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SUPERIORITY OF MICROWAVE DIGESTION OVER OPEN DIGESTION FOR HIGH-ACCURACY ICP-MS EVALUATION OF HEAVY METAL CONTAMINATION

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ABSTRACT
This study holds critical significance for both analytical chemistry and public health security. Academically, it highlights how analytical results in food safety are heavily influenced by the choice of sample preparation, establishing a clear comparative baseline between open acid digestion and closed-vessel microwave digestion for complex food matrices using ICP-MS. Practically, the high concentrations of Nickel (173.48ppm) and Chromium (380.15 ppm) found in essential dietary crops like cluster beans present a severe public health concern regarding bioaccumulation in the food chain. These findings provide vital empirical data for food safety regulatory authorities (such as FSSAI) to enforce stricter monitoring of agricultural practices, soil health, and irrigation water. Furthermore, this research acts as a key stepping stone for future studies focusing on soil-to-crop heavy metal transfer factors and the implementation of targeted bioremediation strategies.
Keywords: Heavy Metals, Nickel, Chromium, Lead, Microwave Digestion, Open Digestion, ICP-MS.
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INTRODUCTION

This research article focuses on a matrix-based evaluation of heavy metal contamination using microwave digestion and open digestion methods in combination with ICP-MS. By comparing the efficiency, accuracy, and reliability of these digestion techniques across different environmental and food matrices, this study aims to identify the most suitable approach for reliable heavy metal analysis and to contribute to improved monitoring of food safety and environmental quality.[1] Heavy metals are commonly described as elements possessing high atomic weight or density, and the term is widely used to describe metallic elements and metalloids that exhibit toxicity toward living organisms and the environment. In modern usage, “heavy metals” includes not only dense metals but also toxic elements such as selenium, arsenic, and aluminium, despite differences in their physical properties. While some metals like gold are relatively non-toxic, many others pose serious environmental and biological risks due to their persistence and bioaccumulative nature.[2] Heavy metal contamination in food systems has appeared as a major global environmental and public health concern. Toxic metals, namely nickel (Ni), lead (Pb), and chromium (Cr), could enter the food chain through contaminated soil, wastewater irrigation, industrial emissions, and improper agricultural practices. Heavy metals tend to accumulate in biological tissues and are not biodegradable like organic contaminants, thereby increasing the risk of chronic exposure through dietary intake.[3] The toxicological effects of these metals are well documented. Nickel exposure at elevated levels may induce allergic reactions and has been associated with carcinogenic effects. Chromium, particularly in its hexavalent form, is highly toxic and mutagenic. Lead is especially hazardous, even at low concentrations, and could adversely impact the nervous system, kidneys, and hematopoietic system. At the cellular level, metal toxicity is often linked to the production of “reactive oxygen species (ROS)” and free radicals that could result in DNA damage, lipid peroxidation, and disruption of cellular homeostasis. These processes may also interfere with enzyme activity, protein synthesis, nucleic acid function, and membrane integrity.[4] Heavy metals also have significant implications for plant health and agricultural productivity. While trace amounts of specific metals, namely copper, iron, zinc, and nickel, are crucial for

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plant growth, excessive concentrations can disrupt physiological processes including photosynthesis, respiration, and nutrient uptake. This not only reduces quality and crop yield but also increases the risk of metal transfer into the human food chain. Vegetables, which are vital sources of essential nutrients, vitamins, and antioxidants, can act as major pathways for human exposure to heavy metals when grown in contaminated environments.[5] To safeguard public health, organisations, namely the "World Health Organisation (WHO)" and "Food Safety and Standards Authority of India (FSSAI)", have developed maximum permissible limits for heavy metals in food products. However, rapid industrialisation, urbanisation, and the widespread use of wastewater for irrigation have led to increasing instances where these limits are exceeded, particularly in developing regions.[6] Determination of heavy metal concentrations accurately in environmental and food matrices depends critically on effective sample preparation techniques. Digestion methods play a key role in converting complex matrices into analysable forms. Conventional open digestion methods, although widely used, are often associated with longer processing times, potential contamination, and loss of volatile elements. In contrast, microwave-assisted digestion offers controlled conditions, improved efficiency, and enhanced recovery of analytes. When coupled with advanced analytical approaches, namely "Inductively Coupled Plasma Mass Spectrometry (ICP-MS)", these methods enable precise and sensitive detection of trace metals.[7] Food safety is a critical global issue, particularly concerning contamination by heavy metals, namely Ni, Pb, and Cr. These metals are non-biodegradable, toxic, capable of accumulating in biological systems, leading to adverse health effects including carcinogenicity, organ damage, and neurological disorders.[8] Fruits and vegetables are vital components of the human diet; however, they can act as major pathways for heavy metal exposure due to their ability to absorb contaminants from soil, water, and air. Sources of contamination include industrial activities, wastewater irrigation, excessive use of fertilisers and pesticides, and natural geochemical processes. Among heavy metals, Chromium and Nickel are of particular concern due to their widespread industrial use and high toxicity at elevated concentrations. Lead, even at low concentrations, is highly toxic and affects multiple body systems.[9] Accurate determination of heavy metals in food samples requires effective sample preparation techniques. Microwave digestion and open digestion are widely used methods, each with distinct advantages and limitations. Microwave digestion offers controlled conditions and reduced contamination risk, whereas open digestion is simpler but may lead to higher variability.[10]

As they provide micronutrients and other beneficial compounds, vegetables are vital to human nutrition. These include vitamins, antioxidants, significant metabolites in addition to minerals like iron, copper, magnesium, calcium, iodine, sodium, zinc, and potassium.[11] WHO has developed safety guidelines for maximum permissible concentrations of heavy metals in numerous edible foods (vegetables). Such guidelines act as a global benchmark for food safety. Yet, several investigations performed in wastewater-irrigated regions have shown that cultivated vegetables under such circumstances frequently exceed these safety limits.[12] Toxic metal exposure through the food chain could cause major health issues for humans, such as neurological disorders, organ damage, elevated risk of chronic diseases. They are critical ecological and public health concerns since their presence in the environment results in environmental pollution.[13] India's FSSAI has established maximum permissible limits for heavy metals in food items. However, the rapid pace of industrialisation and urbanisation has raised concerns about agricultural product contamination.[14]

EXPERIMENTAL

Microwave Digestion

Microwaves, a form of "electromagnetic radiation (EMR)" travelling at the speed of light, enable rapid heating of materials compared to traditional conduction-based methods. This property is widely utilised in acid-assisted digestion, where food samples treated with appropriate acids are efficiently decomposed into clear solutions suitable for elemental analysis. In sealed, pressurised microwave systems, temperatures can quickly rise well beyond the solvent's normal boiling point, creating optimal conditions for the retention and analysis of volatile elements.[15,16]

Open Digestion

In chemical laboratories, open digestion was a common and simple technique for digesting organic and inorganic sample materials. It describes acid attack in open containers or low-pressure screw-top vials on

a hot plate. As a result of its flexibility in controlling digestion factors, namely temperature, reagent addition, and time, the current procedure is exceptionally beneficial for routine analysis. Yet, the ambient-pressure boiling point of the corresponding acid or acid mixture imposes a limit on maximum digestion temperatures. Risk of contamination from laboratory air, large reagent requirements, and possible loss of trace elements are further drawbacks of open vessel digestions. As a result, open vessel acid digestion wasn't regarded as "state-of-the-art (SOTA)" technology for preparing trace and ultra-trace samples.[17,18]

Materials and Methods

Sample Collection

Fresh samples of pear, mango, and capsicum were collected from Raipur mandi. Samples were washed, dried, and homogenised before analysis.

Reagents and Chemicals

All reagents were of analytical grade: "Deionised water (18.2 MΩ·cm) from Milli-Q system", "Nitric acid (HNO_3, 65%, Suprapur®)", "Hydrogen peroxide (H_2O_2, 30%, Suprapur®)".

Sample Preparation

Microwave Digestion Anton Paar

For preparation, fresh samples (250 g each) of fruits and vegetables (N = 3) were washed and cut using a ceramic knife, followed by freeze-drying using a Christ freeze dryer. The dried samples were then homogenised to obtain a uniform matrix. About 0.5g of each solid sample was taken for digestion. To each sample, an acid mixture consisting of 3mL of HNO_3 and 1mL of H_2O_2 was added. After completion of digestion, the vessels were allowed to cool, and the digested samples were diluted to a final volume of 50mL using deionised water for subsequent analysis.[19,20]

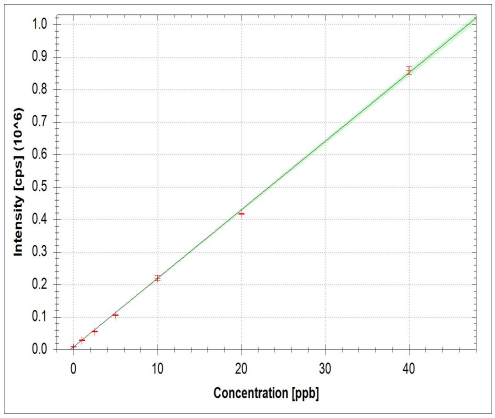
Open Digestion Borosil

For open digestion, approximately 1g of the air-dried and homogenised sample was accurately weighed and transferred into a borosilicate glass beaker or digestion flask. The sample was treated with 10 mL of concentrated HNO.During digestion, additional acid—such as HNO_3 and, if required, $HClO_4$ or H_2SO_4 was added intermittently for ensure complete breakdown of the sample matrix. The digestion process was continued until a clear solution was obtained, indicating complete digestion. The solution was then cooled, filtered if necessary, and diluted to a final volume of 50mL utilizing deionized water before analysis.[21,22]

Instrumental Conditions

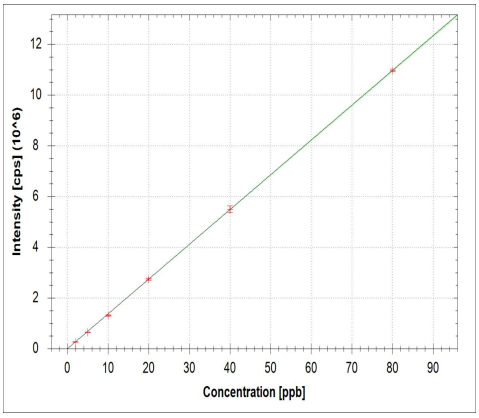
Instrument: Microwave Digestion System; Temperature Program:; Ramp to 180–200°C in 10–15 minutes Hold for 15–20 minutes; Cooling time: 15–20 minutes

60Ni (KED)



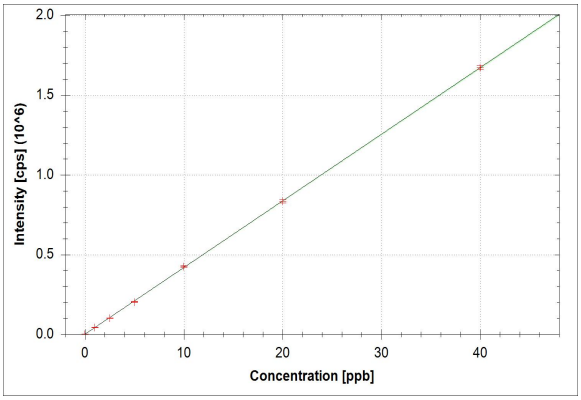
f(x) = 21068.6539*x + 8259.8410
R^2 = 0.9995, BEC = 0.392 ppb
Fig.-1(a): Calibration Curve of Nickel

207Pb (KED)



f(x) = 137064.7707*x + 1567.9307
R^2 = 0.9999, BEC=0.011 ppb
Fig.-1(b): Calibration Curve of Lead

52Cr (KED)



f(x) = 41779.2803*x + 1328.8769
R² = 1.0000, BEC = 0.032 ppb
Fig.-1(c): Calibration Curve of Chromium

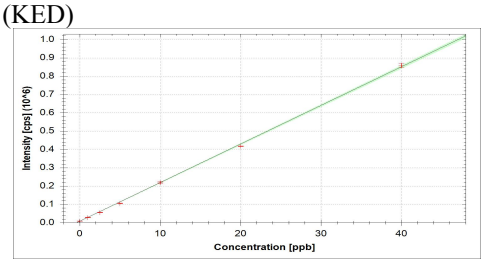
Table-1: Analysis Results According to Microwave Digestion

S. No.	Sample Name	Nickel		Lead		Chromium	
		Result (mg/kg)	FSSAI Limit (mg/kg)	Result (mg/kg)	FSSAI Limit (mg/kg)	Result (mg/kg)	FSSAI Limit (mg/kg)
1.	Pear	1.064	1	<	2.5	1.232	1
2.	Mango	1.849	1	<	2.5	1.399	1
3.	Capsicum	1.246	1	<	2.5	1.148	1

Instrumental Conditions

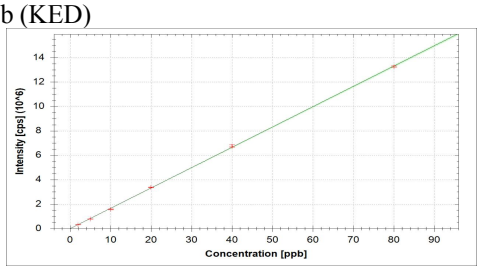
Instrument: Open Digestion Method; Heated on hot plate at 120–150°C; Evaporation to near dryness; Filtration: Whatman filter paper

60Ni (KED)



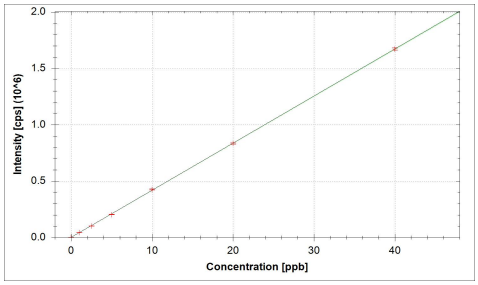
f(x) = 21068.6539*x + 8259.8410
R² = 0.9998, BEC = 0.010 ppb
Fig.-2(a): Calibration Curve of Nickel

206Pb (KED)



f(x) 166286.0580*x + 1603.3631
R² = 0.9995, BEC = 0.392 ppb
Fig.-2(b): Calibration Curve of Lead

52Cr (KED)



f(x) = 41779.2803*x + 1328.8769
R² = 0.9997, BEC = 0.034 ppb
Fig.-2(c): Calibration Curve of Chromium

Table-2: Analysis Results According to Open Digestion

S.No.	Sample Name	Nickel		Lead		Chromium	
		Result (mg/kg)	FSSAI Limit (mg/kg)	Result (mg/kg)	FSSAI Limit (mg/kg)	Result (mg/kg)	FSSAI Limit (mg/kg)
1.	Pear	25.908	1	<	2.5	47.831	1
2.	Mango	173.488	1	<	2.5	380.157	1
3.	Capsicum	7.275	1	<	2.5	14.293	1

RESULTS AND DISCUSSION

Concentrations of heavy metals-Ni, Pb, Cr in fruit and vegetable samples were determined using microwave digestion and open digestion methods followed by ICP-MS analysis in KED mode. In both approaches, the calibration curves displayed excellent linearity, with "correlation coefficients (R^2)" varying from 0.9995- 1.0000, as shown in Tables-1 and 2, confirming high analytical accuracy and reliability. The low background equivalent concentrations (BEC) and "limits of detection (LOD)" demonstrate high sensitivity of the method for trace metal determination. In the microwave digestion method, nickel concentrations in pear (1.064 mg/kg), mango (1.849 mg/kg), and capsicum (1.246 mg/kg) exceeded the FSSAI permissible limit of 1 mg/kg. Among these, mango exhibited the highest nickel concentration, indicating a potential risk of dietary exposure. Lead concentrations in all samples were below the detection limit (<2.5 mg/kg), suggesting negligible contamination and compliance with safety standards. Chromium levels ranged from 1.148 to 1.399 mg/kg, which are close to or slightly above the permissible limit, indicating moderate contamination possibly influenced by environmental parameters, namely soil composition, irrigation water, and agricultural inputs.

In contrast, the open digestion method showed significantly higher concentrations of nickel and chromium in all analysed samples. Nickel levels were extremely high in Pear (173.488 mg/kg), followed by Mango (25.908 mg/kg) and Capsicum (7.275 mg/kg), greatly exceeding permissible limits and indicating severe contamination. Similarly, chromium concentrations were markedly elevated, with Mango (380.157 mg/kg) showing the highest level, followed by Pear (47.831 mg/kg) and Capsicum (14.293 mg/kg), as shown in Fig.-1 and 2 (a, b and c). These elevated concentrations may be attributed to environmental contamination sources such as industrial discharge, polluted irrigation water, or excessive use of agrochemicals. Lead levels remained below the detection limit in all samples, consistent with results obtained from microwave digestion. A comparison between the two digestion methods indicates that open digestion resulted in significantly higher detected metal concentrations than microwave digestion. This difference may be due to potential contamination during sample handling, loss of control over digestion conditions, or matrix effects. Microwave digestion, being a closed system, minimises external contamination and provides more consistent and reliable results.

CONCLUSION

The study confirms that ICP-MS is a highly sensitive and precise procedure to determine trace heavy metals in food samples. Nevertheless, results reveal concerning levels of nickel and chromium contamination, particularly in samples analysed using the open digestion method. While lead contamination was found to be negligible, the elevated concentrations of nickel and chromium may pose potential health risks to consumers. Microwave digestion is recommended as a more reliable and accurate sample preparation method due to its controlled environment and reduced risk of contamination. The outcomes emphasise the significance of constant evaluation of heavy metals in food products and the requirement for stricter control of environmental pollution and agricultural practices. Ensuring safe cultivation conditions and reducing contamination sources are essential to protect public health and maintain food safety standards.

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CONFLICT OF INTERESTS

The authors declare that there are no conflicts of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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