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**ANALYSIS OF SELECTED SOIL SAMPLES FROM LAPAI, NIGERIA USING
MICROWAVE PLASMA ATOMIC EMISSION SPECTROSCOPY**

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ABSTRACT

Microwave plasma atomic emission spectroscopy is a multi-elemental, less expensive instrumental technique introduced as an alternative to more costly inductively coupled plasma techniques. This technique offers a lower maintenance cost, relatively lower cost, good speed, and detection. In this study, the concentrations of Cr, Cu, Fe, Mg, and Zn in selected soil samples were determined using this technique. Three regions were randomly selected for investigation within Cece Village. Samples were collected from the top and lower soil horizons in areas A and B and river sediments using the random grab method with appropriate sampling tools. The samples were processed in the laboratory, including sieving, pulverizing, and digestion. The results of this study revealed that trace concentrations in mg/kg of chromium had the highest value (1.50 ± 0.08) and lowest value (0.50 ± 0.03), copper had the highest value (28.50 ± 1.50) and lowest value (1.12 ± 0.06), iron had the highest value (303.51 ± 11.01) and lowest value (152.04 ± 7.03), magnesium had the highest value (273.52 ± 11.41) and lowest value (1.50 ± 0.08), and zinc had the highest value (170.51 ± 51) and the lowest value (162.12 ± 7.04). The findings of this study revealed that the concentrations of the elements found in various soil samples were within permissible limits. This suggests that the sampled region is safe for crop cultivation and other agricultural activities.

Keywords: Concentration, MP-AES, Plasma, River sediment, Soil horizons,

INTRODUCTION

Anthropogenic activities such as mining, refining, smelting, and agricultural operations have led to an increasing rate of environmental pollution by metals (Sut-Lohmann et al. 2020; Fajana et al. 2020; Begum et al. 2020). Microwave plasma atomic emission spectroscopy (MP-AES) is an instrumental technique based on atomic emission spectroscopy, which was introduced as an alternative to inductively coupled plasma (ICP) atomic emission spectroscopy (AES) and mass spectrometry (MS) (Balaram, 2020). It is a self-sustaining



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pressure instrument that uses nitrogen from air using a nitrogen generator. Other advantages of MP-AES include multi-elemental capacity, low maintenance cost, relatively low cost, good speed, and detection. (Balaram, 2020; Evans et al., 2013). However, these limitations include spectral and matrix interferences, which can be resolved by using multiple spectral lines and the adoption of standard addition methods for analysis (Varbanova and Stefanova, 2015; Olivera, Gonzalez & Nogueira, 2015; Thaler et al. 2018).

Recent research has focused on the use of MP-AES in a variety of fields, including forensics, energy, agriculture, food, health, and the environment (Samarina et al. 2018; Amoakwah et al. 2020; Varga et al. 2020; De Sa et al. 2020; Laura et al. 2020; Chen et al. 2022). Results from MP-AES analyses were found to be superior to those of AAS and, to some extent, comparable to ICP-AES (Balaram et al., 2013; Balaram et al., 2014). The limits of detection of MP-AES are 1.4, 1.3, 0.4, and Pb 2.1 ng/ml. MP-AES has been employed in a variety of studies for the analysis of minor and major elements in geological samples of soil, sediment, rock, and water samples (Balaram et al., 2013; Balaram et al., 2014; Niedzielski et al., 2015; Bastaskoti et al., 2019).

Based on the study by Thirumdas et al. (2019), MP-AES is one of the suggested methods for determining food quality. Malbec wine's geographical origin was determined using MP-AES data from metal analysis of wines (Nelson et al., 2018) and Althoff et al. (2017) identified a few specific metals in cough syrup and reported sample analysis precision between 2.5 and 8.0

MP-AES has also been utilized to monitor the quality of drinking water (Rajput et al. 2019; Kumar et al. 2017) and to analyze seawater (Maxwell et al. 2015; Leggat et al. 2019), industrial effluent, and wastewater (Shanmuganthan et al. 2016). Agricultural soils are often analyzed to monitor heavy metal concentrations and assess soil fertility and nutrient deficiencies (Olivia et al., 2019; Garba, Buhari & Samaila, 2020). MP-AES was less common than AAS or ICP. This is due to variables such as the limited number of publications on sample preparation and institutions' preference for a more sensitive ICP (Balaram, 2020). MP-AES was used in this study to analyze specific soil samples from Lapai because it is highly sensitive, allows simultaneous determination of range of elements that are usually determined environmental studies. Moreso, it is cost effective compared with inductively plasma spectroscopy.



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Heavy metal pollution in agricultural soils is a significant environmental concern due to its potential adverse effects on soil health, plant growth, and food safety. Metals such as chromium (Cr), magnesium (Mg), zinc (Zn), iron (Fe), and copper (Cu) can accumulate in soils through various anthropogenic activities, including industrial processes, agricultural practices, and the use of contaminated water for irrigation. Understanding the extent and impact of these pollutants is crucial for developing effective management strategies to mitigate their harmful effects (Paul et al., 2015; Srivastava et al., 2017; Ahmadi et al., 2019; Xiang et al., 2021). Heavy metals like Cr, Cu, Zn, and Cd in agricultural soils often originate from anthropogenic activities such as the application of fertilizers, herbicides, and pesticides. In some regions, irrigation with treated industrial effluents significantly increases the concentration of heavy metals like Cr, Cu, Zn, and Pb in soils. This contamination can lead to altered biochemical, physiological, and metabolic processes in plants, affecting their growth and overall health (Paul et al., 2015; Ahmadi et al., 2019). The presence of heavy metals in soil can disrupt soil microflora, which is essential for maintaining soil health and fertility (Srivastava et al., 2017). This pollution in soils poses potential ecological risks, with certain metals like Hg and As contributing significantly to these risks. The contamination of crops by heavy metals from soil can lead to human health risks, with metals like As in crops posing a notable threat (Xiang et al., 2021). Spatial distribution studies reveal that certain areas are heavily contaminated with multiple heavy metals, often correlating with specific anthropogenic activities such as industrial effluent irrigation (Paul et al., 2015). Pollution assessment indicates that many agricultural soils are categorized as low to moderately contaminated sites, but specific locations may still pose significant ecological risks (Ahmadi et al., 2019). The interaction of heavy metals in soil can influence their uptake by crops, with some metals having synergistic or antagonistic effects on the absorption of others, such as Zn (Xiang et al., 2021).

The concentrations of five metals (Cr, Mg, Zn, Fe, and Cu) in soil samples collected from the agricultural region were determined. The study also revealed various sources of these metals in these locations by comparing the results obtained with certain standards, and suggested remedial ways of controlling the pollution caused by these metals in the study area. The findings of this research will contribute to the available data on the metal distribution in agricultural samples in the study area.



MATERIALS AND METHODS

Study Area

Cece is a village located on latitude of 90 5' 0'' North and longitude of 60 34' 00'' East in the Northern Lapai Local Government Area of Niger State, Nigeria.

SAMPLE COLLECTION

Soil Sample

Soil samples were collected in January 2022, and a total of five (5) samples were collected. Three agricultural locations within the Village were randomly selected for the investigation. Each soil sample was immediately placed inside a polyethylene bag and sealed tightly.

Table 1: Sampling points and locations with coordinates

Location	Horizon	Sample Id	Coordinates
A	Top soil	AT	N 09 ⁰ 05'04.1, E 0060 33'48.1
A	Low soil	AL	N 090 05'04.1, E 0060 33'48.1
B	Top soil	BT	N 09 ⁰ 05'01.0, E 0060 33'52.7
B	Low soil	BL	N 090 05'01.0, E 0060 33'52.7
R	Top soil	RS	N 09 ⁰ 05'06.8, E 0060 33'35.6

Preparation of the Sample

The collected soil samples were sun-dried for three days to remove moisture. The dried samples were crushed using a mortar and piston, sieved, and oven-dried for 1 hour. Two (2.0) gram of each sample was weighed and transferred to a digestion beaker. Three (3.0) cm³ of concentrated HNO₃ and one (1.0) cm³ of concentrated HCl were added. The mixture was heated to 100 °C until the evolution of gaseous fumes (from organic contents contained in the soil) turned brown, and heating was continued for two (2) min to dryness. Deionized water (50 cm³) was added to the digested soil and the resulting solution was filtered into a 100 cm³ volumetric flask. The filtrate was made up to marked with deionized water and transferred into plastic bottles for heavy metal analysis.



Research Instrument

An Agilent microwave plasma atomic emission spectrometer (MP-AES) with instrument number MY19479002 at the Center for Applied Science and Technology, Usmanu Danfodio University, Sokoto, Nigeria was used for the analysis of Cr, Mg, Zn, Fe, and Cu. The operating conditions were a pump speed of 70 rpm, sample intake of 15–60 s, rinse time of 8–20 s, and stabilization time of 5–15 s. The emission lines considered and correlation coefficients for the analytes are presented in Table 2.

Table 2: Emission wavelengths, nebuliser flow rate and correlation coefficients of analytes

Analyte	Wavelength (nm)	Nebulizer flow (L/min)	correlation coefficient
Cr	425.433	0.9	0.9998
Cu	324.754	0.7	0.99976
Zn	213.857	0.45	0.99793
Mg	283.213	0.9	0.9997
Fe	371.993	0.65	0.99923

Table 3: Results of heavy metal concentrations in soil samples (mg/Kg)

SAMPLES	Cr	Cu	Fe	Mg	Zn
AT	1.11±0.06	1.21±0.06	287.01±9.06	1.50±0.08	170.51±0.08
AL	0.51±0.03	13.01±0.68	292.03±10.93	67.03±2.51	162.12±7.04
BT	1.50±0.08	28.50±1.50	303.51±11.01	273.52±11.41	184.12±9.66
BL	0.50±0.03	1.12±0.06	287.51±12.03	73.04±2.38	164.09±8.51
RS	0.05±0.01	19.5±1.02	152.04±07.03	14.53±0.76	170.03±7.49

RESULTS AND DISCUSSION

Discussion

Chromium

In this study, the highest level of Cr (1.50 ± 0.08 mg/Kg) was found in the topsoil from area B (sample BT), whereas the lowest level (0.05 ± 0.01 mg/Kg) was found in river sediment



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(sample RS). The differences between these mean levels were not statistically significant. They also did not exceed the acceptable limit of ≤ 100 mg/Kg, as suggested by WHO and FEPA in Wanjala et al. (2020). This result is consistent with those reported in previous studies of 0.27 ± 0.03 mg/Kg reported by Wanjala et al. (2020). Cr does not have physiological functions, and it is usually toxic to plants even at very low soil concentrations, often causing diseases such as leaf rolling, limited seed numbers, and eventually death. Leafy crops such as spinach (50.83 ± 4.89 mg/Kg) and lettuce (47.45 ± 6.70 mg/Kg) showed higher potential of bioaccumulation and cultivation should be avoided in regions of higher levels of Cr (Xu et al., 2022).

Copper

This study showed that the lower soil from area B (sample BL) had the lowest Cu content (1.12 ± 0.06 mg/Kg), whereas topsoil from area B (sample BT) had the highest content (28.50 ± 1.50 mg/Kg). Differences in mean concentrations were statistically significant at $p \leq 0.05$. However, they were still within the permissible limit of ≤ 100 ppm, as set by the FAO/WHO in Ajaha et al. (2022) and the WHO in Wanjala et al. (2020). The findings of this study were similar to 3.40 mg/Kg found by Ajah et al. (2020). Bahiru & Teju (2019) found higher value of 146.10 mg/Kg while a lower value of 0.22 ± 0.04 mg/Kg was reported by Wanjala et al. (2020). Cu plays a role in respiration, photosynthesis, and hydrogen and carbon metabolism. Cu forms complexes with amino acids during protein synthesis, giving them a structural role in plants. Cu deficiency in soil results in a reduction in CO₂ fixation in plants. Excess Cu in soil is caused by the use of Cu-containing fungicides, industrial activities, and waste management. The toxicity of Cu leads to the formation of reactive oxygen species in plants grown in soils with high Cu levels.

Iron

In this study, the lowest concentration of Fe (152.04 ± 7.03 mg/Kg) was found in the river soil (sample RS), whereas the highest concentration (303.51 ± 11.01 mg/Kg) was found in the topsoil from area B (sample BT). These values were found to be below the permissible limit of ≤ 5000 mg/Kg, as recommended by the FAO/WHO in Bahiru and Teju (2019). They were also statistically similar, except for the river sediment, which differed significantly ($p \leq 0.05$). This may have been caused by the leaching of the soil by running stream and a difference in the geochemistry of the river sediment. This finding shows an indication of Fe contamination but not toxicity and is lower than values of 12,051 mg/Kg reported by Bahiru



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and Teju., (2019) but similar values of 31.55 to 187.47 mg/Kg in soil were found by Akthar et al. (2022). Fe is an essential nutrient in soil, vital for the optimal development of plants; nonetheless, an elevated level of Fe in the soil could potentially exert detrimental impacts on plant growth. Fe plays roles in photosynthesis and respiratory. However, remediation of excess iron in agricultural soils can be achieved by the use of NPK + lime fertilizers in appropriate combination (Akthar et al., 2022).

Magnesium

This study revealed that the highest concentration of Mg (273.52 ± 11.41 mg/Kg) was found in the topsoil from area B (sample BT), while the lowest concentration (1.50 ± 0.08 mg/Kg) was found in the topsoil from area A (sample AT). Other results reported such as by Wanjala et al., 2020 (11.87 ± 2.10 mg/Kg) were within this range. However, these values were below the standard permissible limit of ≤ 100 mg/Kg, as set by the WHO and FEPA (Wanjala et al., 2020; Ajah et al., 2022), except for sample BT. Mg at AT showed a low concentration. This is caused by magnesium uptake in major amounts for proper growth by oil crops such as groundnuts and sesame that are commonly cultivated in the study areas. The roots of these plants are only a few centimeters deep into the top horizon. At BT, the concentration was higher because of the heterogeneous nature of the soil caused by differences in soil geochemistry. Land fallow, to a small extent, would have caused Mg to recover over time.

Zinc

The highest level of zinc (170.51 ± 0.08 mg/Kg) was detected in the topsoil from area B (sample BT) while the lowest level (162.12 ± 7.04 mg/Kg) was in the lower soil from area A (sample AL). However, these differences were not statistically significant ($p \leq 0.05$). The levels of Zn found in all samples were below the permissible limit of ≤ 300 mg/Kg recommended by the FAO/WHO and FEPA (Ajah et al. 2022). However, these values exceed natural background concentration of 10-100 mg/Kg and suggest influence of factors such as use of fertilization, atmospheric deposition or soil geochemistry on the Zn levels in these locations (Mertenés & Smolders, 2012). These results exceed values of 2.61 ± 0.51 mg/Kg and 114.86 ± 10 mg/Kg reported by Wanjala et al. (2019) and Bahiru & Teju, (2019). Zn, required by plants at < 100 mg/kg dry weight, is an essential element for approximately 200 plant enzymes with structural and functional roles. These enzymes aid energy production and chlorophyll synthesis. Higher levels of Zn are caused by sources such as mining and the use of phosphate fertilizers. To avoid toxicity due to excess concentrations of Zn in soils which causes reddening of leaves crops, whereas soil deficiency reduces the



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yield of cereal seeds. These physiological changes in plants can be used as an early signs to monitor Zn levels in soil. Zn showed a higher concentration in the top layer of the soil because of the use of phosphate fertilizer, although the values at the lower horizon did not vary much. Excess Zn is flooded into the river during storms and is retained in the sediments.

CONCLUSION

The findings of this study revealed that trace concentrations of Cr, Cu, Fe, Mg, and Zn at varying levels were found in various soil samples (topsoil from areas A and B, lower soil from areas A and B, and river soil). These soils are safe for cultivating crops because the concentrations of heavy metals found in them are below international standards. Based on the findings of this study, it is recommended that the soils can be used for cultivation of crops, and continuous monitoring of the soils should be done within a period of time, since human activities can cause contamination of the soil. Further studies should be conducted on other toxic metals that have not been studied in this work, such as Ni, Pb, and Cd. Finally, farmers in the area should also avoid excessive use of chemicals, such as herbicides, fertilizers, and pesticides. This is because residues from most of these chemicals accumulate over a period of time and subsequently reach toxic levels. The sites could be considered as low polluted areas because the concentrations of the metals in the soil samples from all the regions fell below the permissible limits. The heavy metal concentrations obtained from all sites were below the maximum tolerable levels proposed for agricultural soils.

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