






Research Article

Using Nuclear Methods in X-ray Fluorescence Spectroscopy to Quantify Heavy Metals in Senegalese Powdered Milk: Improving Milk Safety

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Abstract

Applying X-ray fluorescence (XRF), this study investigated the elemental analysis and heavy metal contents in five Senegalese powdered milk samples (V1, L1, H1, G1, and D1). The primary focus was on Aluminum (Al), Calcium (Ca), Potassium (K), Phosphorous (P), and Chlorine (Cl), with special attention given to the compliance of these elements with safety standards. The analysis revealed that Aluminum was either absent or present in minimal quantities across all samples, suggesting that the powdered milk is largely free from this element. Calcium levels were found to be consistently higher than the Acceptable Maximum Level (AML) across all samples, with the H1 sample significantly exceeding the AML by approximately 11.1 times, with a concentration of $27,745.06 \pm 310.16$ ppm. This indicates a potential risk of excessive calcium intake from this sample. Potassium concentrations varied significantly; while the V1 sample remained within acceptable limits, the G1 sample exhibited potassium levels substantially above the AML, reaching $51,058.15 \pm 456.13$ ppm, which could pose health concerns if consumed in large quantities. Chlorine concentrations generally met the AML, except for the G1 sample, which slightly surpassed the limit at 3631.04 ± 31.23 ppm. The phosphorus content in the H1 sample was notably high, though further details are needed to fully assess its implications. The study underscores the necessity for continuous monitoring of heavy metal and elemental levels in powdered milk to ensure consumer safety.

Keywords

X-ray Fluorescence, Powdered Milk, Heavy Metal Concentrations, Elemental Analysis and AML

1. Introduction

As a staple of the global food business, powdered milk has a long history and is an essential part of human nutrition. In addition to being a practical and long-lasting way to preserve

milk, it is essential to a wide variety of food preparations [1]. As more people prefer to powdered milk because of its nutritional advantages, safety concerns grow significantly [2]. This

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emphasizes how important it is to thoroughly examine any possibly harmful substances found in powdered milk in order to protect the public's health [3, 4].

The safety and quality of this frequently consumed commodity are questioned in light of possible impurities such as heavy metals, pesticides, mycotoxins, and microbiological agents. Even though strict regulations have been put in place all over the world to monitor and regulate the quality of food products, continued examination and study are necessary to handle new issues and possible hazards [5-7].

For the purpose of evaluating milk's nutritional value and guaranteeing consumer safety, the precise identification of trace components in milk is essential. The elemental makeup of milk samples has been uncovered using a variety of analytical approaches, each with advantages and disadvantages of its own [8]. Understanding the nutritional profile of milk and spotting possible impurities require knowledge of its basic composition. Important functions for human health are played by essential elements like calcium, magnesium, and phosphorus, as well as trace elements like copper, zinc, and selenium. On the other hand, hazardous substances including lead, cadmium, and arsenic can present serious health hazards [9].

Atomic Absorption Spectrometry (AAS), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) are among the methods used to analyze trace elements in milk. Even though every technique has its own benefits, they are frequently accompanied by difficulties such as complicated sample preparation, lengthy analysis times, or the need for specialist equipment [10-13].

Among these methods, X-ray fluorescence (XRF) is particularly well-known and adaptable because to its quick, non-destructive, and simultaneous quantification of several components. In order to perform XRF, a non-destructive method, high-energy X-rays are applied to the milk sample, which results in the emission of distinctive X-rays that are subsequently detected and measured. X-ray fluorescence (XRF) is a non-destructive method that has a number of advantages over Inductively Coupled Plasma Mass Spectrometry (ICP-MS) for the detection of milk powder. XRF analyzes materials without changing their composition, in contrast to ICP-MS. This non-destructive quality is especially important when working with sensitive matrices, like milk powder, because it's critical to maintain the original sample integrity. XRF ensures a more accurate portrayal of the true metal by preventing sample tampering [14-16].

This study's main goal is to conduct a thorough heavy metal analysis on five different powdered milk samples (identified as V1, L1, D1, G1, and H1) that were purchased from dif-

ferent Senegalese marketplaces. In order to evaluate and quantify the amount of heavy metals in each sample, an energy dispersive X-ray fluorescence system will be used. This research will provide important information about the safety and caliber of these widely available powdered milk products.

2. Materials and Methods

2.1. Samples Preparation

Senegalese marketplaces provide milk samples, which are collected and examined in an X-ray fluorescence analyzer. Direct exposure to a silver Ag anode is used in the study as the excitation source, and secondary sources are provided via a large-geometry detector that has been carefully designed and has several filters.

2.2. Samples Analysis

The X-ray fluorescence (XRF) method was employed in the investigation to assess specific samples. At Mn K α , a Niton XLT900s ED-XRF spectrometer with a resolution of 178 eV was used for the X-ray fluorescence (XRF) study. With a maximum power of 2 W and a 50 kV, 40 μ A excitation tube, the system produces a 7 mm beam diameter with a window thickness of 12.7 μ m Be. Different filters were used to target different elements in the samples that were taken from Senegalese markets. These filters included a Cu filter, an excitation source with Ag, a sandwich of Al, Ti, and Mo, and no filter at all. The UniQuant 4 program made the quantitative analysis easier.

It is commonly known that detecting thin samples in energy dispersive X-ray fluorescence has many benefits. Measurements using thin samples have several advantages over approaches that use thick samples. The increase in characteristic line intensity resulting from thicker samples is overshadowed by absorption and enhancement effects introduced by the rise in background generated by multiple scattering within the sample. However, in thin samples, these amplification and absorption effects are less noticeable and are easily ignorable or rectified [17-19].

3. Results and Discussion

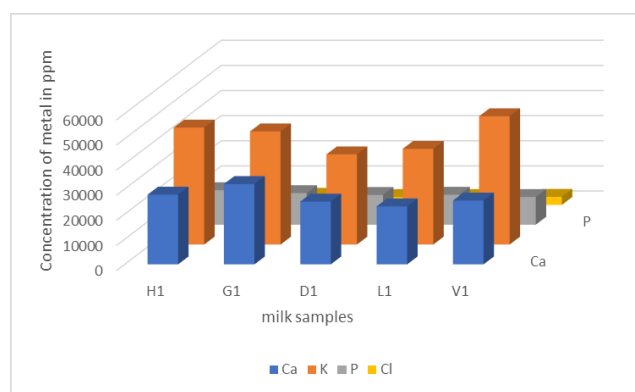
Table 1 shows the concentrations (mg/kg) of the chosen elements (calcium, potassium, phosphorus, aluminum, and chlorine) in milk samples from the Senegalese market.

Table 1. Concentrations (ppm) of selected metals (Aluminum, Calcium, Potassium, Phosphorus, and Chlorine) in different milk samples collected from Senegal.

SAMPLE	Al	Ca	K	P	Cl
H1	< LOD	27745.06 \pm 310.16	46530.57 \pm 517.66	13750.94 \pm 275.35	3374.78 \pm 35.44
G1	< LOD	31988.84 \pm 234.15	45015.56 \pm 353.56	12543.08 \pm 262.15	3631.04 \pm 31.23
D1	< LOD	24981.69 \pm 254.42	35864.07 \pm 389.9	11859.23 \pm 268.16	3123.74 \pm 34.69
L1	< LOD	23068.86 \pm 206.41	38090.09 \pm 349.99	11920.51 \pm 250.46	3106.45 \pm 30.25
V1	< LOD	25397.82 \pm 250.14	51058.15 \pm 456.13	11022.99 \pm 240.57	3183.96 \pm 20.17

Metal concentration measurement in milk samples provide important information about the quality of dairy products and possible environmental effects. Notably, all samples' levels of aluminum (Al) were consistently below the limit of detection (LOD), suggesting that the milk under analysis contained very little of the metal. The samples' quantities of calcium (Ca) varied; sample H1 had the greatest concentration, followed by samples G1, D1, L1, and V1. The geographical location of the farms, variances in the nutrition of the cattle, or the makeup of the soil could all be contributing factors to these disparities. During childhood and adolescence, the body uses calcium minerals to build strong bones; this process usually ends by the time an individual reaches the age of 10. The build-up of calcium in the bones is reduced in childhood and even more in old age, with a greater effect on women. Young people are more likely to develop osteoporosis, a bone disease that increases the risk of fractures, especially females whose diets are deficient in minerals that strengthen bones [20]. In order for muscles to contract, nerve signals to be transmitted, and hormones to be released, calcium is essential. The body moves calcium from the bones to maintain proper cellular function when blood calcium levels fall (usually as a result of insufficient food consumption). Both too much and too little calcium can throw off the body's acid-base equilibrium, resulting in problems including hypercalcemia and other illnesses. It is essential for general health to maintain an ideal balance of calcium consumption, avoiding the negative consequences of both excess and deficiency [21]. Significant variation was seen in the amounts of potassium (K), with sample V1 displaying the highest levels. On the other hand, samples H1 and D1 showed significantly lower potassium amounts, which could be related to differences in cattle diet or local soil features. The amounts of phosphorus (P) showed a significant variation [22].

It is clear from comparing these data that there are variations in the metal content of milk from different sources, as shown in Figure 1. A number of variables, including geographic location, agricultural techniques, and environmental conditions surrounding the dairy farms, may have an impact on these variations.

**Figure 1.** Illustrat concentrations of Al, Ca, K, P, Cl in different milk samples.

Given the possible health effects of these metal concentrations, more investigation into the origins of metals in milk and the formulation of plans to guarantee the security and caliber of dairy products are necessary.

The concentrations of elements (P, K, Cl, Al, and Ca) discovered in the milk samples have been compared to the Acceptable Maximum Levels (AML) shown in table 2 in the context of this investigation.

Table 2. Acceptable Maximum Levels (AML) in ppm of Metals in powdered milk [23].

Metals	K	Ca	Cl	Al	P
AML (ppm)	3,500	2,500	3,400	867	1,000

By comparing the obtained results with predetermined standards, this method seeks to provide important information about the safety and quality of the samples under examination.

The distribution of potassium contents in the milk samples under analysis is clearly depicted in Figure 2. Notably, sample V1 has the highest potassium levels found in this investigation.

After closely examining samples V1, H1, D1, and L1, it is clear that their potassium concentrations are more than three times greater than the Acceptable Maximum Level (AML), which is established at 3,500 ppm.

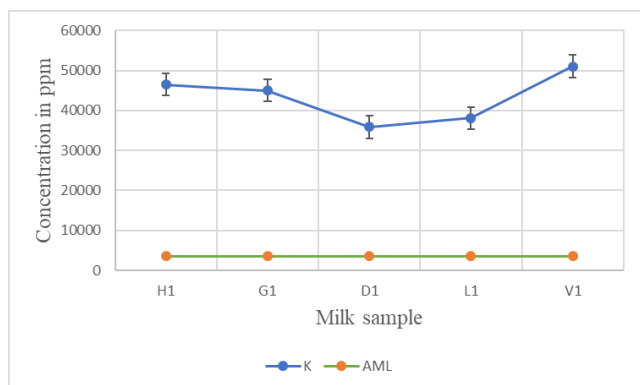


Figure 2. Distribution of K Concentrations in Milk Samples.

Moreover, sample G1 is notable for having a potassium content that is substantially higher than the AML at 51,058.15 ppm. This highlights how crucial it is to keep an eye on and assess the levels of metals in milk, especially potassium, as this may have an impact on food consumption. On the other hand, sample V1 shows a potassium level that is below the AML at 11,022.99 ppm. Based on this discovery, the potassium levels in sample V1 are within the permissible limitations set by the existing legislation. The distribution analysis reveals the much higher potassium levels in samples G1 and V1, giving a visual depiction of metal concentrations in various samples. The significant excess potassium in sample G1, which is higher than the Acceptable Maximum Level (AML), highlights how crucial it is to follow regulations.

Furthermore, the higher potassium levels found in different samples highlight the necessity of additional research into any possible health risks related to these milk concentrations.

The outcomes keep highlighting the notable departures from the requirements set forth by regulations. Although sample G1's potassium concentrations caused alarm, sample H1, G1, D1, L1, and V1's calcium concentrations, as shown in Figure 3, show an even more marked deviation from the Acceptable Maximum Level (AML) of 2500 ppm.

The calcium concentrations in samples H1, G1, D1, L1, and V1 are consistently higher than the AML, according to the data. Sample H1 outperformed the AML by around 11.1 times, with the highest values measured at 27,745.06 ppm. Sample D1 surpasses the AML by approximately 10.0 times, sample G1 by approximately 12.8 times, sample L1 by approximately 9.2 times, and sample V1 by approximately 10.2 times. Because of the substantial over-limit of these concentrations, this observation raises serious questions about regulatory compliance. Although milk contains high amounts of calcium, this vitamin may have nutritional consequences. The difference between the allowed limits and the concentrations that have

been detected highlights the need to monitor the amount of calcium in milk more closely and the possible health risks of consuming too much of it. To guarantee dairy products are safe and of high quality, it is imperative to take these findings into account in the context of dietary guidelines and legal requirements. In order to ensure that milk's nutritious content remains optimal while adhering to regulatory guidelines, more research is necessary to determine the causes of elevated calcium levels. A daily calcium intake of 1,200-1,500 mg is recommended for this age group based on the data that are currently available [24].

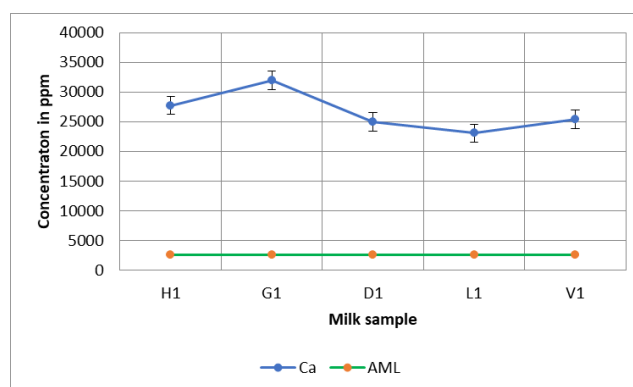


Figure 3. Distribution of Calcium Concentrations in Milk Samples.

The levels of chlorine (Cl) in the milk samples that were studied were carefully evaluated in accordance with the regulatory guidelines, particularly the Acceptable Maximum Level (AML), which is set at 3,400 parts per million. The distribution of chlorine concentrations in each sample was shown in Figure 4 to give a thorough visual depiction of the dispersion of concentrations.

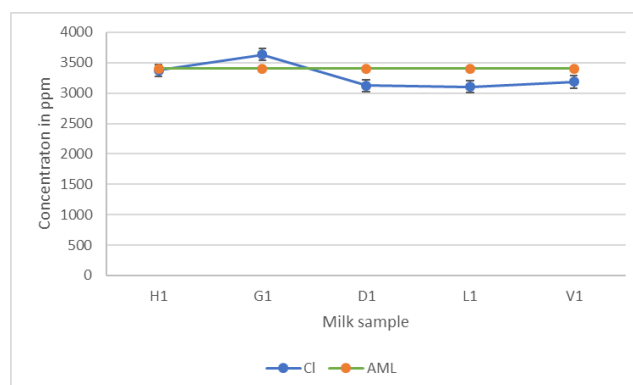


Figure 4. Distribution of Chlorite Concentrations in Milk Samples.

The amounts of chlorine in samples H1, D1, L1, and V1 are below the AML, according to the results. Sample G1, however, had the highest levels of chlorine, at 3631.04 ppm, which was marginally higher than the AML. Although chlorine is a

common ingredient found in food and water, it is important to keep concentrations within permitted limits for both environmental and safety reasons. On the regulatory front, the compliance noted in other samples is encouraging, and more investigation into the effects of milk's chlorine content on the environment may be warranted. Our knowledge of the general composition and safety of dairy products is improved by this analysis, which offers insightful information about the regulatory compliance of chlorine concentrations in milk.

The distribution of phosphorus contents in milk samples is shown in [figure 5](#). The quantities of phosphorus (P) in the milk samples under analysis were carefully compared to regulatory criteria, particularly the 1,000 ppm Acceptable Maximum Level (AML).

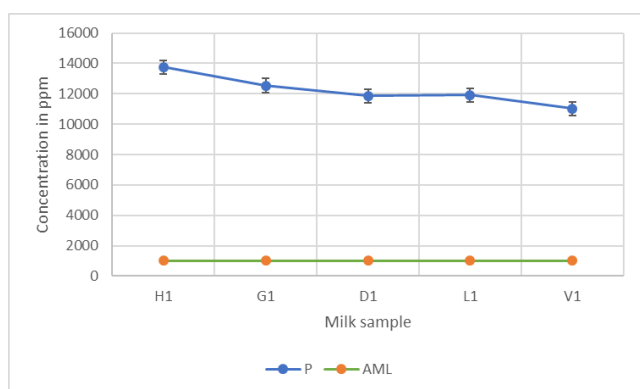


Figure 5. Phosphorus Concentration Distribution in Milk Samples.

The phosphorus concentrations in samples H1, G1, D1, L1, and V1 are shown in [Figure 5](#). According to the results, there are differences in the phosphorus contents amongst these samples. Sample H1 had the highest value, measuring 13,750.94 ppm, which is much more than the AML. This significant departure from the regulation requirements in sample H1 highlights the significance of closely examining and resolving increased phosphorus levels in milk. By highlighting the amount of the divergence in phosphorus concentrations graphically, the distribution analysis offers important new information about the general safety and caliber of dairy products. Ensuring the integrity of milk requires strict adherence to regulatory criteria; any discrepancies, as demonstrated in sample H1, call for additional research and corrective action. Phosphorus is a necessary element, but it's important to keep concentrations within reasonable ranges for safety and legal compliance. This study advances our knowledge of how phosphorus concentrations in milk comply with regulations, opening the door for further debate on refining requirements and guaranteeing the security of dairy products.

The complex interactions between metal concentrations in milk and set regulatory criteria are clarified by this extensive investigation. Variations in the levels of potassium, calcium, chlorine, and phosphorus highlight the necessity of continu-

ous monitoring to guarantee dairy products' safety and quality. Depending on the particular procedures involved, variables like species variability, geographic location, production methods, and equipment contamination can all have an impact on the level of heavy metals in milk [24].

These findings add to a larger conversation about fine-tuning regulatory requirements and preserving a precarious balance between nutrient requirements and safety regulations in our food supply.

4. Conclusion

Using X-ray fluorescence (XRF), this extensive investigation examined the levels of heavy metals in five Senegalese powdered milk samples (V1, L1, H1, G1, and D1). The elements that were analyzed were potassium (K), phosphorus (P), calcium (Ca), aluminum (Al), and chlorine (Cl). All samples had either very little or no aluminum, indicating that the milk was high quality in terms of this element. The persistently high calcium levels in all samples, particularly in H1, which was beyond the Acceptable Maximum Level (AML) by around 11.1 times, are concerning, though. The results of the investigation show that potassium levels vary, with G1 showing noticeably higher concentrations than the AML. Chlorine concentrations were mostly in line with the AML, while G1 displayed a small variation. The significantly elevated phosphorus levels in H1 highlight how crucial it is to keep a careful eye on these ingredients for food safety. The fact that the samples are not all the same highlights the need of taking into account variables that affect the metal content of powdered milk, such as location and farming methods. To guarantee the safety and quality of powdered milk in Senegal, it is advised to implement improved quality control procedures, frequent testing, public awareness programs, and producer collaboration. This proactive strategy protects public health and upholds regulatory compliance in the food supply chain by adhering to international standards and helping to address new issues.

Abbreviations

Al	Aluminum
Ca	Calcium
K	Potassium
P	Phosphorous
Cl	Chlorine
AML	Acceptable Maximum Level

Author Contributions

Papa Macoumba Faye: Conceptualization

Djicknack Dione: Validation

Oumar Ndiaye: Writing – review & editing

Moussa Hamady Sy: Writing – review & editing

Nogaye Ndiaye: Writing – review & editing

Alassane Traore: Supervision

Ababacar Sadikhe Ndao: Supervision

Conflicts of Interest

The authors declare no conflicts of interest.

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