

*Original Research Article*

## Determination of Heavy Metals in Some Selected Pastas in Nasarawa State Markets

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### ABSTRACT

Pasta is a common food product created from wheat flour combined with water or eggs, shaped into various forms, and cooked by boiling or baking. The primary route for heavy metals to enter the human body is through food consumption. This study aims to assess the presence of heavy metals in pasta products. We conducted an analysis using atomic absorption spectroscopy (AAS) to determine the concentrations of manganese, cadmium, copper, chromium, lead, and zinc. The results of our heavy metal analysis revealed the presence of significant concentrations of these elements in four distinct pasta types: HWS, GPS, DS, and CS. Notably, CS exhibited elevated levels of nickel (Ni) contamination, while HWS contained a high concentration of cadmium (Cd). These findings suggest that manufacturing processes employed in the industry may be responsible for the contamination of these heavy metals in pasta products. Furthermore, it is concerning that HWS and CS still contain concentrations of Ni and Cd above the maximum permissible limits recommended by the World Health Organization (WHO). Considering the limits of blank (LOB), limits of detection (LOD), repeatability (R), and reproducibility (Rp) presented in this study, we observed minimal variability or uncertainty in the measurements. This suggests a notable level of precision in the measuring instruments and the tested samples. Consequently, the pasta products investigated in this area can be considered safe for consumption. Nevertheless, we recommend continued research of this nature in other regions of the state to ensure the safety of pasta products across the entire area.

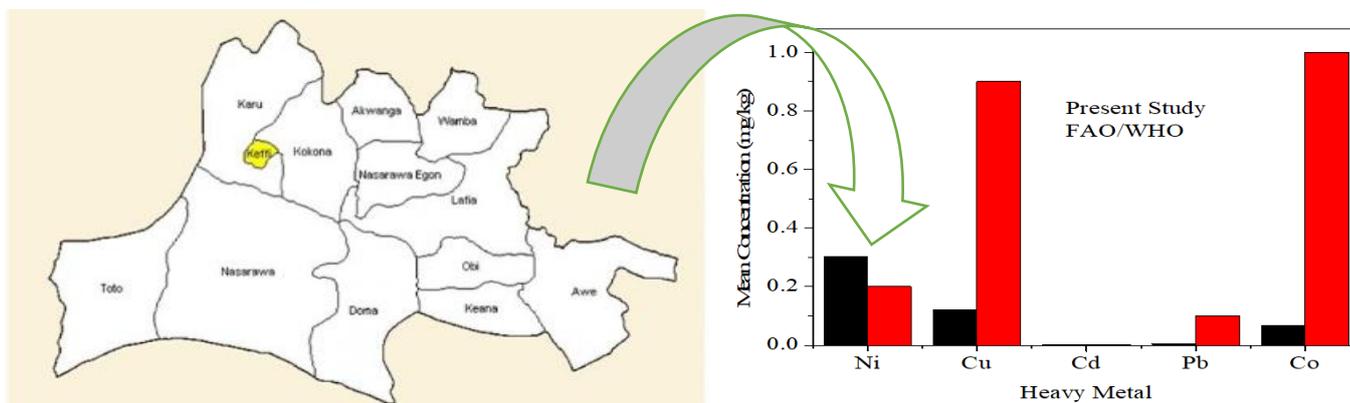
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## GRAPHICAL ABSTRACT



## 1- Introduction

Pastas are types of food made of dough of wheat flour mixed with water or egg and extruded into an infinite variety of shapes. The ingredients can be durum wheat and water, soft wheat and egg or different combinations of other flours with egg or water or a combination of both [1]. Pastas captures a substantial portion in human nutrition because of its vitamin, carbohydrate and mineral contents [2].

Concentration of heavy metals found in food products are continuously need to be monitored for safety and quality assurance. Reporting information about the content of these in commercially available noodles, pastas is vital to evaluate the possible health risk related with their consumption [3]. Charles, 2018 [4] reported that the equipment used in processing food, the containers used in storing the food, other things used as additives to the food and materials used for the packaging of the food are perhaps the possible route in which noodles products gets contaminated with heavy metals [5].

Products of cereal like wheat flour, wheat itself, bread, pasta and cereal itself are part the biggest group in food chain. These group of foods are being consumed on daily bases and can affect human health directly [6]. In their normal form, cereals are rich in source of vitamins, minerals,

fats, oils, carbohydrates and protein. Since heavy metals can be transferred to the crops through soil, a cereal can be polluted directly through the agricultural soil [7]. Different processes like commercial fertilizers application, emissions from vehicles, wastes from industries and emissions from industries can cause heavy metals contamination in soil [8]. This contaminated soil pollutes the crops and human beings gets them through either ingestion, inhalation, dermal contact, oral intake or diet food chain [9]. Heavy metals do not degrade in biological tissue but rather accumulate in the tissues via food chain posing severe health and environmental problems [10]. The details of these potential health risks are presented in Table 1 as reported by Rilwan et al. (2020f) [11].

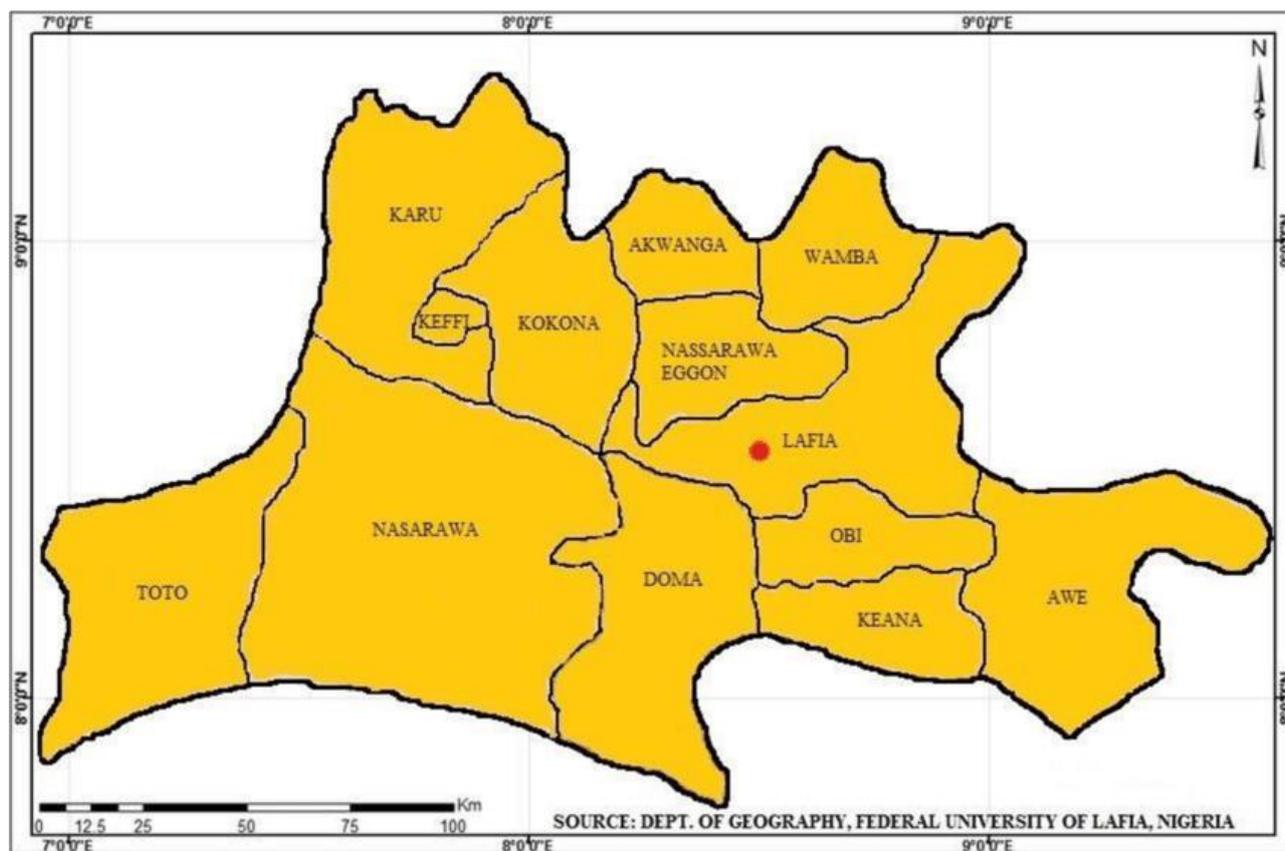
This study covers random sampling of five different brands of pastas from different vendors in Nasarawa State Market. Much works on proximate analysis of pasta, determination of micronutrients in pasta, shelf life determination of pasta, etc. have been documented but not much on determination of heavy metals in pastas are being reported.

**Table 1-** Potential Health Effects of Excess Exposure

| Metals | Potential Health Effects of Excess Exposure   |
|--------|---|
| Ni     | Dermatitis, respiratory issues, lung and nasal cancer, gastrointestinal effects, organ and skin allergic reactions, kidney and liver damage, neurological effects   |
| Cu     | Gastrointestinal symptoms, liver damage, kidney damage, neurological effects, hemolysis, Wilson's disease.  |
| Cd     | Kidney damage, bone health, respiratory problems, lungs cancer, cardiovascular effects, gastrointestinal effects, reproductive and developmental effects, neurological effects.   |
| Pb     | Neurological effects, hematological effects, renal effects, cardiovascular effects, reproductive and developmental effects, gastrointestinal effects, bone health, immunological effects, behavioral and emotional effects, developmental delays, dental effects. |
| Co     | Hypothyroidism, cardiovascular effects, neurological effects, gastrointestinal effects, thyroid gland enlargement, hematological effects.   |

The work was carried out in Nasarawa West, Nasarawa South and Nasarawa North, district of Nasarawa State, Nigeria. Karu is a town in Nasarawa West close to Federal Capital Territory, its geographical coordinates are 8°99'28.44" North, 7°57'25.74" East. It has 2,640 km<sup>2</sup> area. It has a population of about 205,477 people [12]. Lafia, being the largest town in Nasarawa state and Nasarawa state capital, is a town in Nasarawa

South, its geographical coordinates are North, 8°30'55.15"East, 8°50'47.44" and has a population of 348,000 based on the census that took place in 2006 [13]. Akwanga is a town in Nasarawa North, its geographical coordinates are,8°54'23.98" North, 8°24'26.84" East. It has an area of 996 km<sup>2</sup> and a population of 93,780 at the 2006 census. These locations were selected to determine the concentrations of heavy metals content of some selected pastas in the three regions of Nasarawa State. The map of the study location is presented in Figure 1. Karu modern market is one amongst the popular markets in Karu, L. G. A, located along Abuja-Keffi road. Food items, fabrics, clothes, meat, vegetables, plastics, cooking utensils, cosmetics, drinks and beverages are stocked in the market. It is highly patronized by the residents in the entire community. Lafia modern market is the largest market in Lafia, along Makurdi adjacent to romantic bakery by traffic light with varieties of commodities like electronics and electrical, food items, clothes and fabrics, traditional beads as well as a large bush meat section sold there at affordable prices. The market is very crowded and quite disorganized probably due to its super busy nature. Not only is there a persistent traffic grid lock around the market, it is also extremely dirty because of a smaller number of waste disposal system which lead to decaying of food and animals remains throughout the market. Akwanga modern market is the largest market in the L.G.A. located along Lafia, adjacent to Union bank. The modern market stocks varieties of items ranging from clothes, fabrics, food items, plastics, cooking utensils, vegetables, meat, cosmetics, drinks and beverages.



**Fig 1:** The Map of Nasarawa State

## 2 - Methodology

Samples of Pasta (Dangote, Honey-well, Golden-penny, Crown) were purchased from the aforementioned market and taken for analysis in the laboratory. The samples of soil obtained from various locations were properly blended to make sure homogeneity is attained. These samples were later heated for 12 hours in a 32L DIN-12880-Class-3.1 oven certified for temperature safety, setting a temperature of 80°C to get rid of the moisture. With the aid of molar, pestle and 2 mm sieve the resulting dried samples were made fine particles. Three grams (3 g) of these fine particulate soils were laid into a Pyrex flask and a 25 cm<sup>3</sup> of nitric acid (concentrated, 70% High Purity HNO<sub>3</sub>) (with specific gravity of 1.42 g/mL) against 75 cm<sup>3</sup> of hydrochloric acid (35% Hcl concentrated) (with specific gravity of 1.18 g/mL) been in the ratio of 1:3, was then added to

dissolve the metals. The resulting mixture was then laid on a VM-300S hot-plate (with maximum loading capacity of 50 mL and speed range of 300 rpm) and left to be cool. The obtained solution was later filtered via whatman filter paper no. 42 and filled up to a 50 mL mark with water (distilled). This was taken to Perkin Elmer Atomic Absorption Spectrophotometer Analyst 400 for metal ion content analysis. The AAS Analyst 400 model was utilized for the determination of heavy metal content in previously digested soil samples. The setup process involved fixing the nitrous oxide, acetylene gas, and compressor. The compressor was then activated, and the liquid trap was purged to eliminate any trapped liquid. Following this, both the Extractor and the AAS control were switched on. To ensure the precision of the analysis, meticulous cleaning procedures were performed. The slender tube and nebulizer piece were thoroughly cleansed

using a purifying wire, and the burner's opening was cleaned using an arrangement card. Subsequently, the AAS programming worksheet on the connected PC was opened, and the empty cathode light was inserted into the light holder. The light source was turned on, and the cathode beam was carefully adjusted to precisely target the arrangement card, ensuring optimal light throughput. Once this was achieved, the machine was ignited. In preparation for analysis, a fine amount of the sample was placed in a 10 ml graduated cylinder containing deionized water, and the aspiration rate was measured. An analytical blank was meticulously prepared, followed by the creation of a series of calibration solutions with known quantities of the analyte element (standards). These standards, along with the blank, were atomized sequentially, and their respective responses were recorded. Calibration curves were constructed for each standard solution, enabling the subsequent atomization and measurement of the sample solutions. Finally, the concentrations of various metals within the sample solution were determined by referencing the absorbance values obtained for the unknown sample against the calibration curves. This methodology allowed for the accurate quantification of heavy metal concentrations in the soil samples [14, 15]. The Limit of Blank (LOB), Limit of Detection (LOD), and Limit of Quantitation (LOQ) were evaluated which are important parameters in analytical chemistry, particularly in the context of analytical method validation. These parameters help determine the sensitivity and reliability of an analytical method as pointed out by David & Terry in 2018 [16]. The formulas for calculating these limits vary depending on the specific method and statistical approach used, but here are common approaches for each: Limit of Blank (LOB) as in Equation (1), represents the highest apparent analyte concentration that is expected to be indistinguishable from the background signal (blank) with a certain level of confidence.

It is typically calculated according to David & Terry (2018) [16] as:

$$LOB: \text{Mean signal of blank} + k \times (\text{Standard Deviation of blank}) \quad (1)$$

Limit of Detection (LOD) as in Equation (2), represents the lowest concentration of an analyte that can be reliably detected but not necessarily quantified. It is typically calculated according to David & Terry (2018) [16] as:

$$LOD: LOB + K \quad (2)$$

Where  $k$  is a constant that depends on the desired level of confidence. Common values for  $k$  include 1.645 for a 95% confidence level and 2.33 for a 99% confidence level when assuming a normal distribution. Repeatability and reproducibility are important measures of the precision or variability of an analytical method. These measures help assess how consistent the results are when the same analyst repeats the analysis (repeatability) or when different analysts or laboratories perform the analysis (reproducibility). They are often expressed as standard deviations or coefficients of variation. Here are the formulas for calculating repeatability and reproducibility: Repeatability (R) as in Equation (3), also known as intra-laboratory precision, assesses the precision of results obtained within the same laboratory by the same analyst or instrument on different days or under different conditions. It is typically calculated according to David & Terry (2018) [16] as the standard deviation (SD) or coefficient of variation (CV) of a series of replicate measurements on the same sample:

$$R: \sqrt{\left[ \frac{\sum (x_i - \bar{x})^2}{(n - 1)} \right]} \quad (3)$$

Reproducibility (Rp) as in Equation (4), also known as inter-laboratory precision, assesses the

precision of results obtained by different analysts or different laboratories using the same method. It is typically calculated similarly to repeatability according to David & Terry (2018) [16], but it involves measurements from multiple laboratories or analysts. The formula for reproducibility standard deviation (RSDR) is similar to repeatability:

$$R_p: \sqrt{\left[ \frac{\sum(x_i - \bar{x})^2}{(m - 1)} \right]} \quad (4)$$

Where  $x_i$  equals each individual measurement,  $\bar{x}$  equals the mean of the measurements  $n$  is the number of replicates and  $m$  is the number of laboratories or analysts. These formulas as reported by David & Terry (2018) [16] provide quantitative measures of the precision within a single laboratory (repeatability) and the precision between different laboratories or analysts (reproducibility). The choice of whether to use standard deviation or coefficient of variation depends on your preference and the reporting requirements of your analytical method validation or quality control procedures.

### 3- Results and Discussions

#### 3.1 Results

**Table 2:** Content of Heavy Metals in mg/kg.

| Heavy Metal | HWS    | GPS    | CS     | DS     | FAO/WHO | LOB    | LOD                    | R       | Rp      |
|-------------|--------|--------|--------|--------|---------|--------|------------------------|---------|---------|
| Ni          | 0.065  | 0.051  | 1.051  | 0.037  | 0.2000  | 0.0360 | 3.0 x10 <sup>-6</sup>  | 0.00224 | 0.00224 |
| Cu          | 0.089  | 0.128  | 0.133  | 0.139  | 0.9000  | 0.0800 | 3.0 x10 <sup>-8</sup>  | 0.00447 | 0.00447 |
| Cd          | 0.0053 | 0.0012 | 0.0006 | 0.0007 | 0.0025  | 0.0006 | 3.0 x10 <sup>-10</sup> | 0.00020 | 0.00020 |
| Pb          | 0.0045 | 0.0046 | ND     | 0.0054 | 0.1000  | 0.0045 | 9.0 x10 <sup>-9</sup>  | 0.00014 | 0.00014 |
| Co          | 0.1619 | ND     | 0.016  | 0.023  | 1.0000  | 0.0160 | 5.19 x10 <sup>-9</sup> | 0.00115 | 0.00100 |
| <b>pH</b>   | 3.78   | 4.81   | 5.69   | 4.58   | -       | -      | -                      | -       | -       |

ND = Not detected; HWS = Honey well spaghetti; GPS = Golden penny spaghetti; CS = Crown spaghetti; DS = Dangote spaghetti; LOB = Limit of blank; LOD = Limit of detection; R = Repeatability; Rp = Reproducibility; FAO = Food and Agricultural Organization; WHO = World Health Organization.

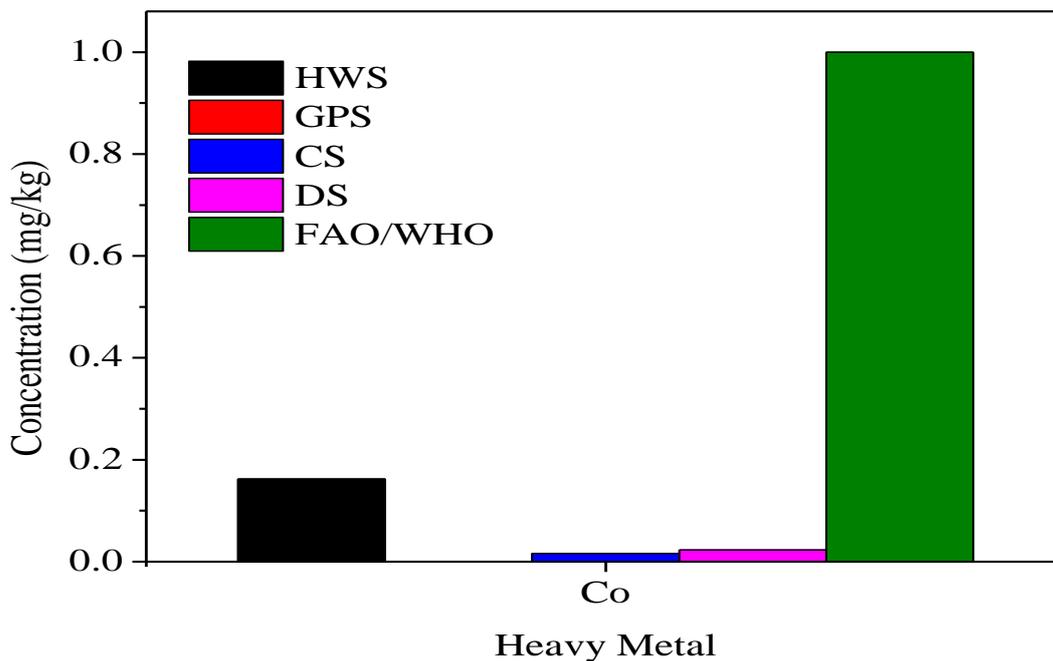


Fig. 2: Content of Co in Samples of Food (mg/kg)

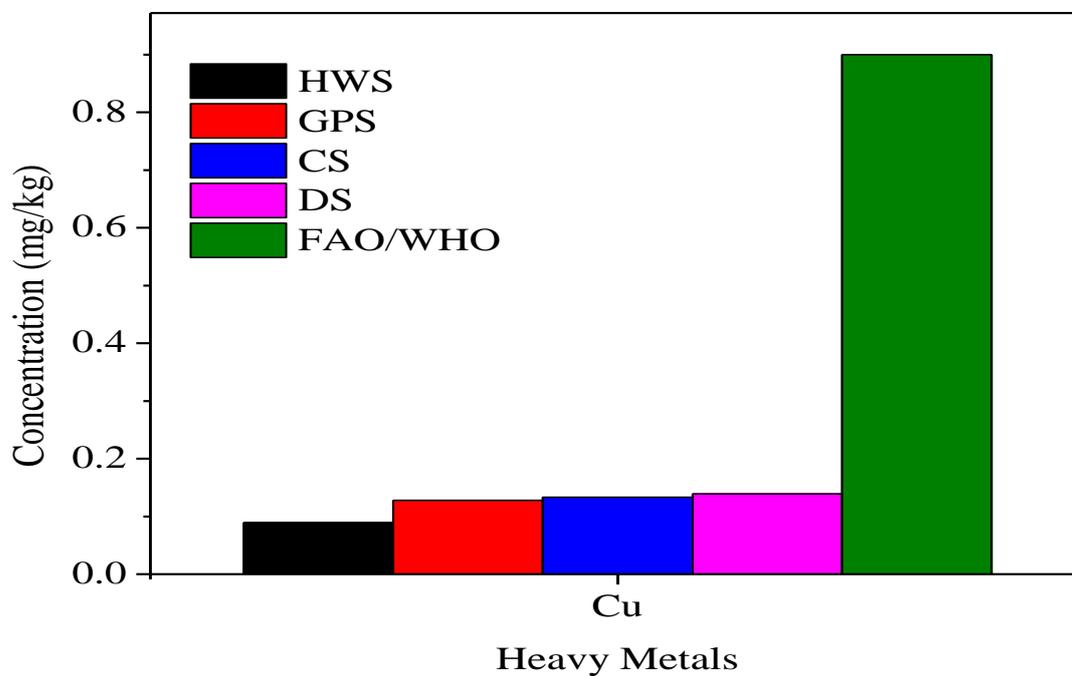


Fig. 3: Content of Cu in Samples of Food (mg/kg)

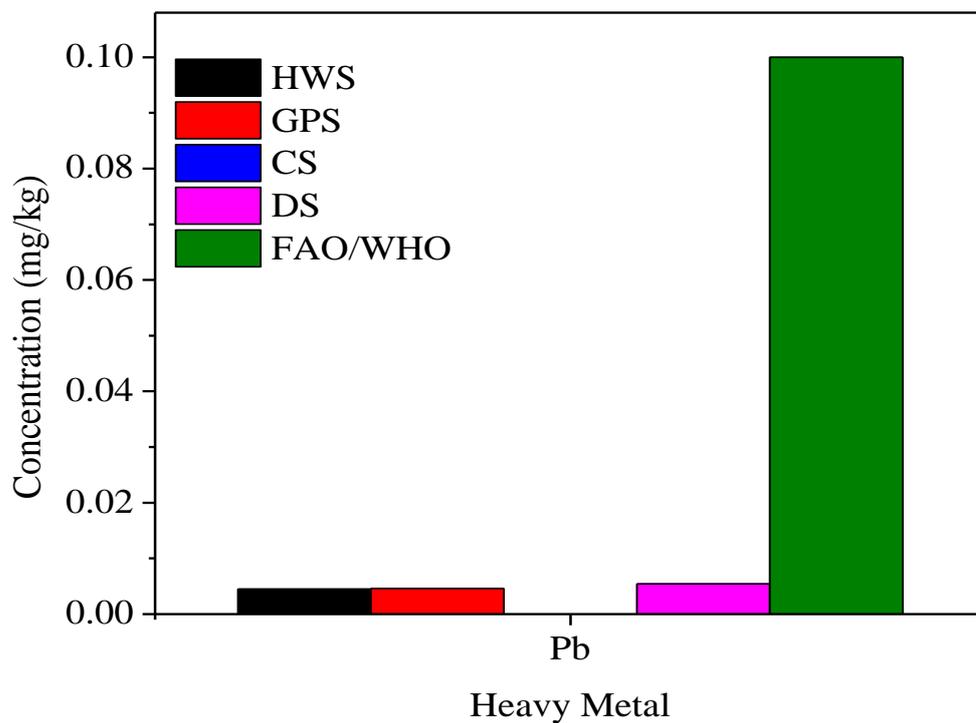


Fig. 4: Content of Pb in Samples of Food (mg/kg)

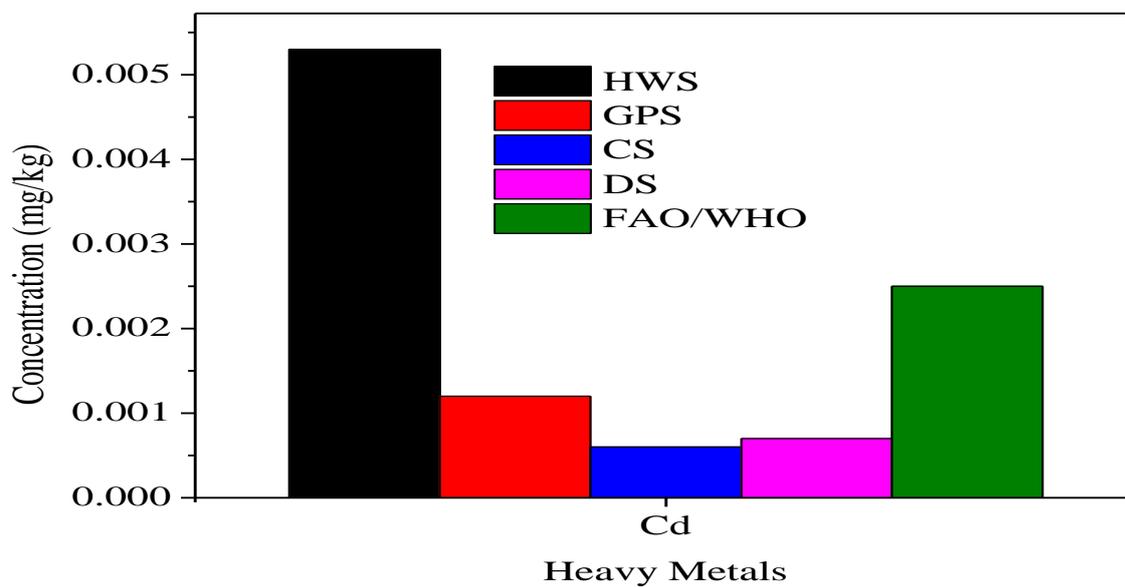
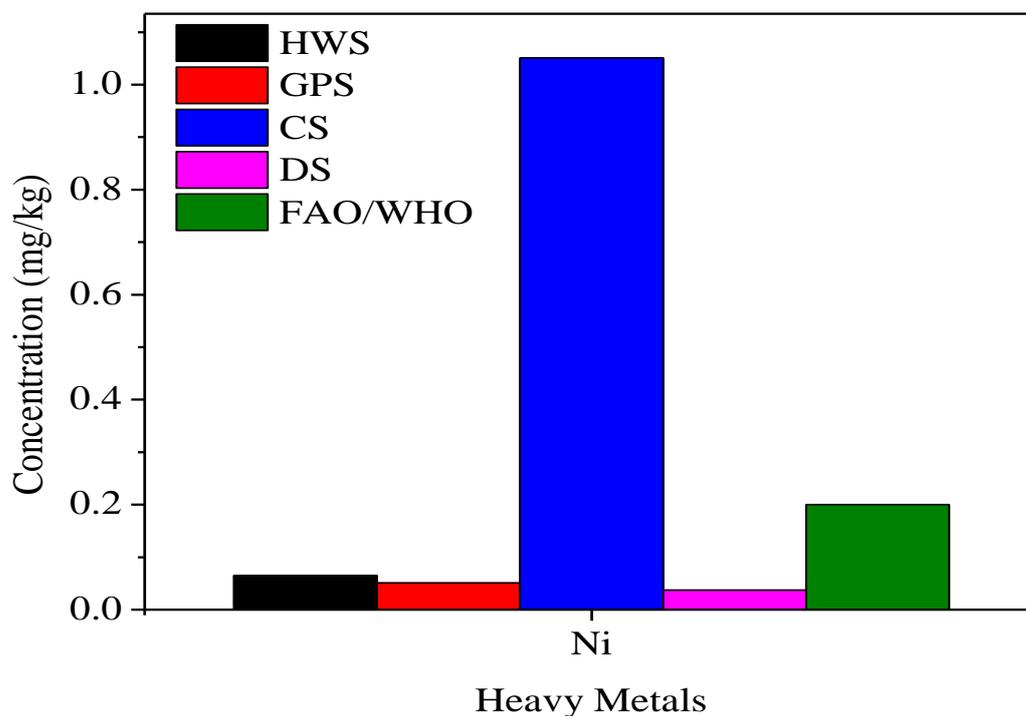


Fig. 5: Content of Cd in Samples of Food (mg/kg)



**Fig. 6:** Content of Ni in Samples of Food (mg/kg)

### 3.2 Discussion

Based on the results presented above, historical concerns regarding cobalt toxicity have primarily centered on respiratory issues among individuals involved in cobalt processing. However, in this case (Fig. 2), the cobalt content in HWS (0.1619 mg/kg), CS (0.016 mg/kg), and DS (0.023 mg/kg) falls within the permissible limits established by the FAO/WHO in 2016 reported by [17-22] for food products. Symptoms associated with gastrointestinal effects, such as cramps, abdominal pain, diarrhea, nausea, and vomiting, as well as liver damage, are often linked to excessive copper exposure. The Institute of Medicine's Food and Nutritional Board has reported that instances of non-hereditary copper homeostasis defects due to copper toxicity are exceedingly rare. Our analysis (Fig. 3) indicates that the copper levels in HWS (0.089 mg/kg), GPS

(0.051 mg/kg), CS (1.051 mg/kg), and DS (0.037 mg/kg) are all well below the permissible limits, presenting no risk. Lead poisoning, known as plumbism or saturnism, can result in severe health consequences, including seizures, anemia, coma, or even death [23-27]. Exposure to lead can occur through various routes, including water, dust, contaminated air, food, or consumer products. However, the lead content in HWS (0.0045 mg/kg), GPS (0.00046 mg/kg), and DS (0.0054 mg/kg) is below permissible limits, and CS (Nd) showed no trace of lead (Fig. 4). Cadmium is a prevalent heavy metal in the environment, but our analysis (Fig. 5) demonstrates no risk associated with GPS (0.0012 mg/kg), CS (0.0012 mg/kg), and DS (0.0007 mg/kg) samples, as they adhere to FAO/WHO standards. However, HWS (0.0053 mg/kg) exceeds the FAO/WHO limit, indicating potential risks, including weakened and brittle

bones, cancer, anemia, cough, and irreversible kidney failure, which can lead to fatality [28-32]. High nickel exposure in polluted environments can have various pathological effects on humans [33-36], including cardiovascular and kidney diseases, lung fibrosis, and respiratory tract cancer [37-39]. In our results (Fig. 6), HWS (0.065 mg/kg), GPS (0.051 mg/kg), and DS (0.037 mg/kg) exceed the FAO/WHO standards, indicating potential risks. Notably, CS (1.812 mg/kg) significantly surpasses the standard limit. The low values of LOB, LOD, R, and Rp in our study indicate minimal variability and uncertainty in measurements, signifying a high level of precision in both the measuring instruments and the tested samples.

#### 4 Conclusion

The current study has underscored the significant presence of heavy metals in HWS, GPS, DS, and CS pasta varieties. Notably, CS exhibited a notably high concentration of nickel (Ni), while HWS displayed elevated levels of cadmium (Cd). All samples prove acidic based on the pH values recorded. These findings raise concerns regarding the manufacturing processes employed in the pasta industry, which may be the source of heavy metal contamination in these products. Importantly, the risk of heavy metal intoxication, specifically Ni and Cd, remains unresolved, as both HWS and CS contained concentrations exceeding the maximum permissible limits recommended by the World Health Organization (WHO), Centers for Disease Control and Prevention (CDC), and the Food and Agriculture Organization (FAO). Assessing the analytical parameters, including the limits of blank (LOB), limits of detection (LOD), repeatability (R), and reproducibility (Rp) presented in this study, we observed minimal variability and uncertainty in our measurements. This suggests a commendable level of precision in our measuring instruments and sample analysis. Consequently, we can cautiously assert the safety

of pasta consumption within the investigated area. However, to ensure the broader safety of pasta products, we strongly recommend ongoing research of this nature in other regions of the state to encompass the entirety of our jurisdiction. This proactive approach will uphold public health and safety standards in pasta consumption across the state.

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