

## Studies on Water-Harvesting Ponds (Haffirs) in Gedarif State, Eastern Sudan: II. Determination of Haffir Water Heavy Metals using ICP-OES vs. Atomic Absorption Spectrometry

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### Abstract

Some areas and states of the Sudan suffer from scarcity of water from November to June, despite the high rainfall rates during the Autumn/kharif season (July-October). Therefore, people resort to water-harvesting ponds (haffirs) for drinking, agriculture and the domestic needs and activities. Haffirs store water during the rainy-season to be used from November up to June depending on their sizes and consumption rate. The objectives of the present study, as one of a series of studies on water in general, and haffirs water specifically, was to investigate the possibility of water contamination by heavy metals (HMs), using 2 methods, viz. Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES 725 E) and atomic absorption spectrometry (AAs). Water was collected from 3 haffirs, representing 3 regions within the Gedarif State (GS), and subjected to analysis. Water samples were taken every 2 wk from July to February. Samples were prepared as required for each method, and the concentrations of the HMs were determined. Precise and accurate measurements are paramount in the field of toxicology and nutrition. Both techniques have advantages, however, the ICP-OES is increasingly favored by scientists around the world. The HMs of concern were: Pb, Cu, Zn, Fe, Cd, Mn, Ni, Mo, Ti, V, Sr, and Cr. The results of both techniques showed that levels of several HMs were higher than the WHO permissible levels (PLs). However, the results of the ICP-OES were by far higher than those of AAS. Toxicity is dose-dependent and depends also on frequency of exposure. The ICP-OES method seems to be more accurate than that of AAS in determining the HMs levels in water and must be the recommended method for decision-making.

**Keywords:** Water-Harvesting Ponds; Haffirs; Heavy Metals; Water Pollution; Contaminants; ICP-OES; Atomic Absorption; Gedarif; Sudan

### Introduction

Gedarif State (GS, eastern Sudan), and Western Sudan States (the greater Kordofan and Dar Fur states) store rainwater in Haffir, i.e. an excavated area of land where rain-water and run-off water is harvested during the rainy-season (July - October). This harvested water is intended to be stored for human and animal consumption, in addition to other activities during the dry-season (November - June). The size of haffirs varies, depending on the location, hydrology, soils, rainfall; it ranges between 5,000 and 30,000m<sup>3</sup>. Haffirs are divided into traditional and standard. Traditional haffirs go back to >100 yr. Standard haffirs are developed using modern engineering [1-4]. The environmental pollution by heavy metals (HMs) from anthropogenic sources, e.g. smelters, mining, power stations and the application of

pesticides containing metals, fertilizers and sewage sludge, in addition to the irresponsible disposal of wastes by various industries. They can become mobile in soils, depending on soils pH and their speciation. So, a fraction of the total mass can leach to aquifer or can become available to living organisms [11]. HMs have a crucial role in the metabolic processes. However, due to the excessive use and dumping at high concentrations, they become toxic to both human and others [10]. Moreover, people around the haffirs used to wash the tractors, lorries and the likes near or inside the haffirs.

HMs are defined as elements in the periodic table having atomic number  $> 20$  or densities  $> 6 \text{ g/cm}^3$ . HMs are also referred to as trace elements and are the metallic elements of the periodic table [6-8]. The most common toxic HMs include As, Pb, Hg, Cd, Cr, Cu, Ni, Ag, and Zn. The release of high amounts of HMs into water bodies creates serious health and environmental problems. HMs also occurs in small amounts naturally and may enter into aquatic system through leaching of rocks, airborne dust, forest fires and vegetation [6,7,12-14]. Increased urbanization and industrialization have increased the levels of trace metals, especially HMs in water ways. There are  $> 50$  elements that can be classified as HMs, but only 17 that are considered to be both very toxic and relatively accessible, Pb, Cu, Zn, Ni and Cr; however, should be given particular attention in terms of water pollution and discharge effects [16,17].

Toxicity levels depend on the type of metals, its biological role, and the type of organisms that are exposed to it [2-4,34,35]. Once in the aquatic environment, HMs are partitioned among various aquatic environmental compartments (water, suspended solids, sediments and biota). The metals in the aquatic environment may occur in dissolved particulate and complex form. The majority of metal contaminants partition onto particulate matter, e.g. clay minerals, Fe and Mn oxides/hydroxides, carbonates, organic substances (e.g. humic acids) and biological materials, e.g. algae and bacteria [6].

AAS offers reliable quantification of a select range of elements, usually limited to one or a few at a time. This limitation hinders comprehensive multi-elemental analysis and necessitates separate measurements for each element of interest. In contrast, ICP-OES allows simultaneous analysis of multiple elements, thus, providing a more efficient workflow and saving valuable time and resources. Interference and matrix effects are common challenges faced in elemental analysis. AAS is particularly susceptible to these issues, due to its narrow WL range and sensitivity to background radiation. Interference from matrix components can cause erroneous results and require laborious sample preparation methods to minimize or eliminate such interferences. ICP-OES, with its broader WL range and better tolerance for complex matrices, offers superior resistance to interferences, leading to more accurate results. AAS is known for its sensitivity, especially in flame-based systems. However, this sensitivity can vary significantly depending on the sample matrix, the instrument configuration, and the element being analyzed. The detection limits of AAS can be relatively high, limiting its usefulness for trace analysis. ICP-OES provides greater sensitivity across a wider- range of elements, with significantly lower detection limits. This advantage enables us to detect and quantify elements even at ultra trace levels. An ICP-OES instrument is more expensive to buy, but measures samples much quicker than AAS. Whereas AA measures the amount of light of a certain WL absorbed as certain elemental atoms enter an excited state, ICP-OES is the measurement of the light emitted by the elements in a sample introduced into an ICP source. AAS measures sequentially, while ICP measures simultaneously. For each element analyzed in an AAS, the instrument runs the test separately and each hollow cathode lamp emits its light and the absorption is measured for a single element in each run [1,2,5,18].

### **Objective of the Study**

The objective of the current research was to compare the results/sensitivity of each of the 2 methods in the detection of HMs in water; since the toxicities of these metals are dose-dependent, and  $>10$  Sudanese states depend on haffirs water for at least 8 months/yr.

## **Materials and Methods**

### **Study area**

This study was conducted in GS, eastern Sudan, which lies entirely between latitude 14-16 N and 33-36 E longitude. GS plays a significant role in the economy and agricultural products of the country. The population of the state is estimated as 1,827,181 consisting of most of the tribes of the country. The majority (65.9%) of the population works in rain-fed agriculture. The vegetation of the state is poor savannah. The soil is clay and muddy. There are several rivers that pass through the state (*viz.* Atbara, Elrahad, Sitait and Basalam). Average of the rainfall is approximately 612 mm. Several mountains and forests are scattered throughout the state. But in rural and agricultural areas, people depend on large water reservoirs (Haffirs) [1-3].

### **Samples collection**

Water samples (72) were collected from Elazaza, Tarfa and Kafay haffirs in plastic bottles, completely randomized design with 3 replicates CRD. After filtration, 10% nitric acid (2 ml conc. HNO<sub>3</sub>) was added. Samples were collected 3 times, 8 samples/haffir/month, i.e. a total of 24 sample/haffir and were stored at 1 - 4°C [1-,3,16,17].

### **Sample preparation**

The pH of the water sample was adjusted to pH 3.6 using 10M HCl. From the preserved samples, 100 ml was transferred to a conical flask and to it 5 ml of 65% conc. nitric acid and some boiling chips were added. The sample was evaporated using hot- plate to about 10 to 20 ml or before the appearance of a precipitate. Heating continued with the addition conc. HNO<sub>3</sub> until digestion is completed as indicated by light color and clear solution. Clean up was performed by washing the beaker wall with distilled water (DW), followed by filtration. The filtrate was transferred to 100m volumetric flask with 5 ml portions of water for rinsing, cooled and diluted to the mark. Then analyzed by (ICP-OE) [1-3].

### **Preparation of chemicals**

NaOH (ACS), 40g, was dissolved in DW and dilute to 100 ml. Diethyldithiocarbamic acid-sodium salt (20g) was dissolved in 380 ml of deionized water (DIW) and filtered through a 0.45-μ Millipore® filter. The filtrate was extracted 2x with 15-ml portion. Exactly 102g of potassium biphthalate was dissolved in 500 ml of DIW; 14 ml of 1M HCl was added and diluted to 1L with DIW. NaOH (1M). was prepared by dissolving 4g of NaOH in 100 ml of DIW [1-3].

### **Treatment to assist preservation of HMs**

Exactly 10% nitric acid was added to the water sample after filtration, then the samples were kept at 4°C [1-3].

### **Nitric acid digestion method (APHA3030E) by ICP-OES**

#### **Water samples preparation**

Acid water (100 ml) was poured in a conical flask and 5 ml of conc. Nitric acid (65%) was added to it, followed by adding boiling chips to the sample. The conical flask, which contains the sample was boiled in a hotplate (350°C) to evaporate up to 10 to 20 ml, where the concentration and precipitation occur. Heating was continued and conc. HNO<sub>3</sub> was added until digestion was completed as shown by light color and clear solution (APHA3030E2005) [1-3].

#### **Cleanup**

The conical flask was washed with DW and the solution was filtered by filter paper ashless (MN640M.125mm). The filtrate was transferred to 100 ml volumetric flask; 5 ml portions of DIW were drooped in the filter paper, then the sample was cooled and diluted till reaching 100 ml. The sample is now ready for analysis by ICP-OE (APHA3030E) [1-3].

### Water sample preparation and cleanup for analysis by AAS

One hundred ml of water sample was collected and filtered through a 0.4 $\mu$  Millipore filter (filter was washed with 100 ml of DIW immediately prior to filtration). Then added 16 drops of conc. HCl. Exactly 100 ml of the acidified water sample was transferred into 250 ml conical flask and to it added 2 ml of the phthalate buffer and adjusted the pH to 3.6  $\pm$  0.1. The next step is adding 7 ml of the diethyldithiocarbamate solution, transferred to a 500-ml separatory funnel, followed by adding 15 ml methyl isobutyl ketone (MIBK), shaken vigorously for 30 sec, and phases were allowed to separated. Layers were drawn into a glass stoppered test tube so as to be analyzed by AAS [1-3].

### Chemical analysis

The prepared water samples were placed in the glass -stoppered test tube, which takes the samples into the ICP-OES (model 725E), Khartoum Central Petroleum Laboratories, that reads the spectra automatically and shows the amount/concentration of Pb, Cu, Zn, Fe, Mn, Mo Ni, Cr Co, Ti, Sr and V in the screen. The AAS instrument model 210VGP of the same laboratories was used. All operating conditions were preloaded in the internal computer, including lamp settings, secondary WLs [1-5].

### Analysis of the data

Data obtained were subjected to analysis of variance (ANOVA) and all values from chemical analyses were presented as mean  $\pm$  SD and coefficient of variation (C.V. %).

### Results and Discussion

Table 1-3, in addition to figure 1 and 2 are examples of the data collected from the series of the studies conducted in the study are taken to fulfil the objective of this paper, i.e. comparing the results obtained by the 2 methods. The rest of the data was presented previously and the still more data will be published soon. Table 1 shows the results of 10 HMs using ICP-OES, whereas table 2 shows the levels of 9 HMs using AAS method. Table 3 shows the difference in sensitivity of the 2 methods when relating the ICP method to that of AAS in terms of percent. However, both methods reflected that most of the measured metals were higher than the WHO PLs (Table 1 and 2), but the concentration according to ICP-OES were by far higher than those indicated by AAS. It is obvious that these values are not attributed only to the instrument, but also to the sample's preparation methods for each technique.

HM*	Azaza (Mean $\pm$ SE)	C.V. (%)	Trafa (Mean $\pm$ SE)	C.V. (%)	Elkafey (Mean $\pm$ SE)	C.V. (%)	WHO (2014)
Pb	1.080 $\pm$ 0.21	41.9	2.150 $\pm$ 0.21	41.9	0.270 $\pm$ 0.21	41.9	0.0100
Cu	1.291 $\pm$ .11	18.6	1.492 $\pm$ 0.107	18.6	0.462 $\pm$ 0.11	18.6	2.0000
Zn	0.148 $\pm$ 0.01	13.7	0.037 $\pm$ 0.012	13.7	0.0207. $\pm$ 0.01	12.27	0.2000
Fe	3.170 $\pm$ 0.03	2.4	9.160 $\pm$ 0.37	2.4	2.5000 $\pm$ 0.04	2.40	0.5000
Co	0.0018 $\pm$ 0	0.0005	0.001 8 $\pm$ 0	0.0005	0.0018 $\pm$ 0.00	0.0005	0.0003
Cd	0.0009 $\pm$ 0	0.0004	0.00089 $\pm$ 0	0.0004	0.00089 $\pm$ 0.0	0.0004	0.003
Mn <sup>2+</sup>	2.190 $\pm$ 0.19	31.8	1.510 $\pm$ 0.19	31.8	1.1300 $\pm$ 0.19	31.8	0.4000
Ni	0.049 $\pm$ 0.04	45	0.085 $\pm$ 0.044	45	0.076 $\pm$ 0.04	45	0.0700
Mo	0.054 $\pm$ 0.00095	9.1	0.064 $\pm$ 0.00095	9.1	0.0510 $\pm$ 0.0009	9.1	0.0700
Cr	0.0013 $\pm$ 0.0001	6.5	0.0019 $\pm$ 0.0001	6.5	0.0031 $\pm$ 0.0001	6.5	0.0500

**Tab1e 1:** Means of concentration (Mean  $\pm$  SE) using ICP-OE methods.

\*HM = Heavey Metals.

HM	Azaza	C.V.	Tarfa	C.V.	Elkafay	C.V.	WHO
		%		%		%	2004
Pb	0.591 ± 0.14	0.98	0.845 ± 0.08	8.50	0.235 ± 0.001	1.30	0.01
Cu	1.141 ± 0.01	0.11	1.001 ± 0.072	0.52	0.222 ± 0.06	1.86	2
Zn	0.121 ± 0.006	7.9	0.020 ± 0.013	18.6	0.011 ± 0.005	1.23	0.2
Fe	2.45 ± 0.05	0.16	9.09 ± 0.06	2.19	2.09 ± 0.021	0.75	0.5
Co	0.0012 ± 0.0001	29.4	0.0022 ± 0.0001	29.40	0.000012 ± 0.001	29.40	0.0003
Cd	0.0009 ± 0.00001	25.3	0.0009 ± 0.00001	25.30	0.0009 ± 0.00001	25.30	0.003
Mn <sup>2+</sup>	1.003 ± 0.094	6.07	0.41 ± 0.0048	1.31	0.810 ± 0.001	0.11	0.4
Cr	0.001 ± 0.00004	3.2	0.0012 ± 0.0003	23.24	0.001 ± 0.00004	33.80	0.03
Ni	0.043 ± 0.02	9.5	0.049 ± 0.007	23.10	0.07 ± 0.0061	3.19	0.07

Table 2: Means (± SE) of concentrations als AAS methods.

HM	Azaza (%)	Tarfa (%)	Kafay (%)	*Mean (%)
Pb	54.72	39.30	8.70	34.24
Cu	88.38	67.09	48.05	67.84
Zn	81.75	5.71	53.14	46.86
Fe	77.28	99.23	83.60	86.70
Co	66.66	220.00	66.66	117.77
Cd	ND	ND	ND	ND
Mn <sup>2+</sup>	45.79	27.15	71.68	48.20
Cr	76.92	63.15	33.33	57.80
Ni	87.75	57.64	ND	48.46

Table 3: Sensitivity (%) of ICP detected water concentrations compared to AAS detected concentrations for 9 HMs.

ND = Not Different.

\*ICP Conc/AAS Conc. X100.

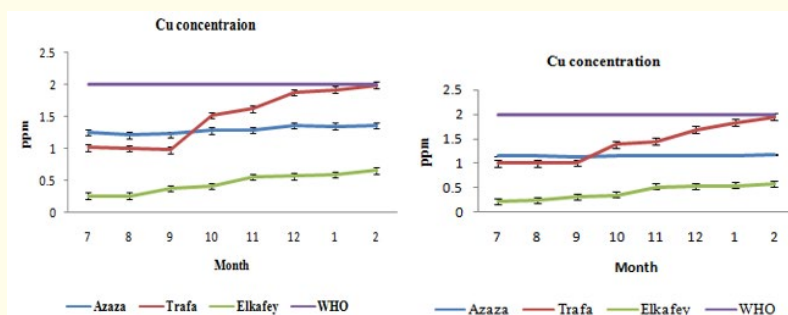


Figure 1: Concentration of Cu in (ppm) ICP-OE (left) and AAS methods (Right) as an example.

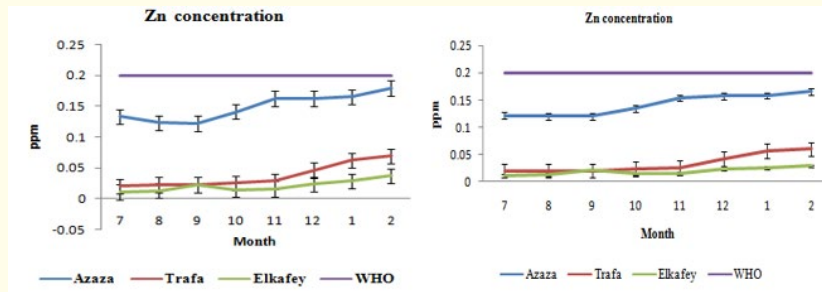


Figure 2: Concentration of Zn in (ppm) ICP-OE (left) and AAS methods (Right) as an example.

For example, regarding Pb levels, using ICP-OES range was 0.27 - 2.15 ppm, whereas the AAS results for the same element range was 0.235 - 0.845 ppm, this indicates that ICP-OES showed higher levels/sensitivity (34.24 to 54.72%). Another example is Cu; the difference was ranging from 48.05 to 88.38% between the 2 methods, with a mean of 68.74% in favor of ICP-OES. For the other HMS the following means were detected: Zn (46.86%), Fe (86.7%), Co (11.77%), Mn<sup>2+</sup> (48.2%), Cr (57.8%) and Ni (48.86%). The only HM that resulted in identical levels was Cd.

As mentioned earlier, AAS offers reliable quantification of a selected range of elements, usually limited to one or a few at a time. This limitation hinders comprehensive multi-elemental analysis and necessitates separate measurements for each element of interest. In contrast, ICP-OES allows simultaneous analysis of multiple elements, thus, providing a more efficient workflow and saving valuable time and resources (reagents, money and effort) [8-10]. Toxicity is dose-dependent. For health authorities and environmentalists [33-36], the dose/concentration is critical for decision-making [19-30]. Resources are also limiting factor for laboratories and researcher. The more sensitive the technique/procedure and the instrument, the more reliable is the results and hence, the decision to be taken.

## Conclusion

Therefore, it is concluded that for protecting the population health and the environment in general ICP-OES is the choice.

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