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Original article

Using Ionic Coupled Plasma (ICP) for estimating some Heavy Metals and elements in some Tea Samples and calculating their Health Risk Assessment

Hamad Hasan*[1], Marwa Khalifa[2], Monira Saleh[1]

[1]*Chemistry Department, Faculty of Science, Omar Al-Mukhtar University, Libya*
[2]*Higher Institute of Medical Sciences and Technology, Al-Marj, Libya*
Corresponding Email: hamad.dr@omu.edu.ly

Abstract
Tea is one of the most widely consumed beverages globally, including in Libya. This study investigates the concentrations of heavy metals in twelve tea samples available in Libyan markets using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The health risks associated with these metals were evaluated using the Target Hazard Quotient (THQ) and Hazard Index (HI). The results of this study recorded that some metals were detected in the studied Tea samples, including: Fe, Zn, Mn, Al, Cu, Cr, Ni, Ca. On the other hand, the metals of Pb, Co, Cd, Ag. As, and Sb were not detected in the studied samples. The contents of detected metals were fluctuated in the ranges of (0.24 – 1.08), (0.22 – 2.85), (1.68 – 6.69), (1.80 – 6.02), (0.060 – 0.13), (0.010 – 0.023), (ND – 0.022) and (16.97 – 27.87 μg/g), for Fe, Zn, Mn, Al, Cu, Cr, Ni and Ca, respectively. The higher concentrations were obtained for Mn, followed by Cr, then Fe. Results showed elevated levels of manganese (Mn) exceeding the Maximum Residue Limits (MRLs), while other metals such as zinc (Zn), iron (Fe), aluminum (Al), copper (Cu), chromium (Cr), nickel (Ni), and calcium (Ca) were within acceptable ranges. However, THQ and HI values for Mn and Cr in some samples exceeded safe thresholds, indicating potential health risks. These findings highlight the need for stricter quality control and monitoring of imported tea products in Libya.
Keywords: Heavy Metals, Tea, ICP-MS, Health Risk Assessment, Libya, THQ, HI

Introduction

Tea**,** a widely consumed beverage second only to water, plays a significant role in maintaining intestinal health by influencing the gut microbiota and bolstering immunity against intestinal disorders. Additionally, tea possesses antioxidant properties that protect cell membranes from oxidative damage. However, the cultivation of tea plants can be susceptible to various diseases and pests. To mitigate these challenges and ensure optimal quality, farmers often rely on pesticides [1]. Pesticides, although crucial for pest control and plant health, can have adverse effects on human health when used excessively. The cultivation, processing, and storage of tea leaves, especially black tea, can result in the accumulation of heavy metals. The World Health Organization (WHO) has established stringent guidelines for the permissible levels of heavy metals in tea. To guarantee the safety of consumers, rigorous monitoring of these substances in tea samples sourced from both tea gardens and commercial brands is imperative. This research endeavors to quantify the concentration of heavy metals in tea samples and evaluate the associated health risks. By conducting a comprehensive analysis, we aim to contribute to ensuring the safety and quality of tea products available to consumers.

The concentration of different metal components in tea can vary significantly, potentially impacting human health [2]. The accumulation of heavy metals in tea leaves can be attributed to both natural factors, such as soil contamination, and anthropogenic factors, such as the use of pesticides and fertilizers [3]. While some trace metals, like chromium, iron, cobalt, nickel, and zinc, are essential for normal physiological functions, other heavy metals, such as Pb, Ca, and arsenic, are not only non-essential but also toxic [4]. A critical biological characteristic of metals is their propensity for bioaccumulation. Consequently, bioaccumulation is a key factor in risk assessment strategies. For example, the calculation of the available percent of Aluminum (Al) and Zinc (Zn) in tea consumed by humans revealed that tea can contribute up to 37.2% of the daily dietary intake of Al, while only a small fraction, approximately 1.78%, is absorbed by the intestines [5].

Inductively coupled plasma mass spectrometry (ICP-MS) is a powerful technique for analyzing analytes that employs an inductively coupled plasma to ionize a sample. This process atomizes the sample, generating atomic and small polyatomic ions, which are subsequently detected. ICP-MS is renowned for its capability to detect trace metals and various non-metals in liquid samples with exceptional sensitivity. Furthermore, it can differentiate between isotopes of the same element, making it a versatile tool for isotopic labeling studies [6]. ICP-MS offers superior speed, precision, and sensitivity compared to atomic absorption spectroscopy. However, compared to other mass spectrometry techniques such as TIMS and GD-MS, ICP-MS introduces various interfering species, including argon from the plasma, air contaminants, and impurities from glassware and cones [7].

Tea is a staple beverage in Libyan households, consumed daily across all age groups. However, tea leaves can accumulate heavy metals from soil, water, and atmospheric deposition. Chronic exposure to these

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metals poses significant health risks. The detection of chemical compounds was carried out on different samples in Libya such as hydrocarbon compounds [8-14], radioactive elements [15-19], or plants and their applications [20-55]. The metals have importance if they are used as complexes for different uses, but they also have other negative effects on the environment, different studies were carried out on different samples [56-94]. This study aims to quantify heavy metal concentrations in tea samples and assess the associated health risks.

Methods

Sampling

Twelve tea samples were collected from various Libyan markets. The samples are given in (Table 1).

Table 1. The studied Tea samples

Sample No	Sample Name	TeaType
1	Alagzalin	Black Tea
2	Ahmed Tea	
3	Zahra	
4	AlSafina	
5	Al wadg	
6	Nesma	
7	AlBaraka	Green Tea
8	Algzalin	
9	AlNajee	
10	AlBaraka	
11	Al Nabt	
12	Nessma	

Heavy metal determination

Samples were digested using concentrated nitric acid (HNO_3). 0.5 grams of each sample was added to a beaker containing 50 ml of distilled water, then 5 ml of concentrated nitric acid was added. The mixture was heated on a hot plate until near dryness, then cooled and filtered. The filtrate was brought to a volume of 100 ml in a measuring flask with distilled water (60-61). and analyzed using ICP-MS. Health risk assessment was conducted using THQ and HI formulas based on daily intake and reference doses.
Risk assessment was also done by calculating the target risk quotient (THQ), designed by the US Environmental Protection Agency which was designed by the US Environmental Protection Agency to determine safe levels of frequent long-term exposure to chemical pollutants. The THQ is a relationship between the measured concentration and the oral reference dose, weighted by the length and frequency of exposure, amount ingested, and body weight. A THQ reading of 1 or above indicates a health risk.

$$\mathbf{THQ = ([\ EFr * ED\ tot * IFR * C]) / ([RfD0 * BWa * ATn]) * 10^{-3}}$$

Where:

EFr: the exposure frequency (365 days/year); EDtot: is the exposure duration (80 years for the Italian population; it was the same for the Pakistani population as determined from survey results) according to ISTAT 2013. IFR: is the food ingestion rate (g day-1); C: is the concentration (μg g-1); RfDo: is the oral reference dose (μg g-1 day-1). BWa: is the adult body weight (60 kg). ATn: is the average time for non-carcinogens (it is equal to EFr × EDtot). The health risk index imposes a level of risk due to the presence of pesticides and metals in tea samples.

Results

Heavy metal contents

Some of heavy metals were detected in this study in tea samples by I.C.P, The metals including: Fe , Zn , Mn, Al, Cu, Cr, Ni, Ca, On the other side the metals of pb, Co, Cd, Ag, As and Sb were not detected in the studied samples, the contents were given in the (Tables 2-3) and (Figures 1-2). The contents of detected metals were fluctuated in the ranges of (0.24 – 1.08), (0.22 – 2.85), (1.68 – 6.69), (1.80 – 6.02), (0.060 – 0.13), (0.010 – 0.023), (ND – 0.022) and (16.97 – 27.87 μg/g), for Fe, Zn, Mn, Al, Cu, Cr, Ni and Ca, respectively. The higher concentrations were obtained for Mn, followed by Cr, then Fe. By comparing the obtained contents with MRL (Maximum Residual Limit) for some metals (Fe, Zn, Mn, Co, Cr), the obtained results of heavy metals in this study were low comparing with (MRL) , except for manganese contents, where the results of this study recorded contamination and toxicity the studied samples by Mn, where its values (3.41 ± 1.87 μg/g) are high than MRL values, (Table2).

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Table 2. The contents of Fe, Zn, Mn, and Al in tea samples.

Metal / Sample No	Fe	Zn	Mn	Al
1	0.41	0.69	1.74	2.35
2	0.36	0.55	1.68	2.02
3	0.88	0.47	4.03	4.14
4	1.08	0.79	3.96	2.59
5	0.41	2.85	1.75	2.19
6	0.37	0.42	2.09	3.03
7	0.71	0.34	6.48	2.96
8	0.48	0.40	6.69	6.02
9	0.27	0.23	1.83	2.31
10	0.64	1.44	5.26	3.52
11	0.24	0.22	2.10	2.34
12	0.72	0.29	3.35	1.80
±SD	0.259	0.748	1.87	1.17
Average	**0.54**	**0.72 1**	**3.41**	**2.93**

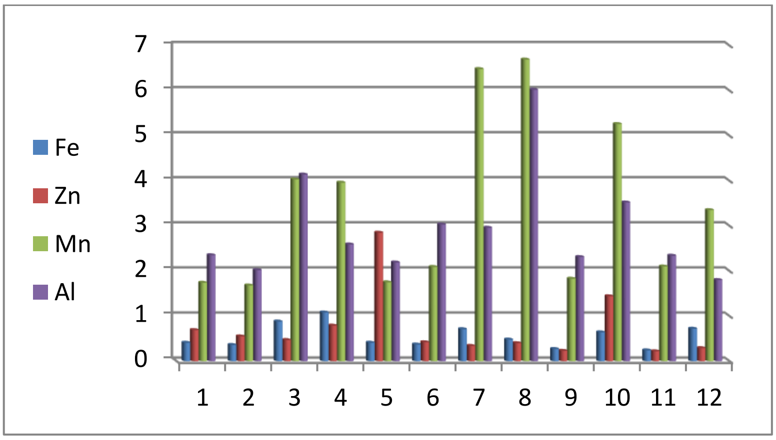

Figure 1. The contents of (Fe, Zn, Mn, and Al) of the studied tea samples

Table 3. The contents of Cu, Cr, Ni and Ca in the studied tea samples.

Metal / Sample No	Cu	Cr	Ni	Ca
1	0.11	0.011	ND	25.80
2	0.12	0.010	ND	21.83
3	0.08	0.012	0.015	26.11
4	0.062	0.013	0.014	19.16
5	0.10	0014	ND	27.87
6	0.13	0.012	0.016	22.83
7	0.07	0.023	0.022	22.05
8	0.060	0.013	0.020	21.43
9	0.061	0.014	ND	17.93
10	0.10	0.021	0.013	20.6
11	0.063	0.011	ND	16.97
12	0.060	0.012	0.011	26.58
±SD	0.025	4.037	0.008	3.53
Average	**0.084**	**1.17**	**0.009**	**22.43**

ND: Non detected

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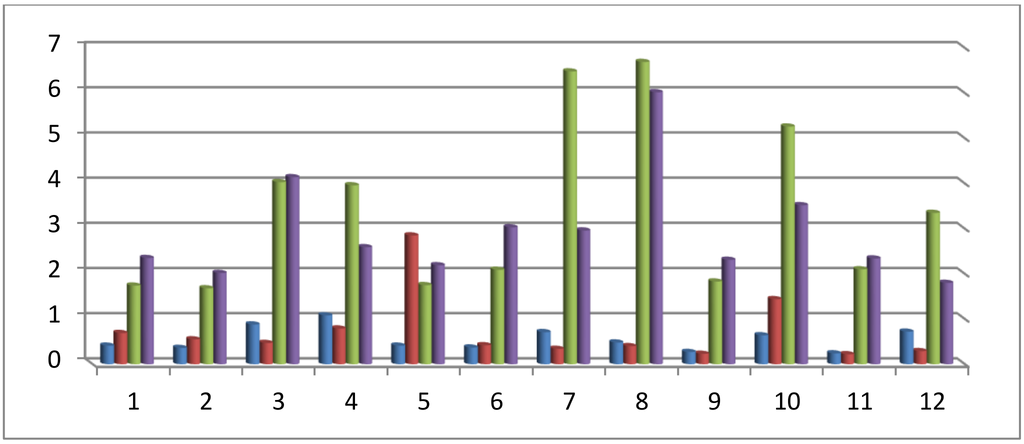

Figure 2. The contents of (Cu, Ni, and Cr) of the studied tea samples

Table 3. Compared the detected heavy metals with MRL values

Metal	Zn	Fe	Cu	Mn	Cr	Ni
Concentration	0.72 ± 0.7	0.54 ± 0.25	0.084± 0.025	3.41± 1.87	1.17± 0.4	0.009 ±0.008
MRL s	100	500	0.01	0.16	2.3	-

MRL: Minimum Residual Limit

By using diagrams (Figures 3 - 14) to evaluate higher contents of detected heavy metals in the studied samples, the results recorded all samples containing high concentrations of Mn, then the other metals were varied from one sample to another. Also, the results recoded variations of the percentage distribution of metals between the studied, the major metals in tea samples are Aluminum (Al) and manganese (Mn), besides the presence of some relatively high amounts of Zn and Fe. The Tea samples numbers of (1, 2, 3, 6, 9 and 11) showed higher contents of Al percentage values of (44, 43, 43, 50, 49 and 47 %), also there are high percentage values of Mn (33, 35, 42, 35, 39 and 42 %) from the total amount of detected heavy metals in tea samples of this study. On the side, the higher contents of Mn were recorded in the samples of (4, 5, 7, 8, 10, and 12) with values of (47, 60, 49, 48, and 54 %). Small percentage values of the other metals, Zn, Cu, Ni and Cr were observed. Relative increase in Fe contents as was recorded in tea samples.

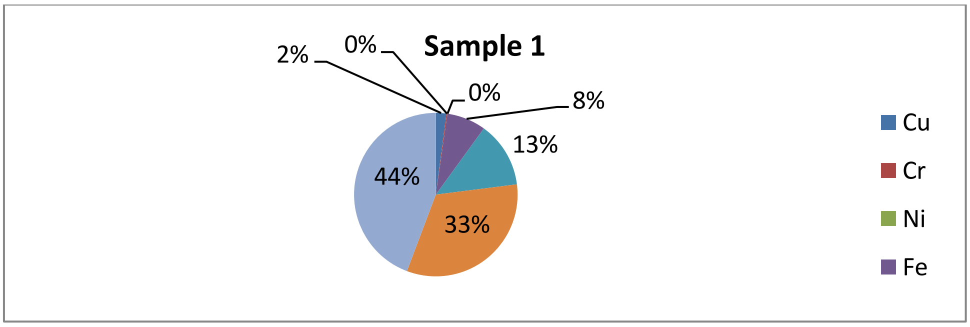

Figure 3. Pia gram of metal distribution of sample 1

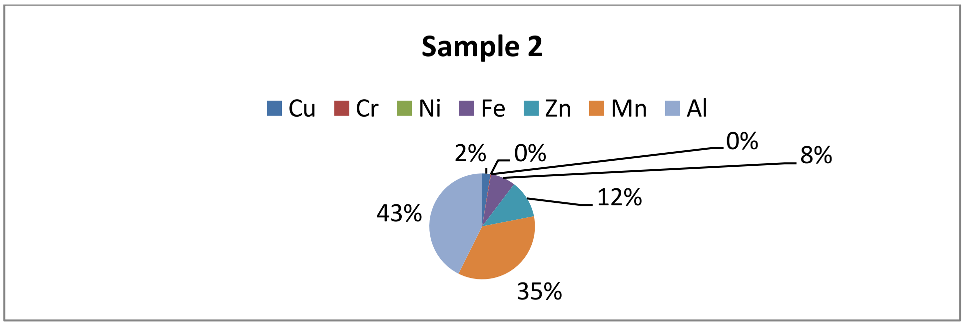

Figure 4. Pia gram of metal distribution of sample 2

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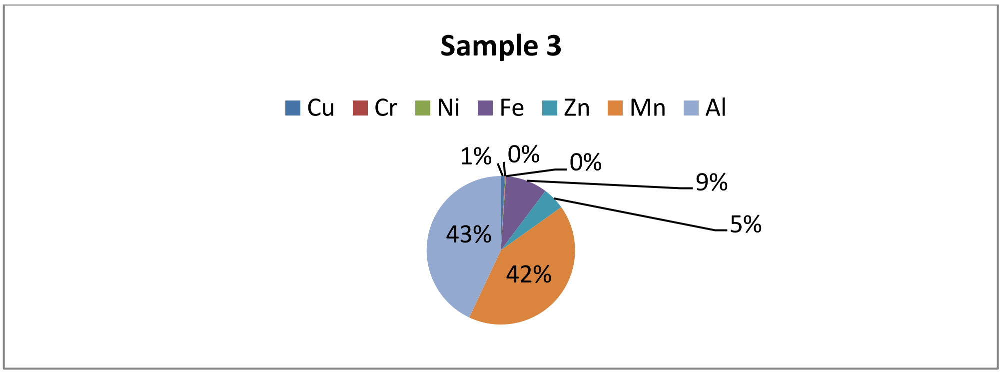

Figure 5. Pia gram of metal distribution of sample 3

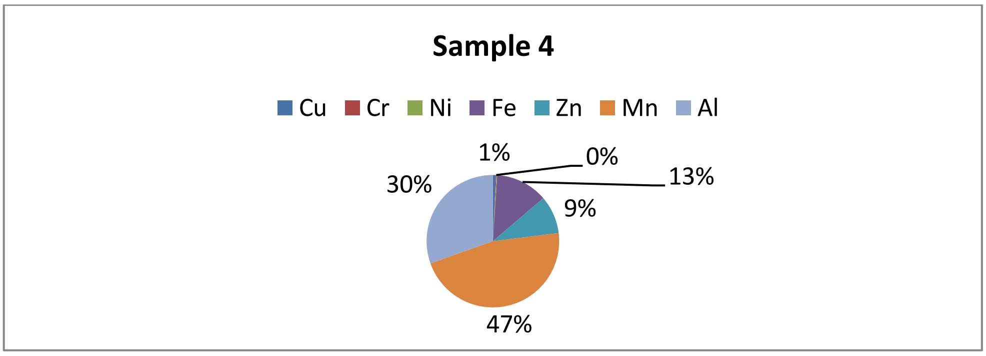

Figure 6. Pia gram of metal distribution of sample 4

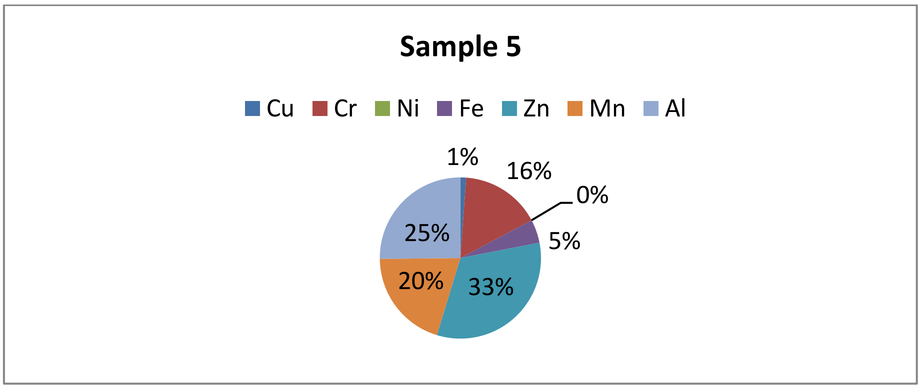

Figure 7. Pia gram of metal distribution of sample 5

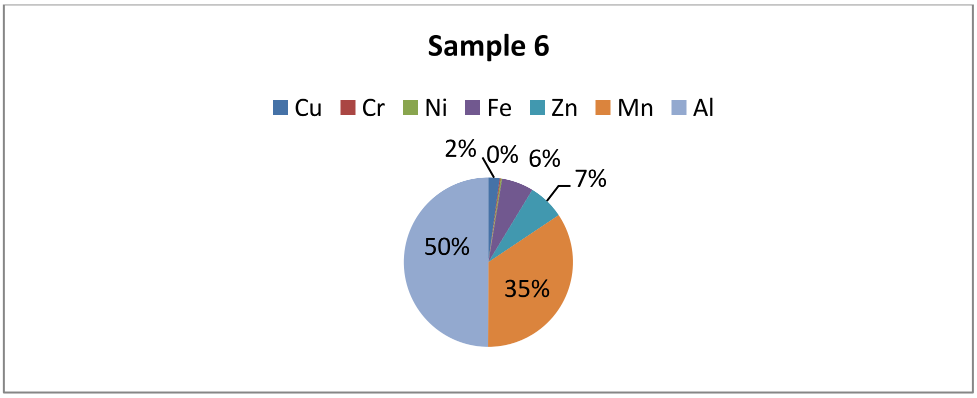

Figure 8. Pia gram of metal distribution of sample 6

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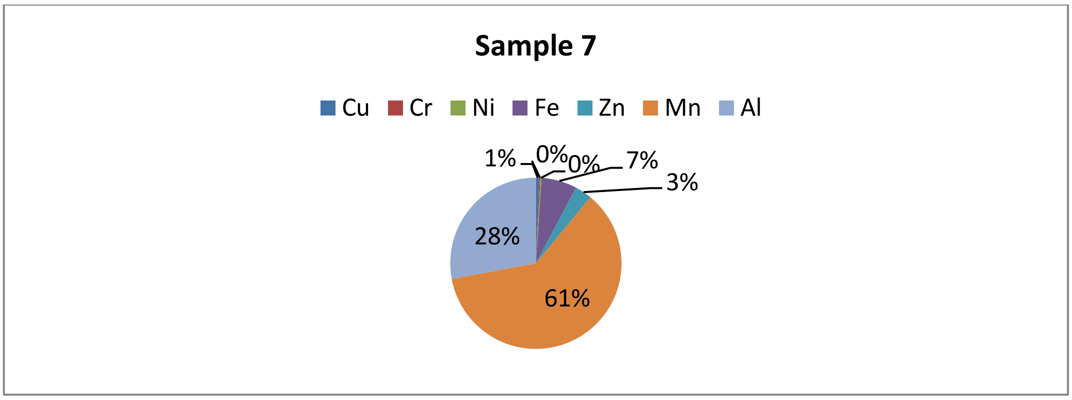

Figure 9. Pia gram of metal distribution of sample 7

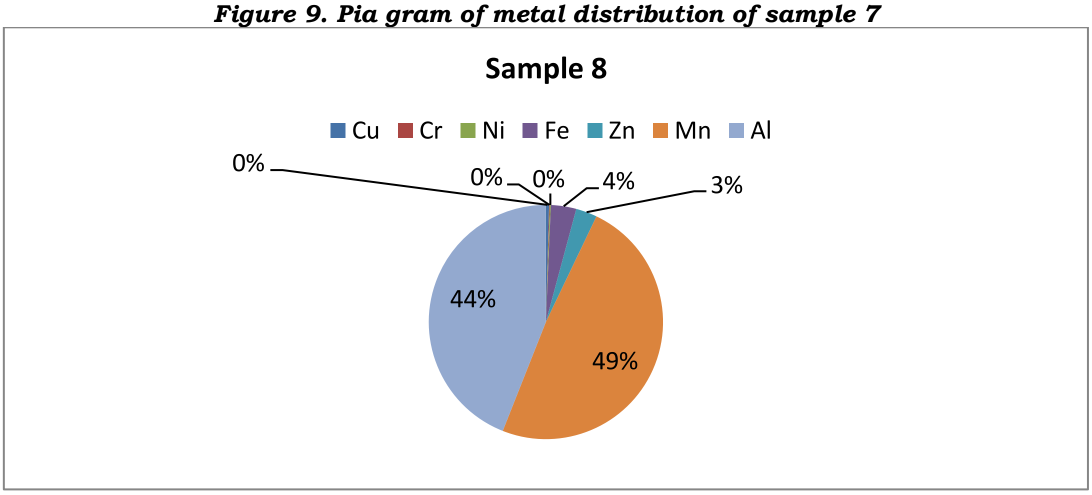

Figure 10. Pia gram of metal distribution of sample 8

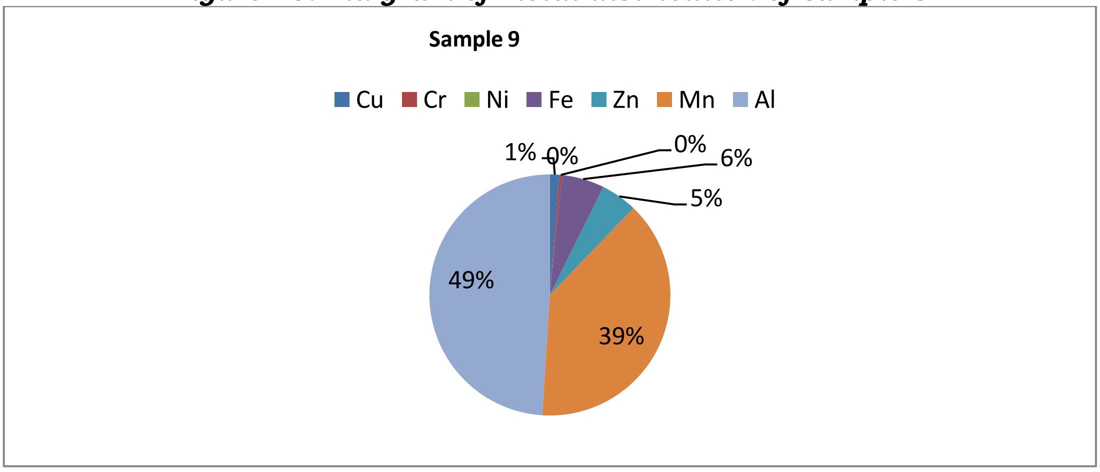

Figure 11. Pia gram of metal distribution of sample 9

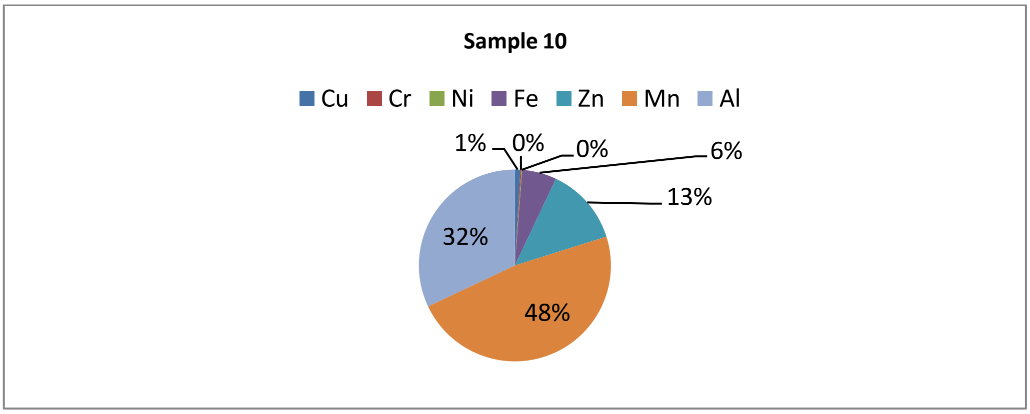

Figure 12. Pia gram of metal distribution of sample 10

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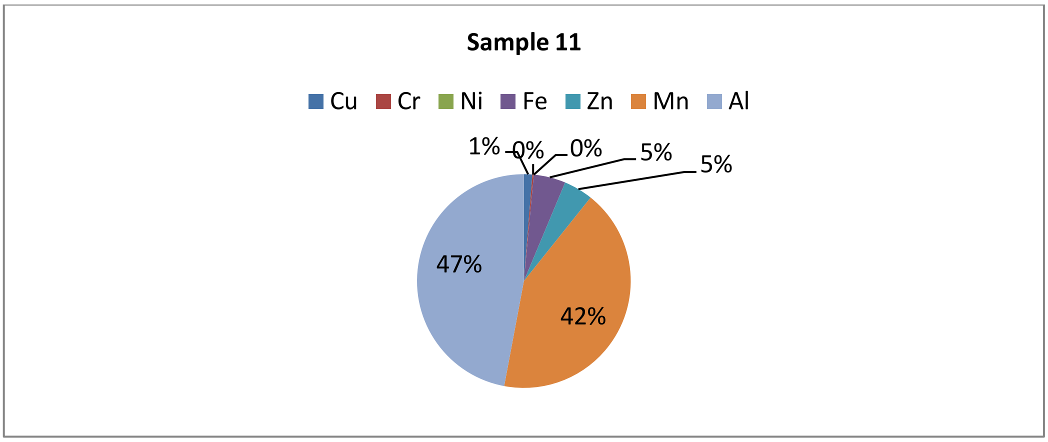

Figure 13. Pia gram of metal distribution of sample 11

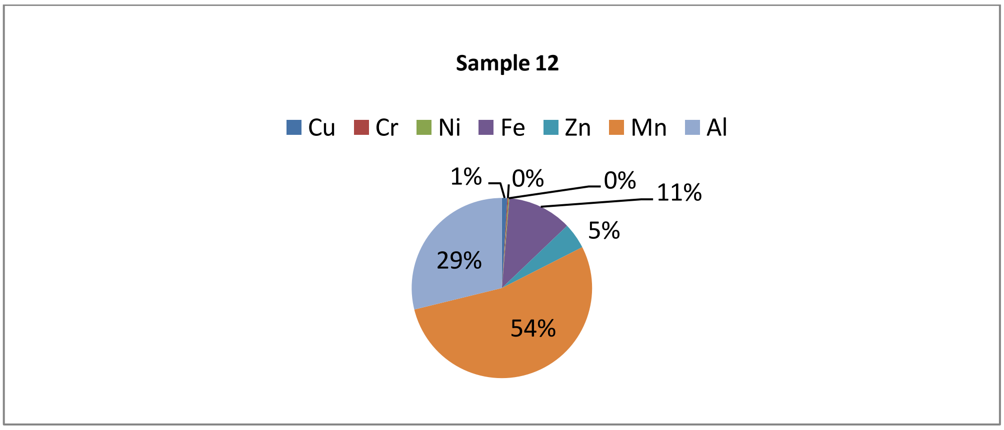

Figure 14. Pia gram of metal distribution of sample 12

Discussion

This study aimed to detect and quantify the levels of heavy metals in a diverse range of tea samples. These samples were collected through a rigorous random sampling process, encompassing both locally sourced tea and branded products. By analyzing these samples, the study sought to provide valuable insights into the potential exposure to heavy metals through tea consumption and to inform strategies for ensuring the safety and quality of this popular beverage. The analyzed tea samples exhibited varying levels of heavy metal accumulation. For instance, numerous studies have documented the presence of copper in tea samples originating from diverse geographical locations, including Iran, Lithuania, and China [95], potassium in Pakistani tea, and lead in Tunisian tea [96]. These findings underscore the metal-accumulating potential of tea plants. Additionally, other studies have reported the accumulation of aluminum and iron in tea leaves.

These observations highlight the potential for heavy metal contamination in tea, emphasizing the importance of stringent quality control measures to safeguard consumer health and well-being. The concentration of Al, Mn, and Fe in the Libyan tea samples was notably higher compared to other metals. This elevation may be attributed to the elevated metal content of the soil in the tea-producing regions and potentially to selective absorption by the tea plant. The low solubility of these metals suggests they are bound in less soluble complexes. The variability in metal content across different tea brands can be linked to their geographical origin, influenced by soil composition and leaching characteristics. Long-term tea cultivation can lead to soil acidification and increased levels of bioavailable heavy metals, potentially contributing to higher heavy metal accumulation in tea leaves [97]. The observed variations in the current study may be attributed to differences in the agro-climatic conditions of the imported tea. While the toxic effects of heavy metals have been extensively documented, the therapeutic efficacy of tea is contingent upon the concentration of its bioactive compounds.

It is imperative to maintain a delicate balance between a concentration that is sufficiently high to elicit beneficial effects and one that is low enough to avoid potential toxicity. A narrow therapeutic window exists within which the desired therapeutic outcomes can be achieved without compromising safety. The aqueous tea extracts contain appreciable quantities of metal ions that can potentially contribute to daily dietary intake. However, these levels remain below the established daily dietary requirements for humans. The presence of metal ions in tea extracts is a natural consequence of the plant's interaction with the soil and water environment [98]. These ions, such as Fe, K, Mn, and Ca, are essential micronutrients that play crucial roles in various physiological processes. While the concentrations of these ions in tea extracts may

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vary depending on the type of tea, growing conditions, and processing methods, they generally fall within safe limits for human consumption. The accurate quantification of these elements in beverages, water, food, plant, and soil samples is therefore a crucial task. Green leafy vegetables represent a significant dietary source of these metals.

To ensure the safety and efficacy of tea consumption, it is recommended to regularly consume aqueous extracts for their essential nutrients. However, it is crucial to monitor and analyze tea samples from Libya and other regions to prevent exceeding daily intake limits of potentially harmful substances. Geographical variations in heavy metal concentrations have been observed among different tea brands, highlighting the importance of regular testing and quality control measures [99]. The observed inconsistencies in metal concentrations across diverse growing regions can be attributed to the inherent differences in the underlying chemical and physical properties of these areas. These variations may include soil type, mineral content, water quality, climate, and agricultural practices, all of which can significantly influence the accumulation of metals in plant tissues. Routinely consuming aqueous extracts for essential nutrients is also recommended. Regular monitoring and frequent analysis of tea in Nigeria and other regions are necessary to prevent exceeding daily intake limits.

In conclusion, the geographical origin significantly influenced the heavy metal concentrations across different tea brands. Notably, we observed substantial differences in the water solubility of these metals. Previous research has reported similar variations in metal concentrations among different tea brands. The observed wide range of metal concentrations could be attributed to the distinct chemical and physical properties of the growing regions [98-99].

Health risk assessments

To assess potential health risks associated with heavy metal exposure, THQ and HI were calculated. A THQ or HI value exceeding 1 indicates a potential health risk. Cobalt was detected in one sample, collected from a source containing Zn, Mn, and Cr at concentrations of 5.19, 1.27, and 1.19 ppm, respectively. The calculated THQ for this sample exceeded 1, suggesting a potential health risk associated with cobalt consumption. Chromium was detected in tea samples with a mean concentration of 1.19 ppm, which falls within acceptable limits. The THQ limit was exceeded in our study [1], levels of cobalt and manganese, both of which are known to be highly toxic and may pose significant long-term health risks.

Table 4. The results of the THQ values

Metal	THQ
Cu	0.138
Cr	1.19
Ni	0.014
Ca	37.003
Fe	0.89
Zn	1.27
Mn	5.18
Al	4.83

Mn levels exceeded MRLs in several samples, with different concentrations of Zn, Fe, Al, Cu, Cr, Ni, and Ca within permissible limits. THQ values for Mn and Cr surpassed 1 in some samples, indicating potential non-carcinogenic risks. HI values also exceeded 1 in select samples, suggesting cumulative health concerns.

Conclusion

The heavy metals recorded in this study include different types of heavy metals such as Fe, Zn, Cr, Mn, Ni, and Cu, besides the presence of different concentrations of Al and Ca. The risk indexes showed an increase in the values of Cr, Zn, and Mn compared with safety limits.

Acknowledgment

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Conflict

No conflict of the results of this study with other studies.

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