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Article

Quantitative Analysis of Heavy Metals (Pb, Cd, Cu, and Zn) in Senegalese Powdered Milk Using XRF Spectroscopy: Implications for Nuclear Analytical Techniques in Food Safety

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Abstract: This article conducts a quantitative analysis of heavy metals (Pb, Cd, Cu, and Zn) in Senegalese powdered milk using X-ray Fluorescence (XRF) spectroscopy, focusing on samples from the Senegalese market. Lead concentrations remained below the limits of detection, while copper and zinc exhibited variability, reaching maximum levels of 27.59 ppm and 81.71 ppm, respectively. Cadmium concentrations ranged from 9.96 ppm to 16.48 ppm. Health risk assessment, involving Daily Intake (DIM) and Reference Dose (RFD) calculations, revealed Health Risk Index (HRI) values below 1 for both adults and children, indicating acceptable health risks associated with powdered milk consumption. A comparison of HRI profiles between adults and children showed slightly higher risks for the latter. The study underscores the importance of rigorous quality control to address uncertainties in results and highlights XRF's significance in assessing metal concentrations for nuanced risk assessments. These findings provide valuable insights into potential health risks associated with metal exposure in Senegalese powdered milk.

Keywords: X-ray Fluorescence (XRF) spectroscopy, heavy metal, powdered milk, Health Risk Index, Daily Intake

1. Introduction

In recent years, the assessment of heavy metal contamination in food products has emerged as a critical component in ensuring both food safety and public health [1]. Powdered milk, being a staple in many diets worldwide, has been subjected to increased scrutiny due to its potential role as a carrier of heavy metals. Among the heavy metals of concern are lead (Pb), cadmium (Cd), copper (Cu), and zinc (Zn), each capable of eliciting adverse health effects if consumed in significant quantities [2, 3].

Industrial and agricultural activities have contributed to the escalation of heavy metal levels in the environment, including the air, soil, and water [4]. The potential transfer of these heavy metals into the food chain, particularly in livestock grazing on contaminated land, raises concerns about the accumulation of contaminants in their meat and milk [5, 6].

Lead (Pb), cadmium (Cd), copper (Cu), and zinc (Zn), characterized by their high atomic numbers, are among the most common heavy metals raising health concerns [7]. Understanding their presence in food items like powdered milk is crucial, as exposure to these heavy metals can lead to various health issues. The accurate determination of heavy metal concentrations relies on advanced analytical techniques, each with its strengths and limitations. Commonly employed techniques for heavy metal analysis include Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Atomic Absorption Spectroscopy (AAS), and X-ray Fluorescence (XRF). While ICP-MS and AAS offer high sensitivity and precision, XRF stands out as a non-destructive and rapid analytical method, capable of simultaneously detecting a wide range of elements [8, 9].

This study, conducted within the context of Senegal, aims to rigorously evaluate the risks associated with the presence of these heavy metals in various brands of powdered milk. Utilizing advanced nuclear analytical techniques, specifically X-ray Fluorescence (XRF) spectroscopy, our focus is to provide a comprehensive risk assessment, shedding light on the potential health implications for consumers in Senegal.

2. Materials and Methods

2.1. Samples Preparation

Samples of milk sourced from Senegal's markets are subjected to analysis using an X-ray fluorescence analyzer. This analytical process entails the direct exposure of the samples to a silver Ag anode for excitation. Additionally, a specially optimized large-geometry detector equipped with multiple filters functions as secondary sources during the analysis.

2.2. Analysis of Samples by X-ray Fluorescence

2.2.1. Experimental method

The study employed the X-ray fluorescence (XRF) method for the analysis of specific samples, utilizing a Niton XLT900s ED-XRF spectrometer with a resolution of 178 eV at Mn Kα. The spectrometer operates with a 12.7 µm Be window thickness, a 50 kV, 40 A excitation tube, and a maximum power of 2 W, producing a 7 mm beam diameter. Various filters, including Ag for excitation source, a sandwich of Al, Ti, and Mo, a Cu filter, and no filter, were applied to selectively target specific elements in the samples collected from Senegal's markets. The quantitative analysis was facilitated using the UniQuant 4 software.

2.2.2. Principe

X-ray Fluorescence (XRF) is an advanced analytical method for elemental composition analysis. It involves the generation of X-rays within an X-ray tube, where high-energy electrons collide with a metal target, producing X-rays. These X-rays are directed onto the sample, causing the ejection of inner-shell electrons from atoms, leading to the emission of characteristic X-rays specific to the elements present. The emitted X-rays are captured and analyzed by a detector, recording their energy and intensity, forming a spectrum that acts as a fingerprint of the sample's elemental composition. This spectral data serves as the basis for quantitative analysis, enabling precise determination of element concentrations. XRF's non-destructive nature, requiring no sample alteration, makes it versatile for various sample forms, finding applications in environmental monitoring, geological exploration, and industrial quality assurance. In essence, XRF stands as a sophisticated and indispensable tool for deciphering material composition intricacies in diverse scientific and industrial contexts [10].

2.3. Calculation of Health Risk

The evaluation of health risks associated with heavy metal exposure involves a comprehensive analysis, including the calculation of two pivotal parameters: Daily Intake (DIM) and Reference Dose (RFD). These metrics are instrumental in understanding the potential adverse effects of heavy metal ingestion over time[11].

The Daily Intake is determined by computing the average amount of a specific heavy metal ingested daily through the consumption of a particular food item, such as powdered milk. The DIM formula is expressed as:

$$DIM = (C_{metal} \times C_{factor} \times D_{food\ intake})/B_{average\ weight}$$

Where: Cmetal: Metal concentration in powdered milk (ppm), Cfactor: Conversion factor (0.085), Dfood intake: Average daily intake of milk (0.175 and 0.25 kg per person per day for adult and child, respectively) and Baverage weight: Average body weight (children: 15.0 kg and adult: 70.0 kg) [12].

The Reference Dose is a critical parameter established by regulatory bodies, such as the United States Environmental Protection Agency (USEPA), representing the maximum allowable daily intake of a specific heavy metal that is unlikely to cause adverse health effects over a person's lifetime. RFD values for Pb, Cd, Cu and Zn were 1, 40, 3.5 and 330, respectively [13].

The Health Risk Index (HRI) is typically calculated using the following formula:

$$HRI = \frac{DIM}{RFD}$$

3. Results and Discussion

Table 1 provides an overview of heavy metal concentrations, specifically lead (Pb), copper (Cu), zinc (Zn), and cadmium (Cd), in various powdered milk samples.

Table 1: Concentrations of Heavy Metals (Pb, Cu, Zn, Cd) in Powdered Milk Samples

SAMPLE	Pb	Cu	Zn	Cd
H1	< LOD	16.14 ± 7.34	81.71 ± 6.63	11.53 ± 4.54
G1	< LOD	16.68 ± 7.57	75.66 ± 6.65	11.5 ± 4.54
D1	< LOD	27.59 ± 7.52	67.33 ±6.18	11.21 ± 4.54
L1	< LOD	21.9 ± 5.69	76.46 ± 4.99	9.96 ± 4.25
V1	< LOD	25.86 ± 7.84	65.89 ± 6.33	16.48 ± 4.68

The analysis consistently reveals lead concentrations below the limit of detection (LOD), indicating minimal to negligible levels within the samples. Copper concentrations exhibit slight variations, ranging from approximately 22 to 27 ppm, necessitating a meticulous comparison with regulatory standards to evaluate compliance with safety norms. Similarly, zinc concentrations display variability within an average range of 66 to 82 ppm, requiring a comprehensive assessment against established limits for a thorough safety evaluation. Cadmium concentrations, averaging from 10 to 16 ppm, demonstrate notable variability. It is imperative to acknowledge that the precision of these results depends on the accuracy of the analytical methods used and potential sources of variability in the sampling process. Rigorous quality control measures and sensitivity analyses are essential to address potential uncertainties, contributing to a more nuanced understanding of the health implications associated with the consumption of powdered milk.

3.1. Comparisons of Heavy Metals in Milk Samples

Analyzing copper concentrations in powdered milk samples (Figure 1) reveals significant variations among the samples.

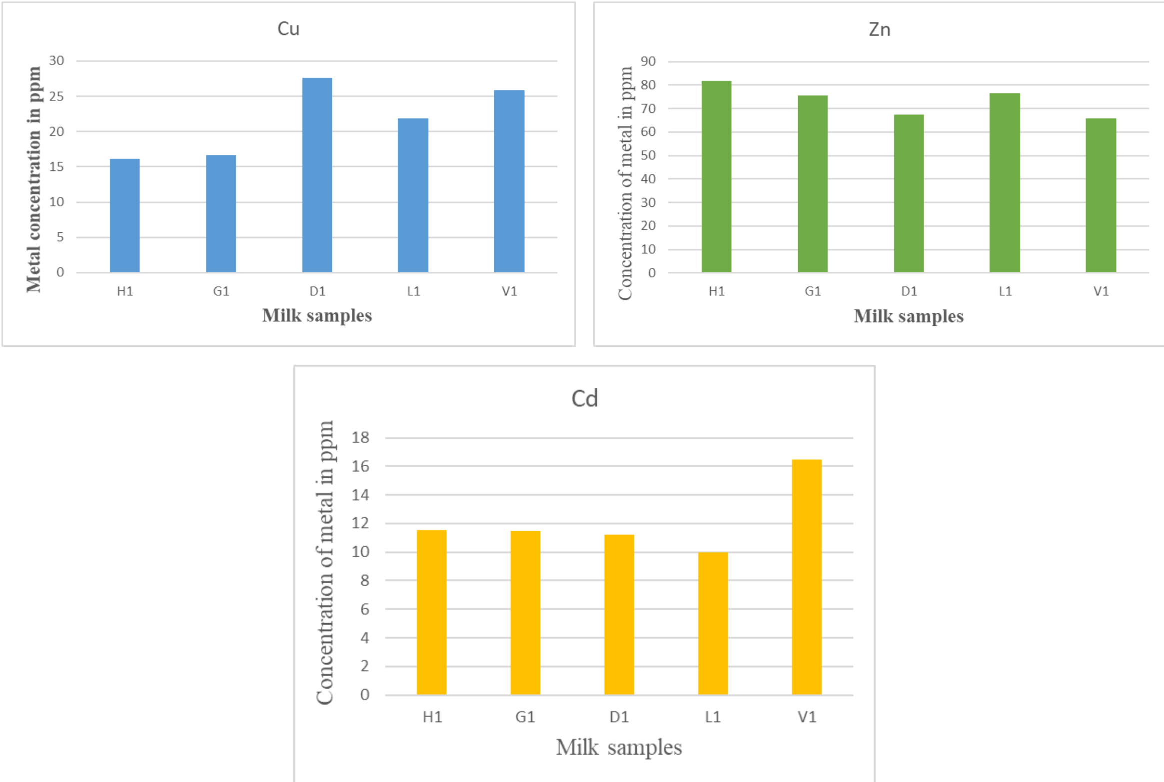

Figure 1: The comparison of the Cu, Zn and Cd content in different milk sample

The substantial differences manifest in both the maximum and minimum concentrations, with sample D1 exhibiting a peak concentration of 27.59 ppm and sample H1 revealing a minimum concentration of 16.14 ppm. This highlights a considerable diversity in copper levels across the samples, with each value representing an individual concentration and emphasizing the substantial variability observed. The direct comparison of these concentrations further solidifies the presence of significant variations among the samples, notably where sample D1 demonstrates a higher maximum concentration compared to sample H1.

The analysis of cadmium (Cd) concentrations in powdered milk samples (Figure 1) reveals significant variations among the samples. Differences are particularly evident in the maximum and minimum concentrations, with a peak of 16.48 ppm in sample V1 and a minimum concentration of 9.96 ppm in sample L1. This diversity emphasizes substantial variability in cadmium levels among the samples, with each value representing an individual concentration.

The analysis of zinc (Zn) concentrations in powdered milk samples (Figure 1) reveals significant variations among the samples. The maximum concentration is observed in sample H1 with a value of 81.71 ppm, while the minimum concentration is recorded in sample L1 with a value of 76.46 ppm. This difference highlights the diversity of zinc levels among the samples, with each value representing an individual concentration. The direct comparison of concentrations reinforces the notion of significant variations, where sample H1 exhibits a higher maximum concentration than sample L1.

3.2. Daily Metal Intake and Health Risk Index: Evaluating Health Risks

The daily intake of metals and their associated health risks depend on various factors, including the specific metal, the form in which it is present, and individual characteristics such as age, sex, and overall health. Different metals have different toxicity levels, and some are essential for the proper functioning of the human body in trace amounts, while others can be harmful even in small quantities. The table 2 provides a detailed analysis of daily metal intake levels and corresponding Health Risk Indices (HRI) for both adults and children. The data encompasses essential metals zinc (Zn) and copper (Cu), as well as the potentially harmful metal cadmium (Cd). Explore the dimensions (DIM) of metal intake and HRIs to understand the potential health risks associated with each sample.

Table 2. Health risk index (HRI) of Cd, Cu and Zn through the consumption of asteurized milk for adults and children

	Adults						**Children**					
	Zn		Cu		Cd		Zn		Cu		Cd	
Samples	DIM	HRI	DIM	HRI	DIM	HRI	DIM	HRI	DIM	HRI	DIM	HRI
H1	0.017	5.261E-05	0.003	0.0010	0.0025	6.125E-05	0.116	0.0004	0.023	0.007	0.016	0.0004
G1	0.016	4.872E-05	0.004	0.0010	0.0024	6.109E-05	0.107	0.0003	0.024	0.007	0.016	0.0004
D1	0.014	4.335E-05	0.006	0.0017	0.0024	5.955E-05	0.095	0.0003	0.039	0.011	0.016	0/0004
L1	0.016	4.923E-05	0.005	0.0013	0.0021	5.291E-05	0.108	0.0003	0.031	0.009	0.014	0/0004
V1	0.014	4.24E-05	0.005	0.0016	0.0035	0.00008755	0.093	0.0003	0.037	0.010	0.023	0.0006

The data provided in the table present a comprehensive assessment of daily metal intake (DIM) and Health Risk Index (HRI) related to zinc (Zn), copper (Cu), and cadmium (Cd) in adults and children consuming powdered milk. These findings contribute valuable insights into potential health risks associated with metal exposure [14].

The DIM values for zinc range from 0.014 to 0.017 for adults and from 0.093 to 0.116 for children, indicating a consistent level of zinc intake across the samples. The corresponding HRI values, consistently below 1, suggest that the zinc intake from powdered milk is within acceptable limits, posing a minimal health risk. Moving to copper (Cu), the DIM values fluctuate between 0.003 and 0.006 for adults and 0.023 and 0.039 for children. Despite this variation, the HRI values consistently fall within acceptable limits. This indicates that the copper intake from powdered milk is unlikely to pose a significant health risk and aligns with established safety standards. The HRI values for cadmium also remain below acceptable thresholds, indicating a potentially low risk. However, continuous monitoring of cadmium concentration in powdered milk is necessary to ensure long-term safety. Overall, the results suggest that the consumption of powdered milk poses health risks that are widely acceptable, with HRI values maintaining safe levels for the specific metals studied. Nevertheless, it is crucial to emphasize the importance of ongoing monitoring to ensure that these levels remain within safe limits over time.

A comprehensive comparison of Health Risk Index (HRI) values between adults and children was conducted, utilizing graphical representation to elucidate distinct risk profiles associated with the consumption of powdered milk in each group. The Figure 2 represents the comparative Health Risk Index (HRI) profiles between adults and children in the context of powdered milk consumption in Senegal.

The overarching conclusion drawn from Figure 2 is that, on average, the HRI curve of all heahy metal (Zn, Cu and Cd) for children is positioned above that of adults, signifying a higher health risk for children in the context of powdered milk consumption. This observation underscores the importance of considering each metal individually to gain a comprehensive understanding of the nuanced risk dynamics associated with different elements. This discrepancy is a result of the inherent variances in daily milk intake and body weight between the two demographic groups. Notably, children, characterized by higher daily milk consumption and lower body weight, present slightly elevated HRI values for specific metals. The application of X-ray Fluorescence (XRF) in assessing metal concentrations in powdered milk emerges as a valuable and indispensable tool for evaluating health risks. The comprehensive analysis provided by XRF enables a detailed understanding of the metal composition, allowing for a precise assessment of potential health implications associated with powdered milk consumption. By employing XRF, researchers gain the ability to identify and quantify specific metals, such as Pb, Cd, Cu, and Zn, contributing to a more nuanced risk assessment. The graphical representation of Health Risk Index (HRI) profiles, as illustrated in Figure 2, becomes a powerful visual aid in discerning differential health risks between demographic groups.

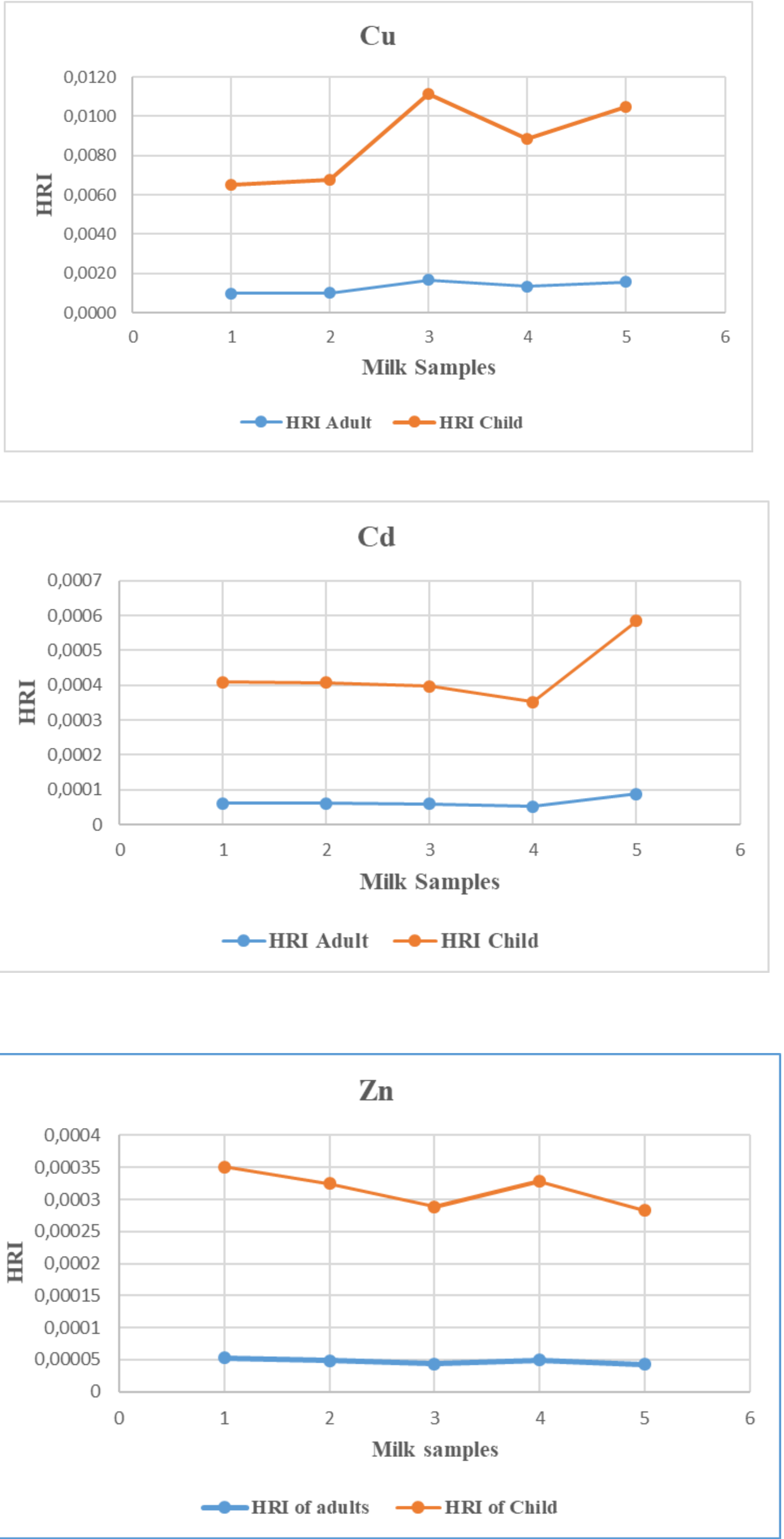

Figure 2: The comparative Health Risk Index (HRI) profiles between adults and children

4. Conclusion

The comprehensive analysis of heavy metal concentrations in Senegalese powdered milk using X-ray Fluorescence (XRF) spectroscopy has provided valuable insights. The study focused on five specific samples V1, H1, L1, D1, and G1 sourced from the Senegalese market, revealing varying concentrations of copper, zinc, and cadmium. Importantly, lead concentrations remained below the limit of detection. The Health Risk Index (HRI) values derived from pàwdered milk consumption in all analyzed samples were consistently within safe limits (HRI < 1) for both adults and children. These findings suggest that the potential health risks associated with heavy metal intake from powdered milk are minimal and align with established safety thresholds. The study emphasizes the importance of continuous monitoring and rigorous quality control measures to ensure the sustained safety of powdered milk products in Senegal. Furthermore, the recommendation to set legal limits for heavy metals in milk is underscored, serving as a crucial step in preventing adverse health effects and promoting community awareness. Overall, the insights gained from this research contribute to the ongoing efforts to ensure the safety and well-being of consumers in the context of powdered milk consumption in Senegal. In light of these findings, it is advisable to institute ongoing monitoring procedures for these contaminants in milk. Furthermore, there is a critical necessity to establish legal thresholds for heavy metal concentrations in milk. This proactive step is essential for averting potential adverse effects of these metals on human health. Simultaneously, it will contribute significantly to raising community awareness about the safety of milk products.

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