



**XRF/AAS ANALYSIS OF CU, FE, MN, AND ZN IN SEDIMENTS SAMPLES IN
KHARTOUM STATE, SUDAN**

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ABSTRACT

In the present study, sediment samples have been collected from three different locations: White Nile, Blue Nile and River Nile in Khartoum State, Sudan. The elemental concentrations of Cu, Fe, Mn, and Zn have been determined using X-ray Fluorescence spectrometry (XRF) and Atomic Absorption Spectroscopy (AAS). The accuracy of measurements has been investigated by using IAEA-Soil-7 for both techniques. A good agreement was found between certified and measured values. The elemental concentrations showed little difference amongst the different sediments samples. Generally, the obtained results show that the highest concentration values of elements were found in loc₂ compared to elemental concentration found in Loc₁ and Loc₃. For different seasons, the highest concentrations were observed in autumn while the lowest were found in winter time. A statistical test (*t*-test) was applied to the data of both methods without any significant difference between the two techniques. The results obtained were compared to WHO permissible limits. Correlations between different elements were performed. Hierarchical cluster analysis was done for the data. The average elemental concentrations were calculated and compared with data from the literature.

KEYWORDS: XRF, AAS, Elemental Concentrations, Sediments Samples.

INTRODUCTION

Heavy metals are considered serious pollutants of the aquatic ecosystem. Heavy metals can enter the soil by several pathways, and their behavior and fate in soil often depend on their source and species. The most important anthropogenic sources of heavy metals for soils include commercial fertilizers, liming materials, agrochemicals, sewage sludge's and other materials used as soil amendment, irrigation waters, and atmospheric deposition from industrial, urban, and road emissions. The greatly increased circulation of hazardous heavy metals in soil, sediment, water, and air has raised considerable concern for environmental protection and human health.^[1]

Sediment is a naturally occurring material that is broken by processes of weathering and erosion and subsequently transported by the action of wind, water, or ice, and/or by the force of gravity on the particle itself.^[2] As a result of complex physical, chemical and biological processes, a major fraction of trace metals is found to be associated with water body sediments. Sediments are multi-phase solids containing silicates, carbonates, hydroxides/oxides, sulfates, and organic substances as major components.^[3] Therefore, ensuring a good

sediment quality is crucial to aquatic life. It is important to evaluate metal contents in sediments because under certain conditions, sediments can act as a source of metals. Moreover, the amount of a given metal that can be released from contaminated sediment depends critically on the metal species.^[4]

Large interest has been devoted in the last decades to heavy metal research in sediment, which is unanimously considered as one of the most important recipients of trace elements originated from several human activities.^[5] Heavy metal concentrations in sediments are mainly dependent on the nature of the parent material from which they are derived, but anthropogenic influences can also alter heavy metal concentrations in terrestrial and fluvial environments. Consequently, the natural background concentration of heavy metals in sediments can be used as a reference value to estimate the contamination level of soils and sediments. Heavy metal toxicity can lower energy levels and damage the functioning of the brain, lungs, kidney, liver, blood composition, and other important organs. Long-term exposure can lead to gradually progressing physical, muscular, and neurological degenerative processes that imitate diseases.^[6]

Various analytical methods, which can quantitatively characterize trace levels of heavy metals in several types of samples such as AAS and XRF. In this study, XRF and AAS were used for heavy metals quantitative analysis in sediments samples.^[7] The main goal of this study is to determine the level of Cu, Fe, Mn, and Zn in sediments samples, during autumn and winter seasons using XRF and AAS techniques, compare the data obtained for the two seasons and compare our study values with data from the literature.

MATERIALS AND METHODS

Study area

The study area was located in Khartoum state, in three main places the White Nile, the Blue Nile, and River Nile. Figure 1 shows the sampling sites.

Table 1: Location code, locations, season and samples codes.

Code	Location	Season	Sample codes
W1	White Nile	autumn	W11-W110
B1	Blue Nile	autumn	B11-B110
R1	River Nile	autumn	R11-R110
W2	White Nile	winter	W21-W210
B2	Blue Nile	winter	B21-B210
R2	River Nile	winter	R21-R210



Figure 1: Shows the sampling sites.

Samples preparation

An air-dried grounded sieved of 60 μ m size sample was prepared. Samples were pulverized, homogenized and manually hydraulic pressed about 15 tons into a 4.9 cm² pellet weighed 1 g prior for XRF analysis.

0.5g of an air-dried, ground, and the sieved sample was placed in an Erlenmeyer flask. 20 ml of extracting solution (0.05N HCl + 0.025N H₂SO₄) were added. Then the solution was placed in a mechanical shaker for 15 minutes. The solution was filtered into a 50 ml volumetric flask and diluted to 50 ml with extracting solution. This solution was kept prior for AAS analysis.

XRF measurements

EDXRF provides a rapid and non-destructive method. All measurement was carried out using XRF Spectrometer with Cd¹⁰⁹ as a source for excitation. The characteristic X-rays emitted from the sample were

Samples collection

Sixteen sediment samples were collected during different season's autumn and winter (30 for each). Along the White Nile, the Blue Nile, and the River Nile. Table 1 shows the location code, locations, season and samples codes. Samples were collected by shovel, and then placed in a polyethylene sack; all samples have been dried crushed and sieved to powder then transferred to the laboratory and prepared for analysis.

detected using Si (Li) detector with Full Width at Half Maximum intensity (FWHM) equal 170 eV at 5.96 keV, each spectrum was collected for a lifetime of 3000s. Quantitative calculations were made by the QAES-Radioisotopes version 1995 Method. Experimental parameters were obtained by calibration, using standard reference material. The software AXIL is designed for full treatment dealing with spectra was recorded⁽⁸⁾. To evaluate the quality of the analytical results obtained, the IAEA Standard Reference Material (IAEA-soil-7) was analyzed. Table 2 shows the accuracy results using SRM (IAEA soil-7) for XRF.

Table 2: XRF accuracy results using standard reference material IAEA-Soil-7

Element	Certified value (ppm)	Measured value (ppm)	Relative error (%)
Cu	11	10.6	3.63
Fe	25700	25100	2.33
Mn	631	616	2.37
Zn	104	76	26.9

AAS measurements

Buck 210VGP atomic absorption spectrophotometer with air-acetylene burner, with a hollow cathode lamp, is designed to measure the elemental concentration of Cu,

Fe, Mn, and Zn in sediment samples. The accuracy of the analytical procedure used is confirmed by the analysis of IAEA-Soil-7. Measured values were very good in compliance with the certified values (Table 3).

Table 3: AAS Accuracy results using standard reference material IAEA-Soil-7.

Element	Certified value (ppm)	Measured value (ppm)	Relative error (%)
Cu	11	9.38	14.7
Fe	25700	25260	1.7
Mn	631	582.4	7.7
Zn	104	112.25	7.9

RESULTS AND DISCUSSION**Elemental concentration in sediments samples**

The elemental concentrations of Cu, Fe, Mn, and Zn were investigated using XRF and AAS. The summary of statistical data obtained for sediment samples was listed in Tables (4-7) and graphically displayed in Fig 3 & Fig 4. The comparison between XRF and AAS results is illustrated in Figures (5 & 6) different seasons.

In the autumn season, samples from loc₁, the results obtained by XRF were showed that the concentration of Cu ranges from 19.5 ppm to 56.1 ppm with an average value of 46.7 ppm and a standard deviation value of 18.23 ppm. Iron concentration ranges from 27800 ppm to 91700 ppm with an average value of 58582 ppm and a standard deviation value of 2858.2 ppm. Manganese, concentration ranges from 754 ppm to 4350 ppm with an average value of 2096 ppm and a standard deviation value of 1950.19 ppm. The concentration of Zinc ranges from 60 ppm to 195 ppm with an average value of 114.6 ppm and a standard deviation value of 51.85 ppm. While the results obtained by AAS for the same sample showed that the concentration of Cu ranges from 3.5 ppm to 77.8 ppm with an average value of 16.2 ppm and a standard deviation value of 22 ppm. Iron concentration ranges from 27.55 ppm to 256.8 ppm with an average value of 133.98 ppm and a standard deviation value of 1.25 ppm. Manganese, concentration ranges from 20.07 ppm to 130.07 ppm with an average value of 79.88 ppm and a standard deviation value of 38.98 ppm. The concentration of Zinc ranges from 5.1 ppm to 46.4 ppm with an average value of 21.57 ppm and a standard deviation value of 13.58 ppm.

In loc₂ autumn samples, XRF results showed that the concentration of Cu ranges from 11.8 ppm to 97.5 ppm with an average value of 55.72 ppm and a standard deviation value of 37.51 ppm. Iron concentration ranges from 55800 ppm to 246000 ppm with an average value of 167960 ppm and a standard deviation value of 89983.8 ppm. Manganese, concentration ranges from

1690 ppm to 3990 ppm with an average value of 2722.5 ppm and a standard deviation value of 952.55 ppm. The concentration of Zinc ranges from 63.6 ppm to 656 ppm with an average value of 253.92 ppm and a standard deviation value of 185.58 ppm. While the results obtained by AAS for the same sample showed that the concentration of Cu ranges from 4.5 ppm to 16.2 ppm with an average value of 10.36 ppm and a standard deviation value of 3.55 ppm. Iron concentration ranges from 39.7 ppm to 264 ppm with an average value of 123.1 ppm and a standard deviation value of 72.43 ppm. Manganese, concentration ranges from 57.91 ppm to 301.65 ppm with an average value of 131.49 ppm and a standard deviation value of 70.75 ppm. The concentration of Zinc ranges from 0.05 ppm to 3.12 ppm with an average value of 0.14 ppm and a standard deviation value of 0.08 ppm.

In autumn season samples from loc₃, XRF results showed that the concentration of Cu ranges from 3.2 ppm to 55.1 ppm with an average value of 26.02 ppm and a standard deviation value of 21.69 ppm. Iron concentration ranges from 1.47 ppm to 6.66 ppm with an average value of 3.58 ppm and a standard deviation value of 2.72 ppm. Manganese, concentration ranges from 0.21 ppm to 1.89 ppm with an average value of 1.27 ppm and a standard deviation value of 0.69 ppm. The concentration of Zinc ranges from 0.12 ppm to 0.96 ppm with an average value of 0.52 ppm and a standard deviation value of 0.38 ppm. While the results obtained by AAS for the same sample showed that the concentration of Cu ranges from 1.55 ppm to 14.6 ppm with an average value of 8.48 ppm and a standard deviation value of 3.49 ppm. Iron concentration ranges from 13.85 ppm to 170.7 ppm with an average value of 80.01 ppm and a standard deviation value of 61.91 ppm. Manganese, concentration ranges from 43.3 ppm to 206.6 ppm with an average value of 106.38 ppm and a standard deviation value of 50.38 ppm. The concentration of Zinc ranges from 1.4 ppm to 8.6 ppm

with an average value of 4.74 ppm and a standard deviation value of 2.27 ppm.

In the winter season, samples from loc₁, the results obtained by XRF were showed that the concentration of Cu ranges from 1.44 ppm to 6.15 ppm with an average value of 3.9 ppm and a standard deviation value of 1.77 ppm. Iron concentration ranges from 5130 ppm to 13800 ppm with an average value of 9356 ppm and a standard deviation value of 3124 ppm. Manganese, concentration ranges from 135 ppm to 246 ppm with an average value of 170.2 and a standard deviation value of 46.3 ppm. The concentration of Zinc ranges from 9.6 ppm to 23.9 ppm with an average value of 16.06 ppm and a standard deviation value of 6.03 ppm. While the results obtained by AAS for the same sample showed that the concentration of Cu ranges from 6 ppm to 192 ppm with an average value of 42.8 ppm and a standard deviation value of 50.09 ppm. Iron concentration ranges from 1289 ppm to 1934 ppm with an average value of 1636.2 ppm and a standard deviation value of 226.59 ppm. Manganese, concentration ranges from 53 ppm to 302.5 ppm with an average value of 208.55 ppm and a standard deviation value of 96.46 ppm. The concentration of Zinc ranges from 29.5 ppm to 203 ppm with an average value of 79.45 ppm and a standard deviation value of 46.08 ppm.

In loc₂ winter samples, XRF results showed that the concentration of Cu ranges from 2.84 ppm to 62.7 ppm with an average value of 27.82 ppm and a standard deviation value of 30.28 ppm. Iron concentration ranges from 11900 ppm to 153000 ppm with an average value of 61720 ppm and a standard deviation value of 63412 ppm. Manganese, concentration ranges from 239 ppm to 2650 ppm with an average value of 1234 ppm and a standard deviation value of 1261.9 ppm. The concentration of Zinc ranges from 3.67 ppm to 261 ppm with an average value of 64.69 ppm and a standard deviation value of 10.9.9 ppm. While the results obtained by AAS for the same sample showed that the concentration of Cu ranges from 26.5 ppm to 95 ppm with an average value of 57.05 ppm and a standard deviation value of 24.29 ppm. Iron concentration ranges from 1342 ppm to 1653 ppm with an average value of 1516.4 ppm and a standard deviation value of 97.16 ppm. Manganese, concentration ranges from 93.5 ppm to 292 ppm with an average value of 201.8 ppm and a standard deviation value of 63.81 ppm. The concentration of Zinc ranges from 28.5 ppm to 88.5 ppm with an average value of 64.75 ppm and a standard deviation value of 19.69 ppm.

In winter season samples from loc₃, XRF results showed that the concentration of Cu ranges from 3.94 ppm to 65.7 ppm with an average value of 17.52 ppm and a standard deviation value of 29.96 ppm. Iron concentration ranges from 24200 ppm to 225000 ppm with an average value of 71660 ppm and a standard

deviation value of 58364 ppm. Manganese, concentration ranges from 364 ppm to 2890 ppm with an average value of 1002.6 ppm and a standard deviation value of 1060.8 ppm. The concentration of Zinc ranges from 18.9 ppm to 44.9 ppm with an average value of 34.56 ppm and a standard deviation value of 11.81 ppm. While the results obtained by AAS for the same sample showed that the concentration of Cu ranges from 22.5 ppm to 176 ppm with an average value of 83.56 ppm and a standard deviation value of 57.34 ppm. Iron concentration ranges from 1519 ppm to 1629 ppm with an average value of 1584.7 ppm and a standard deviation value of 33.21 ppm. Manganese, concentration ranges from 172.5 ppm to 325.5 ppm with an average value of 266.1 ppm and a standard deviation value of 54.65 ppm. The concentration of Zinc ranges from 5.55 ppm to 103.5 ppm with an average value of 70.95 ppm and a standard deviation value of 17.78 ppm.

A statistical test (*t*-test) was applied to the data of both methods in different seasons without any significant difference between the two techniques. From results obtained that the mean elemental concentration of the analyzed elements was found to be similar or close to in different seasons from different locations except for Fe which was found higher in winter season than autumn season may be due to the different agricultural cycle, types of crops grown in different locations.

XRF is a multi-element technique, which is theoretically capable of determining an ever-greater range of elements than AAS. AAS is a more sensitive method compared to XRF. XRF is a non-destructive method compared to AAS. Consequently, the XRF spectrometer employed is a promising tool that may be used for quick screening analysis of environmental samples without the need for time-consuming sample preparation procedures. Whereas in the case of AAS, sample pretreatment methods depend on the type of soil and the chemical method itself.

WHO permissible limits for elements for soil

The results obtained were compared to WHO permissible limits for elements for sediment showed that the values indicate the desirable maximum levels of elements in unpolluted sediment.^[8]

Table 4: Summary of statistical data obtained for sediment samples by XRF in Autumn in ppm

Elements		Mean \pm SD	Min	Max
Cu	W1	46.7 \pm 18.23	19.5	56.1
	B1	55.72 \pm 37.51	11.8	97.5
	R1	26.02 \pm 21.69	3.2	55.1
Fe	W1	58582 \pm 28582	27800	91700
	B1	167960 \pm 89983.8	55800	246000
	R1	92100 \pm 62062.3	16500	165000
Mn	W1	2096 \pm 1950.19	754	4350
	B1	2722.5 \pm 952.55	1690	3990
	R1	1535 \pm 410.2	1050	1890
Zn	W1	114.6 \pm 51.85	60	195
	B1	253.92 \pm 185.58	63.6	656
	R1	52.16 \pm 37.54	12.7	96.1

Table 5: Summary of statistical data obtained for sediment samples by XRF in winter in ppm.

Elements		Mean \pm SD	Min	Max
Cu	W2	3.9 \pm 1.77	1.44	6.15
	B2	27.82 \pm 30.28	2.84	62.7
	R2	17.52 \pm 26.96	3.94	65.7
Fe	W2	9356 \pm 3124	5130	13800
	B2	61720 \pm 63492	11900	153000
	R2	71660 \pm 58364	24200	225000
Mn	W2	170.2 \pm 46.3	135	246
	B2	1234 \pm 1261.9	239	2650
	R2	1002.6 \pm 1060.8	364	2890
Zn	W2	16.06 \pm 6.03	9.6	23.9
	B2	64.69 \pm 109.9	3.67	261
	R2	34.56 \pm 11.81	18.9	44.9

Table 6: Summary of statistical data obtained for sediment samples by AAS in Autumn in ppm.

Elements		Mean \pm SD	Min	Max
Cu	W1	16.2 \pm 22	3.5	77.8
	B1	10.36 \pm 3.55	4.5	16.2
	R1	8.64 \pm 3.49	1.55	14.6
Fe	W1	133.98 \pm 1.25	27.55	256.8
	B1	123.01 \pm 72.43	39.7	264
	R1	80.01 \pm 61.19	13.85	170.7
Mn	W1	79.88 \pm 38.98	20.7	130.7
	B1	131.49 \pm 70.75	57.95	301.65
	R1	106.38 \pm 50.38	43.3	206.6
Zn	W1	21.75 \pm 13.58	5.1	46.4
	B1	0.139 \pm 0.08	0.051	0.312
	R1	4.74 \pm 2.27	1.4	8.6

Table 7: Summary of statistical data obtained for sediment samples by AAS in winter in ppm.

Elements		Mean \pm SD	Min	Max
Cu	W2	42.8 \pm 50.09	6	192
	B2	57.05 \pm 24.29	26.5	95
	R2	83.56 \pm 57.34	22.5	176
Fe	W2	1636.2 \pm 226.59	1289	1934
	B2	1516.4 \pm 97.16	1342	1653
	R2	1584.7 \pm 33.21	1519	1629
Mn	W2	208.55 \pm 96.46	53	302.5
	B2	201.8 \pm 63.81	93.5	292
	R2	266.1 \pm 54.65	172.5	325.5
Zn	W2	79.45 \pm 46.08	29.5	203
	B2	64.75 \pm 19.69	28.5	88.5
	R2	70.95 \pm 17.78	5.55	103.5

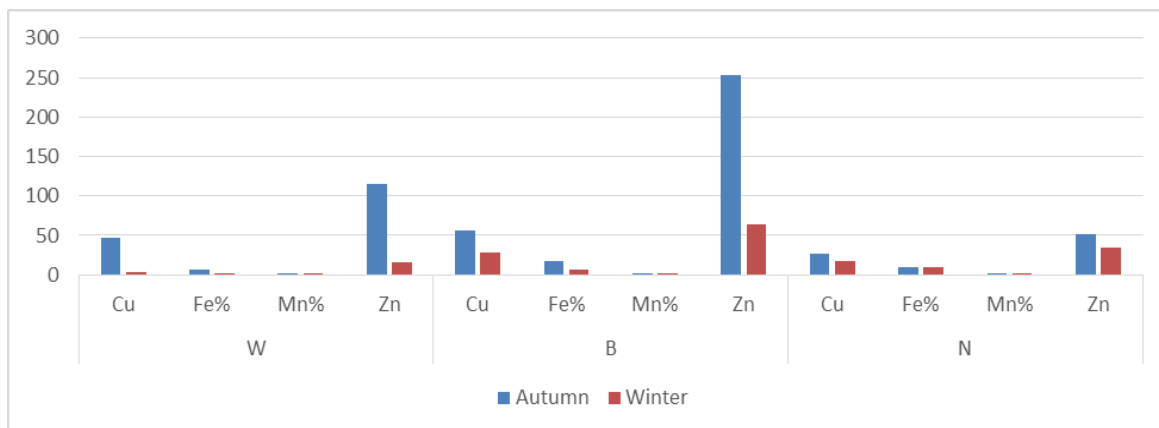


Fig. 2: Mean elemental concentrations (ppm) of investigated elements in different seasons by XRF.

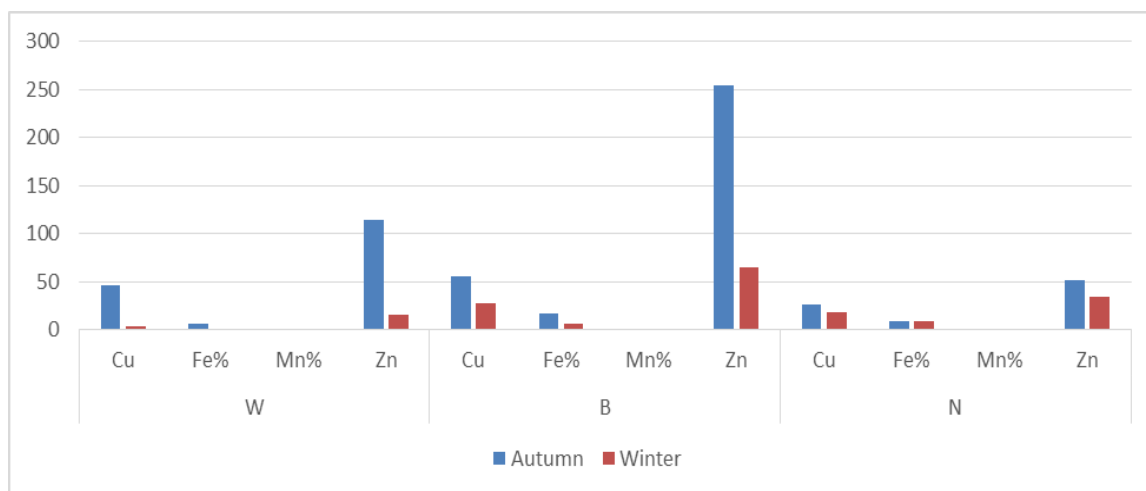


Fig. 3: Mean elemental concentrations (ppm) of investigated elements in different season by AAS.

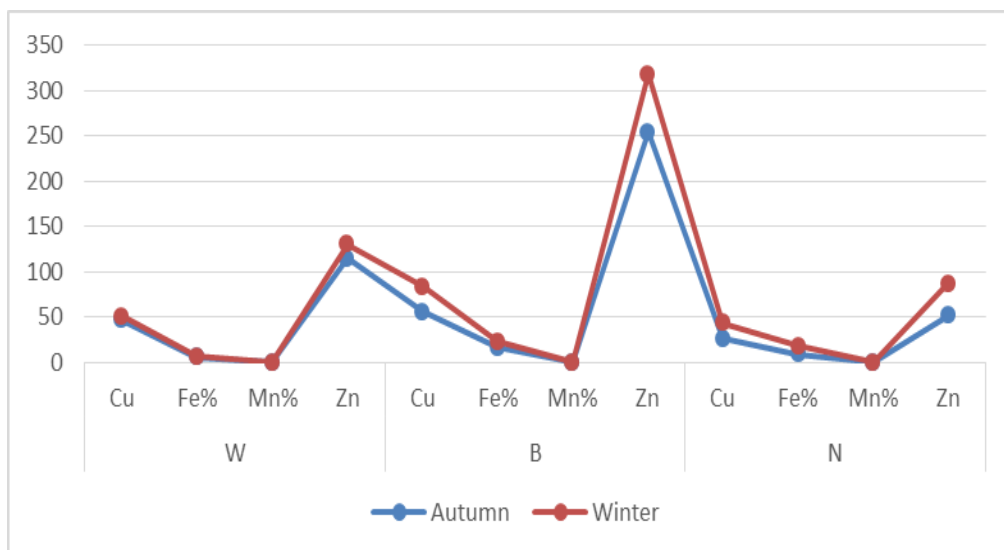


Fig. 5: Comparison of mean elemental concentration (ppm) in the different season by XRF.

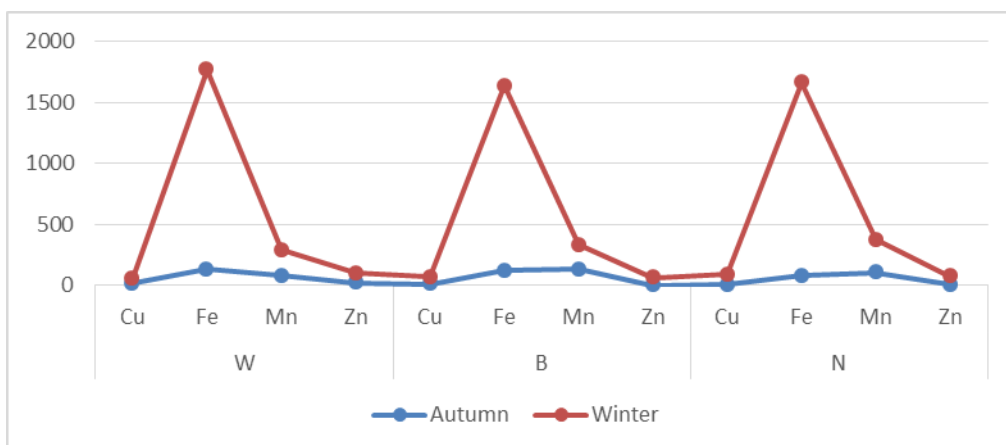


Fig. 6: Comparison of mean elemental concentration (ppm) in the different season by AAS.

Correlation

Correlations between concentrations certain elements can be used as indicators of specific sources.^[9] A pair-wise correlation was performed. Table 8 shows the correlation between the elemental concentration data for sediment samples collected from different locations. The correlation data (bold correlation is significant at $p < 0.05$) results showed that are highly correlated with each other.

Cluster analysis

The results of cluster analysis of elements in sediment samples were obtained as dendrograms displaying two main clusters. In the dendrogram the first cluster containing the Cu, Mn and Zn. The second cluster includes containing only Fe. Figure 7 shows the cluster analysis.

Comparison of elemental concentration between this study and data from the literature

A comparison of the average elemental concentration of sediment samples with previous data from literature was listed in Table 7. The average concentration of Cu was higher in Kosovo, Algeria, Turkey, and this study than the values in India and Egypt. Fe concentrations in Turkey, Nigeria, and Bengal are less than the concentration in Egypt and Algeria, while the concentration of Fe is very high in Kosovo and this study. The concentration of Mn in Kosovo, Turkey, and this study is higher compared to Bengal, Nigeria, and Algeria. The average concentrations of Zn were higher in Nigeria, Egypt, Kosovo, Algeria, and this study than the values in Burkina Faso. Lead concentrations in India and Ghana are less than the concentration in Ethiopia, Egypt, Burkina Faso, and this study.

Table 6: Correlation between the analyzed elements.

	Cu	Fe	Mn	Zn
Cu	1	-0.045	0.247	0.326
Fe		1	0.641	0.482
Mn			1	0.541
Zn				1

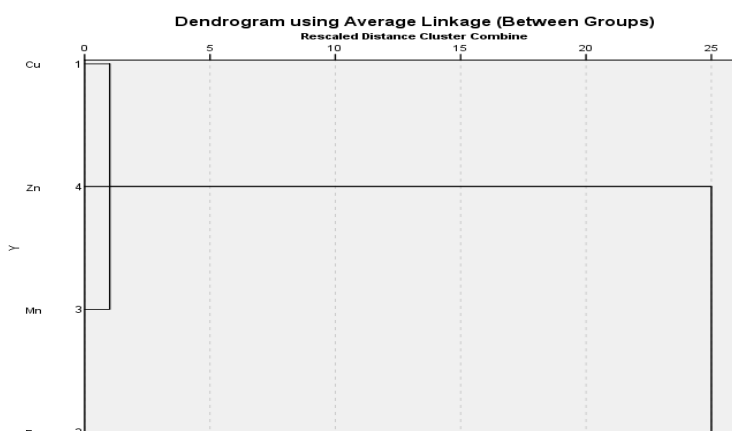


Figure 7: Cluster analysis

Table 7: Comparison of elemental concentration with data from the literature.

	Cu	Fe	Mn	Zn
This work	31	29285	613.2	56.5
Bengal ^[9]	-	12.14	1.1	2.28
Nigeria ^[10]	-	19.31	6.13	40.25
Kosovo ^[11]	61.2	19084	660	122
Algeria ^[12]	25	2400.8	3.02	113
Egypt ^[13]	5.9	2586	-	24
Turkey ^[14]	19.35	28.53	421.8	29.10
India ^[15]	10.04	-	-	12.71

Conclusions and Recommendations

Elemental concentrations of Cu, Fe, Mn, and Zn were analyzed in sediment samples using XRF and AAS techniques. The results obtained show no significant difference between both techniques and results obtained for the different seasons. The elemental concentration of Fe and Mn in sediment samples was found higher in the winter season than their concentration in the autumn season. While the concentration of Cu and Zn in both seasons was found to be close.

The elemental concentrations were found to be within the WHO permissible limits for analyzed elements in sediment samples. XRF spectrometer employed is a promising tool that may be used for quick screening analysis of environmental samples without the need for time-consuming sample preparation procedures. Whereas in the case of AAS, sample pretreatment methods depend on the type of sediment and the chemical method itself.

Recommendations

Monitoring of trace elements, heavy metals and related indicators periodically in rivers sediments. New studies are needed to cover new areas and expanding the sample numbers and increase the sampling depth.

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