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Determination of Lead (Pb) and Cadmium (Cd) in Selected SPAGHETTI Samples Marketed at SOKOTO Central Market

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ABSTRACT: The contamination of lead and cadmium were determined in selected spaghetti samples marketed at Sokoto Central Market, using Atomic Absorption Spectroscopy (A.A.S), after wet digestion of the samples using an oxi-acidic mixture of HNO_3/H_2O_2 (4:1). The lead concentrations were found to be high in Golden penny, Power pasta, Honey well, Dangote standard, Delicia, Marina spaghetti samples (22.3 – 50mg/kg). The lowest and highest contents of cadmium (Cd) were found in Honey well (3.28 ± 0.0245mg/kg) and Golden penny (101.65 ± 0.0029mg/kg), respectively. Power pasta, Delicia and Marina were noted for their low level of cadmium (Cd), which is below the detection limit of the instrument. The presence of lead (Pb) and cadmium (Cd) in high concentration in spaghetti is dangerous to health; more especially to the children. Therefore, the daily intake of these spaghetti should be minimized; more especially children.

I INTRODUCTION

Heavy metals such as cadmium and lead are natural constituents of earth's crust, their ubiquitous resulting largely from anthropogenic activities: Mining activities, burning fossil fuels, paint industry, waste storage and incineration etc (Gabriel *et al*., 2013). Air is considered to be a pollution source with lead and cadmium, as metal Particles emitted from factories, cars etc, and were carried by air currents and are deposited on soil and plant surface. Metals can change their chemical nature but cannot be degraded or destroyed, which means that they are persistent in the environment and can bioaccumulate in the animal or plant body (Gabriel *et al*., 2013).

Foods are important for human survival; they may contain heavy metals which can either be beneficial or hazardous to man, hence their safe level in food need to be investigated (Olabimpe *et al*., 2015).

Plants are important components of the ecosystem because they transfer essential elements from the abiotic environment in to the biotic one. The main sources of elements for the plants are: air, water, soil. Water used for the irrigation of vegetables, may be contaminated with these toxic metals as a result of waste water treatment activities, discharge of domestic waste in the surface water. When due to repeated irrigation with sewage waters the soil capacity to retain these toxic metals is reduced, cadmium and lead can pass in to ground waters by the process known as leaching (Gabriel *et al*., 2013).

Important positive and negative roles of trace heavy metal ions in human health are known. Lots of studies have performed for the determination of trace metal ions in various media including some body tissues and fluids, natural waters etc. Also the investigation of trace heavy metal contents in food samples including honey, vinegar, lemon juice, spaghetti, molasses and other food samples are an important part of analytical chemistry (Saracoglu *et al*., 2002).

A. Major Sources of Exposure to Heavy Metals

The term heavy metal refers to any metallic chemical element or group of metals and metalloids with five times or more the specific gravity of water and is toxic or poisonous at low concentrations. Heavy metals are among the most common environmental pollutants and their occurrence in water and biota indicate the presence of natural and anthropogenic sources (Baise *et al*., 2013).

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Heavy metals classification includes transition metals and higher atomic weight metals of groups (III) to (V) of the periodic table, however, the term trace metals is becoming a popular preference to heavy metals but has not attain such a common usage. The widely recognized term "heavy metals" would therefore be retained in this work. High industrial activities are major sources of air and water pollution by effluents from these industries. In Nigeria, we cannot negate industrialization as a threat to our environment. Therefore its effects on soils and water are increasingly alarming. Our entire earth is an oasis of chemical substances. We have an abundance of water and diatomic oxygen, the chemical substances of overwhelming importance to life, and then we have vast reserve of heavy metals (Ademoroti, 1996).

Humans can be exposed to heavy metals through a variety of means; including consumption of contaminated food. Although heavy metals are usually present in foods at very low levels, long term exposure can have negative health impacts. Two of the more important toxic elements that most be monitored are cadmium (Cd) and lead (Pb), which can enter food either through environmental processes or through contamination in processing and/ or packaging. As a result, it is very important to accurately measure low levels of Cd and Pb in a variety of food matrices (Gabriel *et al*., 2013).

Grains, fruits and vegetables grown on soils contaminated with these metals retain large enough quantities to constitute a major source of exposure to these toxics. Another problem represents the use of chemicals: fertilizers, insecticides, fungicide and herbicides. These products may contain metals and their addition can increase the concentration of toxic metals in soil. However, the chemical and physical form of the metals found in the soil can influence their uptake by plants (Gabriel *et al*., 2013).

Another important source of food pollution with lead and cadmium is represented by the foodstuffs contact with the processing machineries, preserving foodstuffs in metal packaging and of course, the influence of processing factors (Gabriel *et al*., 2013).

B.Lead

Is a grayish soft metal with a melting point and boiling point of 327.5^{o}C, 1740^{0}C respectively, at atmospheric pressure. It has four naturally occurring isotopes with ratios depending on the various mineral resources. It has +2 oxidation states in its inorganic compounds. Tetraethyl lead and tetramethyl lead are organic Pb compounds extensively used as fuel additive which tent to concentrate because of their low volatility during the evaporation of gasoline. 80-9 % of Pb emission in to the atmosphere is caused by the combustion of alkyl Pb additives in motor fuel. Other sources are mining and smelting of Pb ores, refining and manufacturing of compounds and Pb- containing goods, and refuse incineration. The principal route of exposure for general human in population is food, and contaminated drinking water, and lead dust brought home by industrial workers on their clothes and shoes (Manton *et al* ., 2000).

C. Cadmium

Is a soft, light-coloured metal with high vapour causing it to be rapidly oxidized to cadmium oxide in air. In nature, Cd occurs together with Zn at a concentration of about 0.1-1% and is a byproduct of Zn refining as some Zn ores may contain as much as 5% Cd. Zinc production is the largest source of Cd supply. Cadmium compounds are used in metal electroplating, as stabilizers or pigments in plastics, in alkaline batteries and in alloys. In Europe, the major sources of Cd emissions are Zn production, volcanic activity, steel industry and waste incineration, the latter probably being the most troublesome pollution for the near future (Ziemacki *et al*., 1989)

D.Toxic Effects of Heavy Metals on Human

Heavy metals such as cadmium (Cd) and lead (Ld), are highly toxic metals which are quickly absorbed from the alimentary tract. Subsequently, they easily pass through biological barriers and accumulate in the internal organs. Even small amounts of Pb and Cd may cause metabolic disorders (Filon *et al*., 2013).

Lead is the cause of many diseases, including cancer of stomach, ovaries, kidneys and leukemia; it also causes irreversible damage to the nervous system. At high concentration, it causes seizure, coma and death if not treated

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immediately. In adults, lead is very harmful to the cardiovascular system. Exposure to it regularly may cause high blood pressure and are at increased risk for myocardial infarction and stroke (Olabimpe , 2015).

Cadmium is known as a human carcinogen and its critical target is the kidney for general population. Cadmium bioaccumulation inside the body causes kidney impairment and lung dysfunction in infected individual (Judilyn *et al*., 2013). Cadmium even at lower levels over a long period of time can lead to a build up cadmium in the kidney causing a serious damage. It's also responsible for decalcification and deformation of bones, hypertension and impotence (Filon *et al*., 2013).

II.LITERATURE REVIEW

The presence of heavy metals in foods is a matter of serious concern because of their toxicity. There has been a report confirming possible adverse effects of heavy metals on human arising from consumption of food and water containing metals above threshold limit (Keith, 1976).

Olabimpe (2005) reported the determination of heavy metals (Zinc, Copper, Cadmium and Lead) in staple foods consumed by the students within University campus Ado Ekiti. The staple foods {spaghetti, jullof rice, rice with stew, wheat with soup, yam, amala (cassava and yam flour), garri (fried cassava), eba, potato} samples were analyzed after dry ashing digestion for Zinc, Copper, Lead and Cadmium using atomic absorption spectrophotometer. The staple foods were categorized in to cereals, legumes, and tubers respectively. In the staple foods, the mean concentration (mg/kg) of zinc (11.1 ± 5.34 – 16.6 ± 11.9), copper (3.70 ± 3.30 – 5.60 ± 5.10), cadmium (0.02 ± 0.02 – 0.28 ± 0.23), and lead (0.10 ± 0.10 – 0.22 ± 0.17) was reported. Highest concentrations (mg/kg) of cadmium (0.28 ± 0.23) were found in cereals while the highest concentrations of lead (0.22 ± 0.17) were found in legumes and tubers.

Filon *et al*., (2013) reported the lead (Pb) and cadmium (Cd) in province of podlasie while monitoring health quality of food products. Pb and Cd concentrations were determined with the Atomic Absorption Spectroscopy (AAS) method. The following foodstuffs were analyzed: flour, grots, bread, pasta, rice, and soya product. The highest cadmium level was noted in pastas (0.058 ± 0.0330 mg/kg) and the highest Pb level was determined in cuscus (0.120 ± 0.0899 mg/kg).

In the light of the above literature, this project work was undertaken to determine heavy metals in 10 different brand type spaghetti (five indigenous and five foreign) marketed at Sokoto Central Market, using Atomic Absorption Spectrometry (A.A.S).

III. MATERIALS AND METHODS

A. Materials

A.1 Sample Treatment

20g collected subsamples of spaghetti from each brand type were crumbles in to small pieces and finally ground by stainless steel mill.

B. Method

B.1 Sample Digestion

The sample (1 g) was digested using 10ml of oxi-acidic mixture of HNO_3/H_2O_2 (4:1) in a 100ml beaker inside a hood. This mixture was heated up to 120⁰ C for 3hr, cooled and the solution was filtered (using NO.1 Watt man filter paper) in to a 100cm^3 volumetric flask. The residue was washed 3 times and the content was brought up to a volume of

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25ml with distilled water. All other samples were treated in similar manner. Blank digestion was also carried out in the same way except that no sample was added (Belay, 2014).

B.2 Analysis of Pb and Cd using AAS

In this analysis (atomic absorption spectrometry) (AAS) was used due to its good precision and accuracy. The principle of the method is based on nebulizing a sample solution into an air-acetylene flame where it vaporized. Elemental ions are atomized and the atoms formed absorbed radiation of the characteristic wavelength from a hollow-cathode lamp. The absorbance measured, is proportional to the amount of analyte in the sample solution.

B.2.1 Preparation of Stock Solution

(a) Cadmium Stock Solution

Cadmium nitrate tetra hydrate Cd $(NO_3).4H_2O$ (2.744g) was dissolved in10cm^3 of distilled water in a 1000cm^3 volumetric flask and 4.5cm^3 of concentrated H_2SO_4 was added to the solution and diluted to 1000cm^3 with distilled water (Walinga *et al.* ,1989).

(b) Lead Stock Solution

Lead nitrate Pb $(NO_3)_2$ (1.599g) was dissolved in 10cm^3 of nitric acid and diluted to 1dm^3 with distilled water in a 1000cm^3 volumetric flask (Walinga *et al.*,1989)

B.2.2 Procedure

The stock solutions (1000 ppm) of Cd, and Pb, were used to prepare working standard solutions by appropriate dilutions. Spectrophotometer (AA 500, PG analyzer model No P163) was set up in accordance with the manufacturer's instructions for each element to be analyzed. These include fuel (acetylene) and oxidant (oxygen gas) selection, burner type, an optimum wavelength and slit-width settings. The standards, blanks and the samples were aspirated into the flame and their concentrations in ppm were recorded automatically.

The concentration of each analyte (X), in the samples was calculated using equation 2.1.

$$X\ (mg/kg) = \frac{X\ (ppm)\ x\ vol.\ of\ sample\ made}{Weight\ of\ the\ sample} \qquad \text{-----------------}\ (2.1).$$

B.2.3 Statistical Analysis

Data obtained was presented as mean ± standard error of means of triplicate values.

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IV.RESULTS AND DISCUSSION

The results of lead (Pb) and cadmium (Cd) content in spaghetti samples collected at Sokoto Central Market is shown in Table 3.1.

Table 3.1: Concentration of lead (Pb) and cadmium (Cd) in spaghetti (mg/kg)

Samples	Pb	Cd
Golden Penny	22.23 ± 0.0014	101.650 ± 0.003
Power pasta	50.00 ± 0.000	ND
Honey well	22.23 ± 0.0014	3.28 ± 0.02
Dangote standard	22.23 ± 0.0014	15.58 ± 0.001
Dangote slim	ND	11.48 ± 0.001
Aicha	ND	15.58 ± 0.00
Delicia	22.23 ± 0.004	ND
Naama	ND	19.68 ±0.001
Marina	50.00 ± 0.0014	ND
Oriba	ND	64.75 ± 0.01

Values are expressed as mean ± standard error of mean of triplicate reading.
ND = Not detectable.

A. Discussion

The results generally show the presence of lead (Pb) and cadmium (Cd) in the spaghetti samples marketed in Sokoto central market.

Lead concentrations of the Golden penny, Power pasta, Honey well, Dangote standard, Delicia, Marina spaghetti samples (22.3 – 50mg/kg) were above the allowable limit of 0.300mg/kg set by FAO/WHO (2001). On the other hand, Dengote slim, Aicha, Naama, Oriba were noted for their low level of lead (Pb), which is below the detection limit of the instrument. The values reported herein are higher when compared with the values (0.120 ± 0.0899mg/kg) reported by Filon *et al* (2013) and (0.22 ± 0.17mg/kg) reported by Olabimpe *et al* (2015).

For cadmium, the allowable limit set by FAO/WHO (2001) is 0.200mg/l. All the samples except Power pasta, Delicia and Marina have cadmium (Cd) content above this standard. The lowest and highest contents of cadmium (Cd) were found in Honey well (3.28 ± 0.0245mg/kg) and Golden penny (101.65 ± 0.0029mg/kg) respectively. The values reported herein are higher when compared with the values (0.058 ± 0.0330mg/kg) reported by filon *et al* (2013) and (0.28 ± 0.23mg/kg) reported by Olabimpe *et al* (2015).

The high concentrations of lead (Pb) and cadmium (Cd) in the selected spaghetti samples takes it source from the agricultural soil that propagates the plant (Wheat) used in processing the flower from which the spaghetti are produced. Another important source of these high concentrations represents the foodstuff contact with the processing machineries,

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high intensity of industrial activities within the manufacturing region and of course, the influence of processing factors.

Results from this study is quite alarming since cadmium (Cd) and lead (Pb) are known as human carcinogen and their critical targets are kidney and liver for general population (Judilyn *et al.* , 2013).

V.CONCLUSION AND RECOMMENDATION

A. Conclusion

After conducting the study, it showed that high number of selected spaghetti samples exceeded the standard limits set for lead (Pb) and cadmium (Cd).

Out of ten selected spaghetti samples (both domestic and foreign) a high number of them are contaminated with heavy metals (lead and cadmium) , even though, some of selected samples were found below the detection limit of the instrument ; other heavy metals are still present and may exceed the standard limit set. Heavy metals (lead, cadmium) contaminants have a potential to bioaccumulate inside the human body. Therefore, consumption of such spaghetti should be monitored to avoid the adverse effects brought about by metals (lead and cadmium).

B. Recommendations

Due to high level of contamination by heavy metals (Pb and Cd) on selected spaghetti samples (both domestic and foreign) obtained from this study, I suggest that;

i. Further investigation on the concentration of other metals such as Arsenic (As), Manganese (Mn), Chromium (Cr), Mercury (Hg), Iron (Fe), etc, be carried out.
ii. Further investigation to determine the adulterants of the samples.
iii. Consumers of these spaghetti samples products should minimize and monitored its intakes to avoid the possible adverse effect that can be brought by the toxicants (lead andcadmium) in the samples.

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APPENDICE

APPENDIX 1: RESULT OF AAS ANALYSIS OF THE SAMPLES

TABLE 1: Concentration of lead (Pb) in Samples

SAMPLES	ABSORBANCE	CONC (mg/kg)	SD
Golden penny	0.001	0.889	0.0001
Power pasta	0.002	2.000	0.0000
Honey well	0.001	0.889	0.0001
Dangote standard	0.001	0.889	0.0001
Dangote slim	-0.000	ND	0.0000
Aicha	-0.000	ND	0.0003
Delicia	0.001	0.889	0.0003
Naama	0.000	ND	0.0000
Marina	0.002	2.000	0.0001
Oriba	0.000	ND	0.0001

TABLE 1.1: Concentration 0f Cadmium (Cd) in Samples

SAMPLES	ABSORBANCE	CONC (mg/kg)	SD
Golden penny	0.027	4.066	0.0002
Power pasta	0.000	ND	0.0000
Honey well	0.003	0.131	0.0017
Dangote standard	0.006	0.623	0.0001
Dangote slim	0.005	0.459	0.0001
Aicha	0.006	0.623	0.0000
Delicia	0.001	ND	0.0001
Naama	0.007	0.787	0.0001
Marina	0.000	ND	0.0001
Oriba	0.018	2.590	0.0005

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Appendix 2: Chart showing mean concentrations of Pb and Cd in Spaghetti

Figure 2: Chart showing Pb and Cd