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An Assessment to Determine the Level of Some Selected Heavy Metals in Rice (*Oryza Sativa*) Consumed Within Funtua Metropolis

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ABSTRACT

Consumption of foodstuffs is the most likely route for human exposure to heavy metals. This study was designed to investigate the toxic metals (cadmium (Cd), lead (Pb), chromium (Cr), and nickel(Ni)) concentrations in rice. The concentrations of these heavy metals were determined from twelve samples of rice (*oryza sativa*), the samples comprise of six foreign rice samples and six local samples collected from different markets within Funtua Local Government Area in 2019. Concentrations of the heavy metals in the rice samples were analyzed using atomic absorption spectroscopy and were found to be in the order Cr > Pb > Ni > Cd. The highest concentration of cadmium was detected in LR1(0.620mg/kg) and LR5(0.626mg/kg) while the lowest was detected in FR5(0.058mg/kg). The lowest concentration of nickel was detected in in FR6(0.060mg/kg) and LR2(0.090mg/kg) while the highest was detected in LR3(0.910mg/kg) and LR4(0.750mg/kg). The highest concentration of lead was detected in FR6(0.650mg/kg) and LR1(0.580mg/kg) while the lowest was detected in LR2(0.199mg/kg) and LR5(0.212mg/kg). The highest concentration of chromium was detected in LR1(0.840mg/kg), LR4(0.840mg/kg) and FR5(0.760mg/kg) while the lowest was detected in LR6(0.050mg/kg) and FR2(0.080mg/kg). The analysis shows that chromium, nickel, and lead concentrations that accumulated almost all the rice samples exceeded the maximum permissible limits prescribed by the World Health Organization (WHO) guideline as well as the Food and Agricultural Organization (FAO) standard. Cadmium concentrations were found in almost all the samples to conform with the standards. The higher levels of heavy metals accumulated by the samples may be attributed to the increase in agricultural activities such as the application of phosphate fertilizer, sewage sludge disposal and other anthropogenic activities which deserve further investigations.

keywords: heavy metals; foodstuff; consumption;

1. Introduction

Heavy metal pollution of soil affects the quality of the environment leading to serious consequences. Heavy metals group includes Ag, Ba, Cd, Co, Cr, Mn, Hg, Mo, Ni, Pb, Cu, Sn, Tl, V, Zn, and some metalloids such as As, Sb, Bi, and Se. Arsenic, for example, is often considered as a heavy metal due to the similarity of its chemical properties and behavior with the other heavy metals [1]. Heavy metals accumulation in soil, and in the environment in general, may be related to the phenomenon of bioaccumulation ability of living organisms, that is, increasing the concentration at human organism due to industrial activities and the food chain. The main sources of heavy-metal pollution in soil are irrigation, especially with sewage; solid-waste disposal, for example, sludge and compost refuse; the use of pesticides and fertilizers; and atmospheric deposition [1].

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Plants acquire the necessary nutrients, such as N, P, and K, from the environment. However, they may also accumulate unnecessary and toxic metals, such as Pb and Cd. Several plants have the ability to accumulate high metal concentrations [2]. Many studies have reported data for the transfer of heavy metals from soil to plants and vegetables through roots and shoot [3]. Therefore, toxic metals such as As, Cd, and Pb can be taken up from cereal crops and transferred to their grains [4]. Toxic metals may be classified according to their capability of being transferred from soil to plants in mobile metals, such as Cd, and poorly mobile metals, such as Pb. This property may affect their bioaccumulation in plants [5].

Rice is one of the most important and widespread cereals in the world. It is the staff of life for 3 billion people, mainly in Asia [2-4], contributing over 70% of the energy and 50% of the protein provided by their daily food intake. In the last report of EU Commission, India was presented as the first country for rice production with 19% of export. India is followed by Cambodia and Thailand. United States and Vietnam are the countries with less amount of imported rice [7]. About 80% of the rice production of Europe takes place in Italy and Spain, with a further 12% in Greece and Portugal.

On this basis the presence of toxic heavy metals in rice, which may raise the metal daily intake, should be strongly avoided in order to prevent negative effects on human health. The following elements are considered macronutrients in rice and their content is usually in some %: P, S, K, Ca, and Mg [5].

Other elements, like Mn, Fe, Cu, Zn, Se, and Ni, are classified as micronutrients and they are present in lower amount, while As, Cr, Pb, and Cd are undesirable elements because of their toxic effects even in very low quantity [6, 7]. As a consequence, it is crucially necessary to reduce possible accumulation effects in rice grains from the environment for safe food production. Great efforts are necessary to remediate polluted sites. Other approaches could be developed to reduce metals accumulation in edible parts of plants. For instance, favorable agronomic practices and chemical regulators may decrease plant heavy metals uptake [8].

2. Results and Discussion

The results of the AAS analysis for the concentrations of cadmium, chromium, lead and nickel in some foreign and local samples of rice compared with FAO/WHO permissible limits are shown in figure 1 and 2 below respectively. The result of ANOVA is also presented in table 1 below. Furthermore, table 1 shows the multiple comparisons of the heavy metals concentration in all the samples to determine the significant difference between them as statistically analyzed using statistical package for social scientist(SPSS).

Figure 1 and **figure 2** shows a bar chart representing the concentrations of Cr, Pb, Ni and Cd compared with FAO/WHO standard in foreign and local samples of rice respectively. The results of the AAS analysis of the foreign samples as shown in **figure 1** shows that lead (Pb) have the highest concentrations in FR2, FR3 and FR4 with0.433, 0.340 and 0.650 mg/kg respectively than other heavy metals in these samples. The concentrations in FR1, FR4 and FR5 are 0.283, 0.264 and 0.463 mg/kg respectively. The concentrations of Pb in local samples are shown in figure 4.2 for LR1, LR2, LR3, LR4, LR5 and LR6 as 0.580, 0.199, 0.304, 0.506, 0.212 and 0.353 mg/kg respectively. The order of the concentration of Pb in all the samples is FR6>LR1>LR4>FR5>FR2>LR6>FR3>LR3>FR1>FR4>LR5>LR2. The concentrations of Pb in both local and foreign samples compared with FAO/WHO permissible limit in rice shows that the concentrations are higher than the permissible limit except in LR2 and LR5, other samples are therefore contaminated with lead. The higher concentrations of Pb in these samples may be attributed to the soils where the samples were grown been contaminated with Pb. The soils where these samples were grown were contaminated with lead from either of the natural or anthropogenic sources mentioned.

Figure 1 also shows the concentrations of chromium in foreign samples FR1, FR2, FR3, FR4, FR5 and FR6 as 0.019, 0.080, 0.220, 0.350, 0.760 and 0.208 mg/kg respectively. The concentrations in the local samples are shown in figure 4.2 for LR1, LR2, LR3, LR4, LR5 and LR6 as 0.840, 0.810, 0.124, 0.840, 0.250, 0.05 mg/kg respectively. The Cr concentration was found to be highest in FR1, FR2, LR3, LR4 and LR1. The order of the concentration in all the samples is LR1=LR4>LR2>FR5>FR4>LR5>FR6>LR3>FR3>FR1>FR2>LR6. These concentrations are found to be higher than the permissible limits of FAO/WHO and the samples are said to be contaminated. Chromium in the environment particularly in the soil is attributed to natural and anthropogenic sources. The main natural source of Cr in soil is from weathering of their parent materials, fallout and washout of atmospheric Cr-containing particles [9]. Anthropogenic sources include chrome-bearing sludge, refuse from industrial activities, discarded manufactured products and coal ashes. The availability of Cr in these samples may be attributed to the soil where the samples were grown been contaminated by Cr from either of the natural or anthropogenic sources.

The concentrations of cadmium are also presented in **figure 1** and **2** for foreign and local samples respectively. Cadmium concentration was found to be almost constant in foreign samples as shown in figure 1. The concentrations for FR1, FR2, FR3, FR4, FR5 and FR6 as determined from AAS are 0.061, 0.060, 0.061, 0.061, 0.058 and 0.060 mg/kg respectively. Similar concentrations were also found for LR1, LR2, LR3, LR4, LR5 and LR6 as 0.620,0.061, 0.062, 0.061, 0.626 and 0.061 mg/kg respectively. The concentrations of Cd in both local and foreign samples compared with FAO/WHO permissible limit in rice shows that only LR1(0.620mg/kg) and LR5(0.626mg/kg) are above the permissible limit, other samples are below the permissible limit and are therefore not contaminated with cadmium. Cadmium availability is usually attributed to anthropogenic sources, the two major sources of contamination are the production and utilization of Cd and the disposal of wastes containing Cd. Increase in soil cadmium content will result in an increase in uptake by plants from the soil and this is usually greater at low soil pH.

Figure 1 and **2** also shows the concentrations of Nickel in foreign and local samples respectively, the permissible limit of Ni is also shown in each figure. The concentrations in foreign samples for FR1, FR2, FR3, FR4, FR5and FR6 were determined from AAS as 0.450, 0.190, 0.330, 0.129, 0.120 and 0.690 mg/kg respectively. The corresponding concentrations in local samples for LR1, LR2, LR3, LR4, LR5 and LR6 were found to be 0.300, 0.090, 0.910, 0.750, 0,440 and 0.410 respectively. The order of the concentration in all the samples follows that LR3>LR4>FR6>FR1>LR5>LR6>FR3>LR1>FR2>FR4>FR5>LR2. These concentrations except for LR2(0.09mg/kg), FR5(0.12mg/kg) and FR4(0.129mg/kg) are found to be higher than the permissible limits of FAO/WHO and the samples are said to be contaminated with nickel. LR2, FR5 and FR5 conforms with the standard and are free from nickel contamination. Nickel in the environment particularly in the soil is attributed to natural and anthropogenic sources. The main anthropogenic sources are usually from coal combustion, sewage sludge disposal, mining and its greater portion remains in the ash. Globally, about 60,000 tons of nickel may be generated from coal combustion per year.

Table 1 shows the mean concentrations and standard deviations of the heavy metals. The Table shows the multiple comparisons of the concentrations in all the samples from the breakdown of ANOVA result. The analysis compares each sample for example **FR1** and all other samples for a certain metal. The result shows that there is no significant difference between **FR1** ^{c}(0.119 mg/kg) and **LR3** ^{c}(0.124mg/kg) for chromium as shown with subscript later c but are significantly different from other samples, the result also shows no significant difference between **LR1** $^{\mathbf{d}}$(0.840mg/kg) and LR4 ^{d}(0.840mg/kg) for the same metal as shown with subscript later d but are significantly different from other samples, and all those with different or no subscript are significantly different.

Table 1. shows the multiple comparisons of each heavy metal concentrations (mg/kg) in all the samples as statistically analyzed using SPSS.

Sample	Cr	Pb	Ni	Cd
FR1	^{c}0.119 ± 0.0004	0.283 ± 0.0004	0.450 ± 0.0015	^{b}0.061 ± 0.0006
FR2	0.080 ± 0.0007	0.433 ± 0.0008	0.190 ± 0.0004	^{b}0.060 ± 0.0008
FR3	0.220 ± 0.0001	0.340 ± 0.0005	0.330 ± 0.0020	^{b}0.061 ± 0.0023
FR4	0.350 ± 0.0004	0.264 ± 0.0005	0.129 ± 0.0010	^{b}0.0613 ± 0.0016
FR5	0.760 ± 0.0002	0.463 ± 0.0003	0.120 ± 0.0023	^{b}0.058 ± 0.0011
FR6	0.208 ± 0.0005	0.650 ± 0.0002	0.690 ± 0.0016	^{b}0.690 ± 0.0016
LR1	^{d}0.840 ± 0.0002	0.580 ± 0.0001	0.300 ± 0.0029	^{a}0.620 ± 0.0006
LR2	0.810 ± 0.0001	0.199 ± 0.0002	0.090 ± 0.0018	^{b}0.061 ± 0.0017
LR3	^{c}0.124 ± 0.0006	0.304 ± 0.0005	0.910 ± 0.0008	^{b}0.062 ± 0.0017
LR4	^{d}0.840 ± 0.0001	0.506 ± 0.0003	0.750 ± 0.0017	^{b}0.061 ± 0.0019
LR5	0.250 ± 0.0004	0.212 ± 0.0009	0.440 ± 0.0013	^{a}0.626 ± 0.0027
LR6	0.050 ± 0.0002	0.353 ± 0.0008	0.410 ± 0.0006	^{b}0.061 ± 0.0005

* The samples with the same superscript later means that there is no significant difference between them and the mean difference is significant at the 0.05 level or 95% confidence.

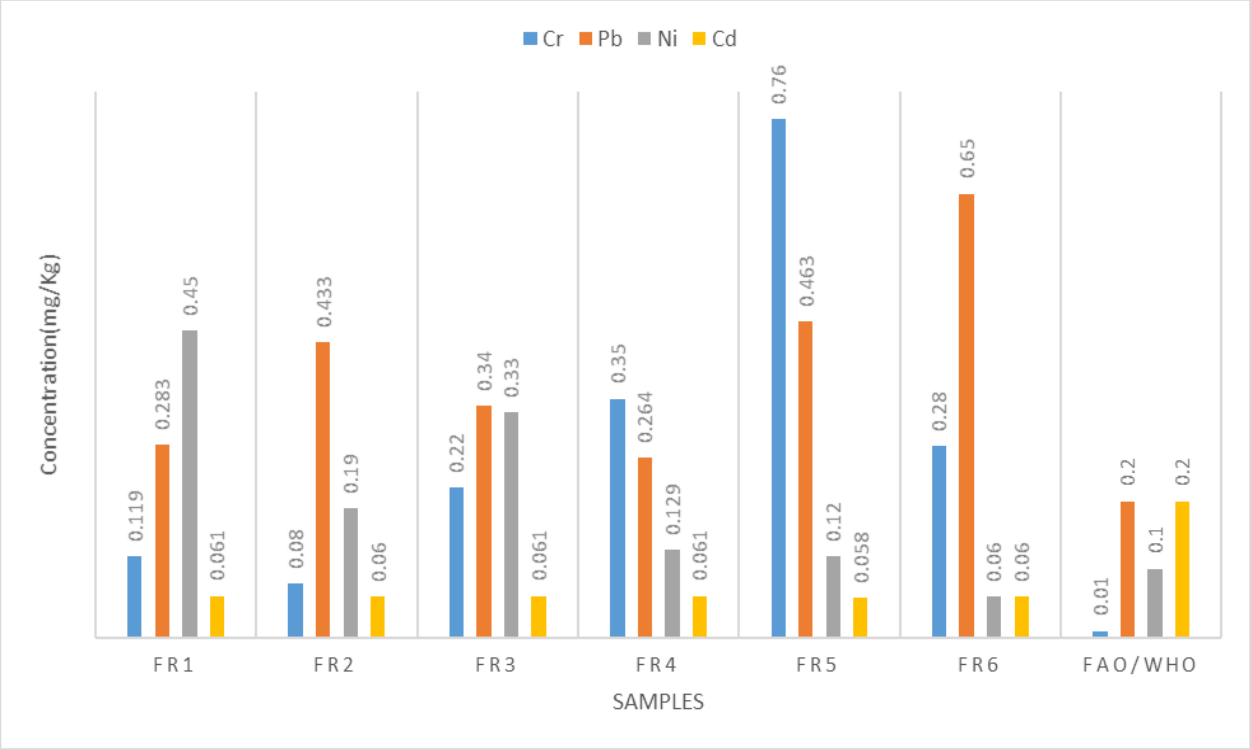

Figure 1. shows a bar chart representing the concentrations of Cr, Pb, Ni, and Cd in foreign samples of rice.

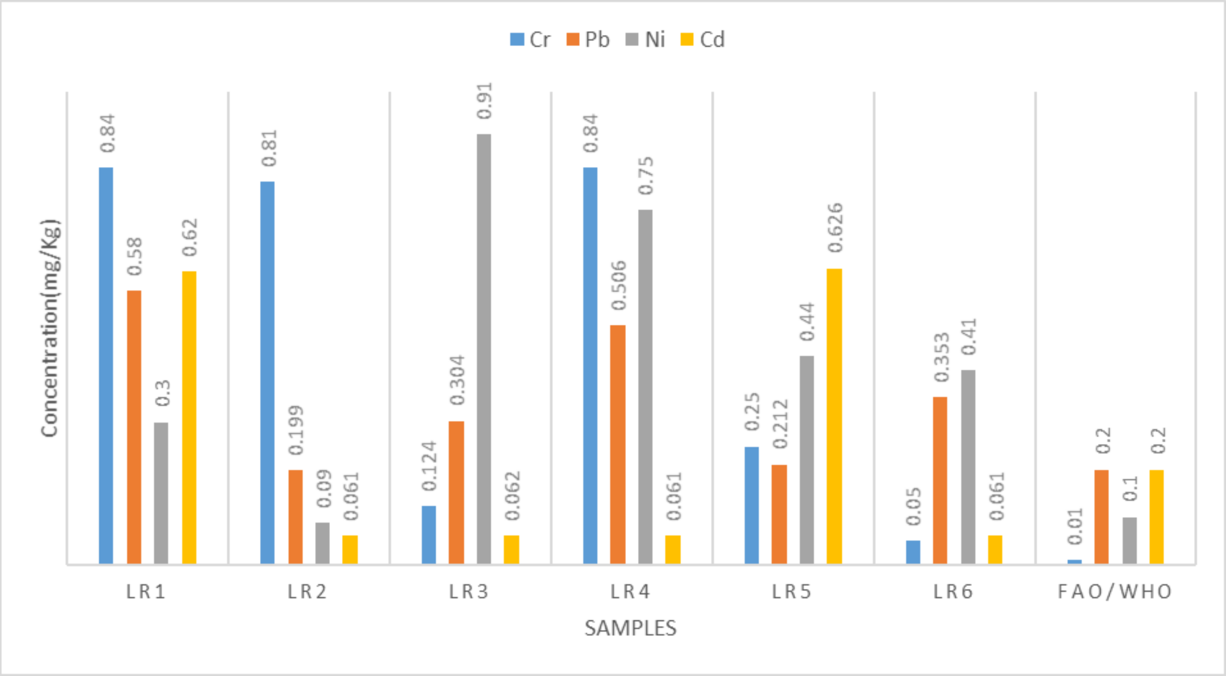

Figure 2. shows a bar chart representing the concentrations of Cr, Pb, Ni, and Cd in local samples of rice compared with FAO/WHO standard.

Figure 1. Structure of novel tris-(bis coumarinyl)methane **3l.**

For lead concentrations, the result shows that all the samples are significantly different as no subscript is shown any concentration. This is the same for nickel concentration, all the samples are significantly different. For cadmium, **LR1**^{a}(0.620mg/kg) and LR5^a(0.626mg/kg) have the same subscript later a implying that there is no significant difference between them, all the remaining samples have the same subscript later b showing that there is no significant difference between them but are significantly different from **LR1** and **LR5**.

3. Experimental

3.1. Study area

This research was carried out with twelve different samples comprising of six local and six foreign rice collected from three different markets within Funtua local government area of katsina state. The markets are; Jabiri (Dandume road), Kasuwar mata (along Sokoto road), and Central market (along Katsina road). Funtua is a town situated at 11^o North on the latitude, 7.31^o East on the longitude, and 612 meters above the sea level with a population of about 136,811 (**figure 3**)

Shown on next page is the map of Funtua local government area showing the location of the three markets where the samples were collected.

3.2 Materials

Apparatus

The following apparatus were used in order to perform the project work. Hot plate, volumetric flask, conical flask, mortar and pestle, filter paper, masking tape, measuring cylinder, porcelain crucible, funnel, stirring rod.

Chemicals

All chemicals of high purity analytical grade reagents were employed; HNO$_3$ (65% w/w) and HCl (35% w/w). Distilled water was used throughout the experiment to prepare all the solutions.

Instrumentation

Digital analytical balance was used for all measurements of samples and chemicals. AAS was used to determine the concentrations of Cd, Pb, Cr, and Ni [10, 11].

3.3 Sample Collection

Six samples of local rice (LR1-LR6) in **table 2** and six samples of foreign rice (FR1-FR6) in **table 3** were randomly collected from different sellers in three different markets within Funtua local government area of Katsina state. Two samples of local and foreign rice were collected from each of Jabiri, Kasuwar mata, and Central market and were labelled accordingly and kept in a clean container to avoid contamination before taken for analysis [8].

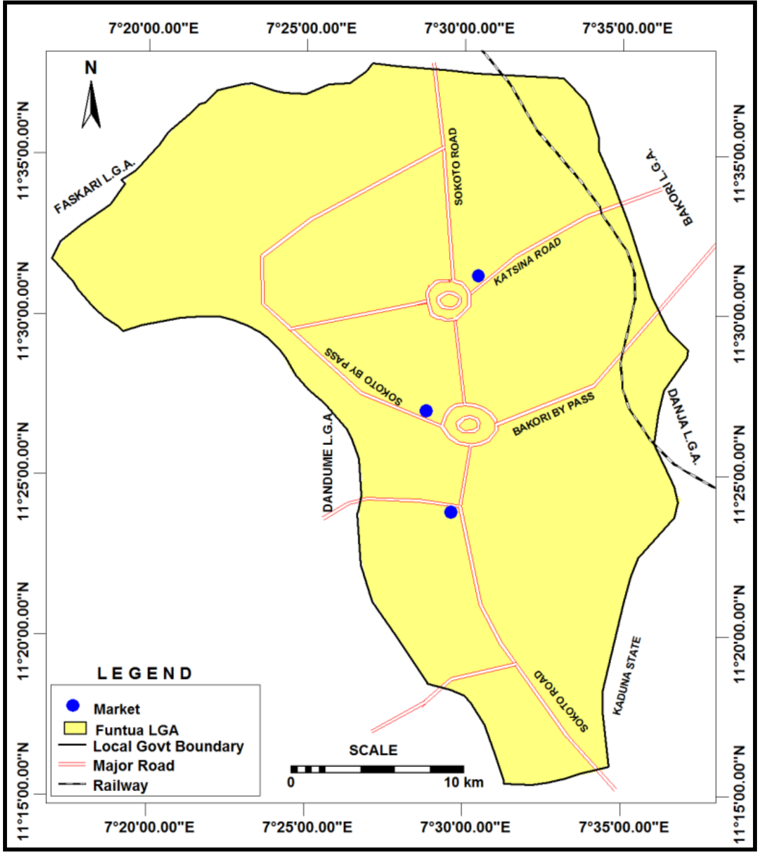

Figure 3: Map of Funtua local government area showing the sampling market

The details of the samples collected are summarized in the tables below;

Table 2. Local samples

Sample code	Source
LR1	Kasuwar mata
LR2	Kasuwar mata
LR3	Central market
LR4	Central market
LR5	Jabiri Market
LR6	Jabiri Market

Table 3. Foreign samples

Name	Batch Number	Sample code	Source
Tomato aroso	320	Kasuwar mata	FR1
White lily	201	Kasuwar mata	FR2
Oriba	319	Jabiri Market	FR3
Marigold	1025	Jabiri Market	FR4

Gugu	248	Central market	FR5
Fresh Thailand	192	Central market	FR6

3.4 Sample Digestion

The sample was dried, manually grounded with a pestle and mortar and labelled accordingly. Approximately 1.0g of the grounded over dried sample was weighed into a porcelain crucible. 9.00 cm^3 of HCl and 3.00 cm^3 of HNO_3 in 3:1 were added and swirled gently. The mixture was heated on a hotplate in a fume cupboard for about 30 minutes at moderate temperature which was later increase to about 80^{o}C until a clear solution was observed while adding distilled water until the appearance of white fume which shows complete digestion. The solution was set aside to cool to room temperature and was filtered into a 50cm^3 volumetric flask and made up to volume with distilled water. This was then stored in a polyethene bottle and labelled accordingly before taken for analysis. Three digestions were made for each sample and the same procedure was repeated for all the samples [12-14].

The mixed acid digestion procedure was chosen over the dry ashing process due to the following reasons;

1. No volatilization loses of metallic elements.
2. The procedure is fairly rapid and is easily adapted for routine use.
3. A large number of nutrients elements can be determined in one digest solution.

3.5 Sample analysis

The method of analysis used in analyzing the samples is the flame atomic absorption spectroscopy. Atomic absorption along with atomic emission spectroscopy was first used by Guystav Kirchoff and Robert Bunsen in 1859 and 1860 [8]. Modern atomic absorption spectroscopy was introduced in 1955 as a result of independent work of A. Walsh and C. T. J. Alkemade.

The samples were analyzed at the Institute of Agricultural Research (IAR) Laboratory of ABU Zaria. The concentrations of Cr, Cd, Pb, and Ni were determined using the Flame Atomic Absorption Spectrometer AA500FS model with high resolution.

The sample was aspirated into the spray chamber of the instrument by passing a high-pressure stream consisting of the combustion gases (air-acetylene). The sample was first converted into a fine mist consisting of small droplets of solution in the nebulizer chamber, an aerosol mist produced by the impact of the sample with the glass impact bead mixes with the combustion gases in the spray chamber before passing to the burner. The flame's thermal energy in the burner desolvated the aerosol mist into a dry aerosol of small solid particles. Subsequently, the thermal energy of the flame volatilizes the particles and produced a vapour consisting of molecular species, ionic species, and free atoms. The thermal energy in the flame was provided by the combustion of the fuel-oxidant mixture used.

The source for the atomic absorption used is the hollow cathode lamp. This consist of a cathode made from the metallic analyte and an anode enclosed within a glass tube filled with a low pressure gas (Ne or Ar), a potential was applied across the electrodes and the filler gas was ionized, the positively charged ions collided with the negatively charged cathode and dislodged some atoms from the surface of the cathode. Some of these sputtered atoms are in the ground state and hence absorb radiation. Since the cathode was designed from the metallic analyte, the hollow cathode lamp provided the absorption lines that corresponded to the analyte's absorption spectrum.

The decrease in the intensity (absorbance) of the light was measured with a photochemical detector and the concentrations of the elements were determined from the calibration curves of the standards used [15-17]. The operating parameters used for the analysis are shown in **Table 4** below

Table 4. Operating Parameters used for the AAS analysis

Elements	Wavelength(nm)	Slit Width(nm)	Flame
Cadmium	228.8	0.4	Air-acetylene
Chromium	357.90	0.4	Air-acetylene
Lead	283.30	0.4	Air-acetylene
Nickel	232.00	0.2	Air-acetylene

3.6 Statistical analysis

The analysis of variance (ANOVA) was determined using the statistical package for social scientists (SPSS) Table 1 shows the analysis of variance comparing the mean concentration of the heavy metals determined in all the samples [18-21]. The analysis of variance was analyzed using SPSS at 95% confidence or probability level of 0.05 in order to determine the significant difference between the heavy metal concentrations in the samples. The results show that there are significant differences in most of the samples, this means that even though some concentrations appears to be comparable, statistical analysis have shown that they are significantly different.

4. Conclusion

The concentrations of heavy metals detected in the rice samples were quite variable. Pb, Cr, and Ni found in the rice samples were above the maximum permissible limits as per the WHO/FAO guideline standards comparison. Cd was found to conform with the standard in almost all the samples. High concentrations of some heavy metals analyzed in the samples grown and sold within the area were attributed to probable high influx of metals as a result of pollution from the Agriculture activities, Municipal and industrial wastes, thereby increasing the potential bioavailability to the plants and also posing the associated risks of affecting the quality human health, particularly the most populous consumers in the long run.

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