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REVIEW ON THE RECENT SAMPLE PREPARATION METHODS FOR THE ANALYSIS OF TOXIC HEAVY METALS IN FOODS AND BEVERAGES

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ABSTRACT

This review critically assesses conventional and emerging sample preparation techniques for analyzing toxic heavy metals in foods and beverages. It highlights limitations of traditional methods like wet and dry ashing such as time consumption, potential analyte loss, and high solvent use while introducing efficient alternatives QuEChERS and DLLME. Analysis of studies, particularly on Nigerian food staples, reveals that wet ashing offers superior metal recovery over dry ashing. Alarmingly, lead and cadmium levels often exceed WHO/FAO safety limits, indicating serious health risks. The emerging methods are presented as cost-effective, efficient, and greener alternatives that overcome the drawbacks of conventional approaches. The findings emphasize the urgent need for robust monitoring programs and the adoption of advanced techniques to ensure food safety and protect public health. Future research should focus on validating these methods across diverse food matrices.

Keywords: Heavy metals, Food contamination, Sample preparation, Wet ashing, Dry ashing, QuEChERS, DLLME

INTRODUCTION

Agricultural pollution is a complex combination of factors that contribute to its great diversity. Therefore, it has some negative consequences on biotic communities in terms of air, water and soil pollution (Yang *et al.*, 2018). In addition, liquid effluents from urbanized cities, industries and agricultural fields contain highly harmful elements such as persistent heavy metals, polya romatic hydrocarbons (PAH), plastics and polymers, pesticides, chemicals and reagents, atmospheric deposition, bioaerosols, grain pollen, redegradable microorganisms, which creates serious environmental and health-problems in living beings (Nilsen *et al.,* 2019). The deposition of certain pollutants in agricultural products, soil, water, air and even in the upper trophic levels of the food chain has disturbed the proper functioning of the terrestrial ecosystem. Despite the global agricultural and economic revolution of the last 50 years, humans have transformed natural ecosystems to suit their selfish needs. This is due to the increase in the demand for food due to the explosion of natural resources, the uncontrolled growth of the world population and the use of chemical substances to promote crop productivity and plant protection (Bergström and Randall, 2016). However, the balance between the positive and toxic effects of heavy metals depends on their concentration in living cells. Therefore, the concentration of metal ions must be maintained in an appropriate range to avoid nutritional deficiencies, while higher concentrations can lead to health problems, since these heavy metal pollutants have become a global phenomenon. Heavy metals bioaccumulate in humans, crops, and vegetables as a result of ongoing consumption.

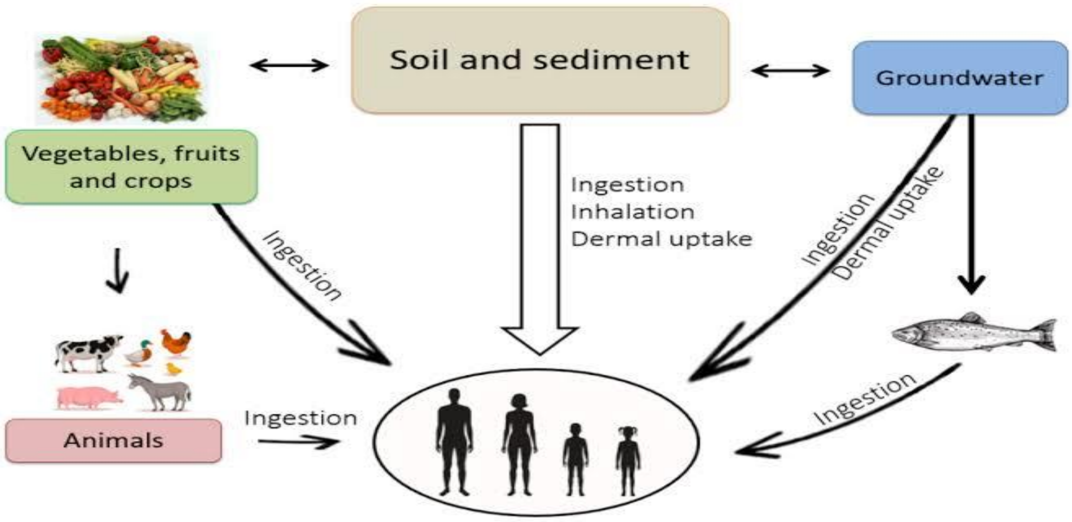

Figure 1: The Flow of Heavy Metals Through Food Chain (Kumar et al., 2019)

Food Safety and Quality

Food safety and quality are of great importance in the food industry and the agricultural aspect of production because they affect the health and well-being of consumers. Food safety refers to the measures taken to prevent contamination of food products, while food quality refers to the attributes of food products that affect their appeal and acceptability to consumers. Many challenges must be overcome to maintain a high standard of food and drink, as contaminated food can cause food borne illnesses that can lead to hospitalization or even death.

Contaminants and Examples

Contaminants are substances that can cause adverse effects on the environment and human health. They can be found in air, water, and soil, and they may originate from various sources, including industrial processes, agricultural practices, and urban runoff. Contaminants can be classified into several categories, but two major groups are organic and inorganic contaminants.

Organic Contaminants includes pesticides like DDT and glyphosate that are used in agriculture, so like volatile organic compounds (VOCs) such as benzene and toluene used in industrial applications while inorganic Contaminants includes metals such as arsenic, lead, mercury, copper, and cadmium, nutrients like nitrates and phosphates which can lead to eutrophication in water bodies, salts; high concentrations of sodium (salinity) can affect soil and water quality.

Heavy metals are a subset of inorganic contaminants that are particularly concerning due to their toxicity, persistence in the environment, and bioaccumulation in living organisms. They are typically defined as metals with relatively high density (typically greater than 5 g/cm³) and include a variety of elements that can be harmful even at low concentrations.

Due to the severe health risks and environmental impacts associated with these heavy metals, it is crucial to monitor their concentrations in various ecosystems. Regulatory bodies like the Environmental Protection Agency (EPA) and World Health Organization (WHO) set limits for heavy metal concentrations in drinking water, air, and soil to minimize exposure and prevent harm. Heavy metal contamination of the food chain as a result of urbanization and industrialization is of great concern because of potential health hazard due to dietary intake of contaminated products. Crop plants, especially tubers, are vital to the human diet and in particular, provide the nutrients to maintain normal health. The prolonged application of fertilizers, pesticides have resulted in the accumulation of heavy metals. The contamination of the food chain is the primary pathway of heavy metals exposure for living beings.

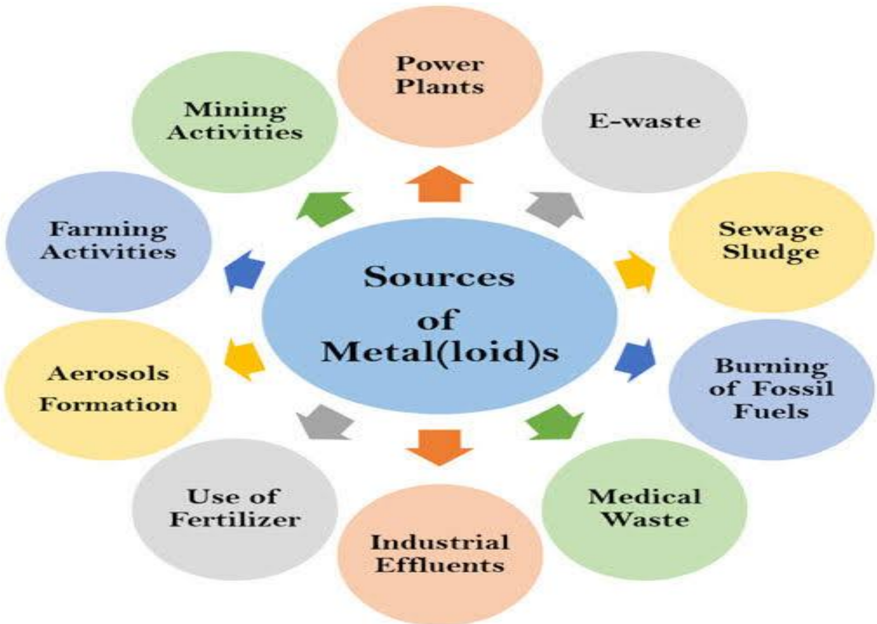

Figure 2: Sources of Hazardous Heavy Metal

Heavy Metals

According to John (2002) and Pain (2008), heavy metals are metallic chemical elements that are hazardous at low concentrations and have a relatively high density. Cadmium 8.65 g/m^3, lead 11.34 g/m^3, and mercury 13.546 g/m^3 are a few examples. They are detrimental to aquatic and human life and can be found in agrochemicals, industry, and urban runoff. While the majority of heavy metals are toxic, some are essential to human health. Iron, for instance, is crucial for the production of red blood cells; manganese is necessary for the development of connective tissues, bone, and blood coagulation; and copper promotes the absorption of iron, glucose, and cholesterol metabolism. At high concentrations, these necessary metals can also be harmful. According to Edvardo et al., (2005), cadmium and lead are non-essential elements that can enter the body through food, sediments, and drinking water. According to ATSDR (1999), Gilman et al., (2001), and the EPA (2008), heavy metal toxicity can cause damage or diminished mental and central nervous functioning, lower energy levels, and damage to the lungs, liver, kidney, and other essential organs. Cancer may also result from prolonged, repeated exposure to low concentrations of some of these metals or their derivatives (WHO, 2004). When they come into touch with the human body in the pharmaceutical and agricultural manufacturing sectors, they can enter through the food, air, or skin (Linnik, 2000; Calkins, 2009).

Health Impacts

Significant levels of hazardous heavy metals are released into the environment as a result of fast expansion and urbanization (Gupta et al., 2019; Shrivastava et al., 2019). These metals build up in crops, soil, and water, get into the food chain, and bioaccumulate to pose major health concerns (Kumar et al., 2019). When consumed or inhaled in excess of prescribed thresholds, exposure to metals like cadmium, lead, arsenic, and others can cause a variety of hazardous effects, including neurological, respiratory, and gastrointestinal issues. Efficient sample preparation is necessary in order to appropriately evaluate these health hazards laboratory analysis. The difficulty of effectively removing trace metals from intricate plant and environmental matrices without loss or contamination is one of the major obstacles that researchers must currently overcome. The accuracy, repeatability, and detection limits of subsequent heavy metal quantification are greatly impacted by decisions made about sample strategy, homogenization, digesting techniques, and solvent selection.

Table 1: Harmful Effects of some Toxic Heavy Metals on Plants and Humans

Heavy metal	Effects on Plants	Effects on Humans	References
As	Seed germination and seedlings height reduction in rice crop	Cardiovascular, neurologic and developmental anomalies	Abbas et al., 2018
Cd	Seed germination reduction, decrease in nutrient use efficiency and reduced shoot and root length in wheat crop.	Bone demineralization, renal dysfunction	Loi et al., 2018
Co	Antioxidant enzyme activities reduction; decrease in plant sugar, starch, amino acids, and protein content in mungbean	Severe health effects on the lungs, including asthma and pneumonia	Jayakumar et al., 2008
Cr	Inhibition of germination process; plant biomass reduction in garlic.	Multi-organ toxicity, nose ulcers and cancer of the respiratory tract	Medda and Mondal, 2017
Pb	Reduction in germination percentage; suppressed growth; reduced plant biomass; decrease in plant protein content in maize crop.	Kidney failure, affect CNS and PNS, damage to the reproductive systems	Hussain et al., 2013

The continuous analysis of heavy metals is essential for protecting human health, maintaining environmental integrity, ensuring compliance with regulations, and supporting scientific research. As technologies evolve and new sources of contamination emerge, establishing robust monitoring programs will be increasingly vital to address these challenges effectively.

Objective of the Research

The core objective is to critically evaluate wet and dry sample preparation techniques for heavy metals analysis in food and beverage matrices, and introduce effective approach to solve the limitations associated with the conventional techniques.

Some Recent Review Methods for the Analysis of Heavy Metals in Various Food Materials

There are numerous methods available for the analysis of these toxic compounds from different substrates, such as wet digestion, dry digestion, but most of the procedure are expensive and difficult to perform. These methods have several limitations, including the time required, the effect of temperature and the high consumption of corrosive inorganic solvents.

In a research conducted by Kalagbor et al., (2014), comparative study between dry and wet ashing methods for quantification of heavy metals (Pb, Ni, Cu, Mn, Co) in three Nigerian vegetables (bitter leaf, scent leaf, waterleaf). Wet ashing yields higher metal recovery due to limitations of dry ashing (partial mineral loss at high temperatures). Quantitatively, wet ashing recovers more metals due to complete organic matrix decomposition and consistently yielded higher concentrations, especially for Co (10× higher) while dry ashing limitations includes high temperatures which cause volatile metal loss (e.g., Co, Ni). Conclusively, wet ashing is more reliable for heavy metal analysis in vegetables (Tuzen et al. (2004), Soylak et al. (2004), Guang et al. (2008).

Table 2: Metal Concentrations (mg/kg) (Kalagbor et al., 2014)

Metal	Dry Ashing Range	Wet Ashing Range
Pb	20.6–47.2	68.2–196
Ni	24.0–38.8	138–276
Cu	20.7–35.1	42.6–99.6
Mn	18.8–116	63.1–172
Co	20.1–40.2	278–393

According to Koki et al. (2019), the levels of heavy metals (Cd, Fe, Zn, and Cu) in sweet potatoes (*Ipomea Batatas)* and yams (*Dioscorea SPP*) followed the following trends: Zn > Fe > Cu > Cd and Fe > Zn > Cu > Cd. Tarauni Market in Kano, Nigeria, is where the samples were gathered. All levels are below WHO limits (no health risk), and the metal Cd is not detected (ND) in both (WHO, 2011). Zn > Fe in sweet potatoes; yam has the greatest value of Fe (7.35 mg/kg). The study modified the digesting process from Divya et al. (2015).

Table 3: Metal concentrations (mg/kg) (Koki et al., 2019)

Sample	Zn	Fe	Cu	Cd
Yam	7.24 ± 0.02	7.35 ± 0.12	0.68 ± 0.11	ND
Sweet potato	7.23 ± 0.01	5.30 ± 0.09	0.03 ± 0.00	ND
WHO Limits	50	43	10	0.2

Citrullus colocynthis (egusi), Irvingia wombolu (ogbono), and cassava chips sampled in open markets across five states in South-Eastern Nigeria (Enugu, Anambra, Imo, Abia, and Ebonyi) were the three staple foods that Anukwuorji et al. (2020) evaluated for heavy metal contamination (Zn, Pb, Cu, and Fe). Three seasons were sampled: wet, harmattan, and dry. Aqua regia was used to digest the samples (HNO_3:HCl, 1:3) + H_2O_2. The results indicate that the highest levels of Pb (0.039 ± 0.006 mg/g) were found in C. colocynthis, surpassing WHO/NAFDAC limits (0.002 mg/g); the highest values of Fe (0.596 ± 0.025 mg/g) and Cu (0.199 ± 0.02 mg/g) were found in C. colocynthis, and there was no significant difference in Zn levels between the foods (0.163–0.223 mg/g). While Pb was undetected in other states (with the exception of trace amounts in cassava chips), Enugu state recorded Pb values that consistently exceeded limits in all foods (e.g., 0.193 ± 0.233 mg/g in C. colocynthis) (WHO/NAFDAC). The highest values of Zn were found in cassava (0.305 ± 0.332 mg/g) and Cu in I. wombolu (0.394 ± 0.451 mg/g). Regarding seasonal variations, the greatest values of Pb (0.022 ± 0.077 mg/g in cassava, for example), Zn & Cu (higher in the wet season), and Fe (0.442 ± 0.092 mg/g in cassava) were found in Harmattan. In contrast to research that demonstrated safe Pb levels (e.g., Alwonegbe & Ikhuoria, 2007), the study concurred with studies that indicated high Pb values in foods sold across certain Nigerian markets (e.g., Nkansah & Amoako, 2011).

Using samples taken from Aliero, Kebbi State, Magajiet al., (2023) measured the levels of heavy metals (Cr, Cu, Cd, and Pb) in commonly eaten consumables and pharmaceutical goods (sugar, Milo, metronidazole antibiotic, and artemether-lumefantrine antimalarial). Dangote sugar, Nestlé Milo, Lovatem antimalarial, and Albegyl antibiotic are the commercial brands of the samples, which are made by wet digestion and FAAS detection. According to the results, Pb and Cu were not discovered, whereas Cr (sugar) and Cd (antibiotic) were determined to be below legal limits (WHO/FAO, NAFDAC). Onyeloni et al., (2022), on the other hand, found that antimalarials included Cu (11.2 ppm) and metronidazole had Pb (4.9 ppm). According to the results, there are no immediate health risks associated with the metals analyzed.

Table 4: The Mean ± SD/RSD of the Triplicate Measurements

Metal	Detected In	Concentration (ppm)	Permissible Limits (ppm)
Co	Sugar only	0.295 ± 0.004	WHO/FAO: 2.3; NAFDAC: 2.0
Cd	Antibiotic only	0.022 ± 0.002	WHO/FAO: 0.1; NAFDAC: 0.2
Cu	None	ND	WHO/FAO: 73; NAFDAC: 40
Pb	None	ND	WHO/FAO: 0.3; NAFDAC: 2.0

ND: Not detected (below instrument detection limit); RSD: Relative Standard Deviation (<10% confirms analytical precision)

Extensive petroleum exploration since 1956 has caused severe environmental degradation, including soil and water contamination. Heavy metals (Cd, Cr, Cu, Ni, Pb) from oil spills accumulate in soils and crops (e.g., Manihot esculenta), threatening food safety and human health. The soil and cassava samples were collected in wet (April–August) and dry (November–March) seasons (2021–2022). The selected study sites are Umuechem (S1), Ebocha (S2), Ofuoma (S3) and Ndashi (S4) -- control. The results in soil sample showed that Cd exceeded WHO limit (0.8 mg/kg) in all sites (e.g., dry season: Umuechem = 15.04 mg/kg), Cr value in Ofuoma (wet season) = 353.7 mg/kg (exceeded WHO limit of 100 mg/kg), Ni value in Ebocha (wet season) = 47.47 mg/kg (exceeded WHO limit of 35 mg/kg) and Cu/Pb are within permissible limits while in cassava sample Cd, Cr, Ni, Pb exceeded FAO/WHO limits, Cr (wet season) recorded 671.45 mg/kg (WHO limit 1.5 mg/kg), Pb up to 44.74 mg/kg (WHO limit 0.3 mg/kg) and control site (Ndashi) exhibited lower metals but still exceeded limits for Cd and Pb. Seasonal variations, higher metal concentrations in cassava during dry season (e.g., Cd increased by 2–4×). High Transfer Factor for Cd and Pb (e.g., Pb accumulation in cassava linked to historical leaded gasoline and oil burning) and low for Cu (retained in roots) Nneka356et al., (2022)

In a study carried by Biose et al.,(2020), heavy metal concentrations of Fe was dominant in both samples Hausa yam (*Dioscorea spp.*) and red potato (*Ipomoea batatas*) highest in unpeeled Hausa yam (283.1 mg/kg) and unpeeled red potato (337.08 mg/kg). The unpeeled yam/potato: Cd (0.16–0.27 mg/kg) and Pb (0.10–0.34 mg/kg) > CODEX limit (0.1 mg/kg) (CODEX 2011). There are significant reduction in Zn, Cu, Cr, Pb (yam) and Fe, Mn, Zn, Cr, Pb (potato) after peeling ($p<0.05$) as such consumption of unpeeled tubers poses health risks due to Cd/Pb accumulation. The samples were collected from Ikpoba Hill/Uselu markets, Benin City, washed, peeled/unpeeled, oven-dried at 105°C, ashed at 500°C and digested using HNO_3 acid. The reduced metal load in peeled tubers by 20–50% is supported by Divya et al., (2015). Hausa yam/red potato accumulated highest metals attributed to farming systems/fertilizers.

Reported by Enyindah, (2023), Analysis of Zn, Fe, and Pb concentrations in yam, cassava and potato collected from Aluu market, Rivers State, Nigeria. The samples were peeled, washed, dried, powdered, digested with aqua regia (HNO_3:HCl = 3:1) and analyzed using Atomic Absorption Spectrophotometer (AAS). The findings recorded Zn concentrations (92.39 mg/kg) in yam, cassava (69.42 mg/kg) and potato (11.04 mg/kg), Fe levels (24.78 mg/kg) in yam, cassava (30.50 mg/kg) and potato (33.87 mg/kg) and that of Pb (2.57 mg/kg) in yam, cassava (3.26 mg/kg) and potato (1.83 mg/kg). The results indicated that yam and cassava are good sources of Zn/Fe but may pose Pb toxicity risks in cassava (3.26 mg/kg), exceeded WHO/NAFDAC limits (2–3 mg/kg), Zn recorded highest value in yam (92.39 mg/kg), lowest in potato (11.04 mg/kg) and Fe highest in potato (33.87 mg/kg)

Heavy metals like Cd, Cr, Ni, and Pb have accumulated in staple crops like cassava, yam, and potato as a result of extensive petroleum exploration, causing serious contamination of the soil and water. These metals frequently surpass WHO/FAO safety limits and pose serious health risks, including toxicity and food safety issues. The validity of these results, however, is heavily dependent on sample preparation protocols. Choices about whether or not to peel, drying temperatures (like 105°C), ashing conditions (like 500°C), and digestion techniques (like HNO_3 or aqua regia) increase variability and affect metal concentration measurements, highlighting the necessity of standard operating procedures to guarantee precision and comparability across research.

Discussion

A key focus of the review is the comparison of sample preparation methods for heavy metal analysis, particularly wet and dry ashing techniques. The research by Kalagbor et al. (2014) demonstrates that wet ashing yields higher metal recovery compared to dry ashing, which suffers from limitations such as volatile metal loss at high temperatures. This aligns with the broader consensus that wet digestion methods, despite their drawbacks—such as time consumption and the use of corrosive solvents—remain more reliable for accurate heavy metal determination in food matrices. However, the review also acknowledges the need for more efficient and environmentally friendly alternatives, such as QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) which remained a vital tool for processing food and environmental samples, with only slight improvements involving new extraction solvents and sorbent materials (Shinkafi et al., 2024) and DLLME (Dispersive Liquid-Liquid Microextraction) gained widespread acceptance as a simple, fast, and miniaturized sample preparation technique. Owing to its simplicity of operation, rapidity, low cost, high recovery, and low consumption of organic solvents and reagents, it has been applied for determination of a vast variety of organic and inorganic compounds in different matrices (Saraji & Boroujeni, 2013), which could address these limitations.

Table 5: Comparison the Performance of Wet Digestion, QuEChERS, and DLLME Methods

Methods	Key process	References
Wet Digestion	Complete destruction of complex matrices	Mester & Sturgeon (2003)
QuEChERS	Rapid, simple, and cost-effective	Anastassiades et al., (2003)
DLLME	Extremely low solvent consumption, high enrichment.	Rezaee et al., (2006)

The review concludes by advocating for the development of simpler, cost-effective analytical methods like QuEChERS and DLLME to overcome the limitations of traditional techniques. Future research should focus on validating these methods across diverse food matrices and expanding monitoring programs to safeguard food safety and public health. Addressing heavy metal contamination demands a multidisciplinary approach, combining advanced analytical chemistry, environmental science, and policy-making to mitigate risks and ensure sustainable food systems.

CONCLUSION

The execution of detailed studies on the fate and levels of hazardous elements in the environment, foodstuffs and in human beings has become a major task in environmental research and especially in analytical chemistry. This has led to a demand to develop new methodology.

For years, many different methods have been used for the analysis of toxic heavy metals. The wet dissolution method (WDM) is the most popular conventional technique, but its limitations are the time required, the heat required, the slowness and the use of large volumes of corrosive inorganic solvents. These results have led to the search for more efficient and environmentally friendly techniques. Therefore, this review presents the latest sample preparation procedures (QuEChERS and DLLME methods) for the analysis of toxic heavy metals in foods and beverages. Such developments may be justified to address the limitations associated with the traditional WDM method

RECOMMENDATION

There is a need for further research to develop simple and inexpensive methods such as QuEChERS, DLLME in heavy metals analysis.

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