.006	0.013	0.011
	WHO	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Fe	This work	0.146	0.319	0.41	0.154	0.17	0.324	BDL	0.237	BDL	0.269
	WHO	8.16	8.16	8.16	8.16	8.16	8.16	8.16	8.16	8.16	8.16
Mn	This work	0.003	0.0197	0.0213	0.016	0.0135	0.009	0.006	0.012	0.008	0.028
	WHO	0.54	0.54	0.54	0.54	0.54	0.54	0.54	0.54	0.54	0.54
Zn	This work	0.0098	0.0166	0.0119	0.00908	0.0243	0.017	0.01	0.043	0.0151	0.0328
	WHO	0.16	0.16	0.16	0.16	0.16	0.16	0.16	0.16	0.16	0.16
V	This work	BDL	BDL	BDL	0.0017	BDL	BDL	BDL	BDL	0.00015	0.0004
	WHO	0.013	0.013	0.013	0.013	0.013	0.013	0.013	0.013	0.013	0.013

Where BR-Basmati Rice, BS-Beans, GCR-Guinea Corn (Red), GCW-Guinea Corn (White), LLR-Lapai Local Rice, MW-Maize (White), MY-Maize (Yellow), MT Millet, RSR-Rising Sun Rice, WT-Wheat, BDL-Bellow Detection Limit.

From Table 2, the results of BS, GCR, GCW, MW and MY for Mg were 1.670, 2.309, 1.623, 1.638 and 1.095 % respectively and all these values were above the WHO permissible limits in human beings (0.139). The results of the samples for Calcium, Iron, Manganese, Sodium, Vanadium and Zinc were within the WHO permissible limits. According to Senofonte, Violante, and Caroli (2000), Ca and Mg are essential macro nutrients required for proper functioning of the human body. For instance, Ca is a constituent of bone and required for muscular contraction. Mg plays a role in the transport process of Na and K across cellular membrane and in the functional integrity of the neuromuscular system.

Heavy metals have been proved to be toxic to both human health and the environment. Owing to their toxicity and their possible bioaccumulation, they should be subjected to mandatory monitoring and survey. From the results of the ten grain samples analyzed using NAA technique, the concentrations of trace, toxic and mineral elements in the samples varied and this may be due to many factors arising especially from cultivation, post-harvest and storage processes. Elemental composition of soil, fertilizers and agricultural chemicals (herbicides, fungicides and insecticides) usually play a significant contribution to the elemental composition in cereals (Yamusa et al., 2013). It is suggested that further research and investigation be carried out on the status of heavy metals in other food stuffs and agricultural soils in the areas where the food crops are cultivated.

## **CONCLUSION**

This study evaluated the concentrations of Al, As, Ba, Br, Ca, Cl, Co, Fe, K, La, Mg, Mn, Na, Rb, Sc, Sm, Sr, Th, V, Zn in beans, guinea corn (red and white), maize (white and yellow), millet, and rice (Basmati, foreign, and local) that are consumed almost daily in Nigeria using the Neutron Activation Analysis (NAA) technique. The results obtained were compared with World Health Organization (WHO) permissible limits in order to ascertain if the cereal types posed some health risks. It was revealed that the concentrations of all the heavy metals: Co, Fe, La, Mn, Sm, Th, V, Zn studied and some of the mineral elements: Ca, K, Mg, Na in the cereal samples were below WHO permissible limits with the exception of wheat that showed a higher value in Mg (2.309 %) compared with the WHO permissible limit of 0.139 %.

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