

## **Studies on Water-Harvesting Ponds (Haffirs) in Gedarif State, Eastern Sudan: III. Determination of Haffir Soil and Water Contamination with Pesticides and Hydrocarbons**

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### **Abstract**

Several states in Sudan suffer from lack of water, especially during November to June (dry-season). Therefore, some states, including Gedarif, resort to water-harvesting in pond (haffirs), which are used for drinking, agriculture and other domestic needs and activities. The current paper is one of a series of studies, 3 of them already published covered heavy metals contamination of the soil, water and the sensitivities of the ICP-OES vs. atomic absorption methods on determining their concentrations. The current paper covers the presence and levels of pesticides and hydrocarbons as pollutants in the 3 studied haffirs. Soil samples were taken from the surface of the haffirs, 30 cm and 60 cm depth, in addition to water samples every 2 wk. Samples were prepared as required, extracted, cleaned, concentrated and the concentrations of pesticides and hydrocarbons (HCs) were determined by GC-MS. The herbicide 2,4-dichlorophenoxyacetic acid (2,4-D), and the insecticides carbaryl, lindane, in addition to oils and fuel were detected at different levels. The study recommended that haffirs site selection, design, facilities, logistics and protection must be given the required attention by the health authorities, water corporations, the states and the municipalities. Further studies should be carried out to study the other pollutants, e.g. microorganisms.

**Keywords:** Water-Harvesting Ponds; Haffirs; Water; Soil; Contamination; Pesticides; Poly Aromatic Hydrocarbons; Gedarif; Sudan

### **Introduction**

Water dominates all life activities; therefore, it is of worldwide concern. The fresh water available, due to the pollution and mismanagement is decreasing. In the Sudan, there is a very strong relationship between availability of safe, clean water, poverty and meagre governmental resources. The possible solution of the latter situation is the rain-water harvesting in ponds (Haffirs). The rural people in Eastern Sudan, viz. Gedarif State, and Western Sudan (The Greater Kordofan and Dar Fur regions; 8 states) store rainwater in Haffirs. A haffir is an excavated area of land where rain-water and run-off water is harvested during the rainy-season. This harvested water is intended to be stored for human and animal consumption, in addition to other activities during the dry-season (November to June). The size of haffirs varies, depending on the location, needs, hydrology, soils, rainfall; it ranges between 5,000 and 30,000m<sup>3</sup>. Wind (throughout the year) and/or water runoff (during the rainy-season) carries soil particles and water from different areas, e.g. agricultural fields, industrial areas, mining, etc. Particles and wash water will go directly to the haffirs water. For instance: from the agricultural areas plant nutrients, e.g. Phosphorus,

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nitrogen, and potassium in the form of fertilizers, manure, sludge, insecticides, herbicides, fungicides, rodenticides and avicides can find their way to the haffirs. Moreover, crop residues, animal feces and urine, from farms and pasture can also find their way to the haffirs [1-4]. Areas of limited and intensive pesticides use showed measurable residue levels, indicating the movement of organochlorine pesticides (OCP) residues by various environmental factors [5-8]. Moreover, people around the haffirs used to wash the tractors, lorries and the likes near or inside the haffirs. This could be source for oils, lubricants, HMs, detergents, etc. One would also expect polyaromatic hydrocarbons (PAH), polychlorinated biphenyls (PCBs), dioxins and furans (PCCD/F) and other persistent organic pollutants (POPs) and persistent toxic substances (PTS) [9]. The first 3 papers on these haffirs dealt with heavy metals contamination in soil and water [2-4].

### **Objective of the Study**

The specific objectives of the current work are identifying and determining the level of pesticides residue and hydrocarbons (HCs) in 3 studied haffirs soil and water before and during the storage period.

### **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/streams that pass through the state. Average of the rainfall is approx. 612 mm. In rural and agricultural areas, people depend haffirs [1-4]. The haffirs were: Azaza, Tarfa and Elkafay.

#### **Determination of pesticide residues**

##### **Collection of soil samples**

Nine soil samples were taken from haffirs during June following the previously mentioned procedure from the soil using spiral auger of 2.5 cm diameter [1,3].

##### **Collection of water samples**

Water samples (72) were randomly collected from the haffirs in plastic bottles (July, September, November and January). 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 [1,2].

##### **Soil samples preparation**

Air-dried soil sample (3g) was transferred into 50 ml tube and 7 ml H<sub>2</sub>O was added, and allowed to be hydrated for 30 minutes. Acetonitrile (10 ml) was added to the sample, mechanically shaken for 5 minutes to extract the pesticides. The sample was centrifuged at 1,500 rpm. Citrate buffer salts was added to the contents and the tube centrifuged and the sample was shaken immediately for at least 2 minutes and finally centrifuged for 5 minutes at 3,000 rpm [1,3]. For the clean-up, 1 ml aliquot of the supernatant was transferred to a 2 ml MgSO<sub>4</sub>, PS and C18. The sample was mixed for 30 - 60 seconds and centrifuged for 2 minutes at  $\geq 5,000$  and the supernatant was filtered through a 0.2  $\mu$ m syringe filter directly into a sample vial and samples analyzed by GC-MS [5].

##### **Drinking water preparation**

One L of water sample was measured into a 2L separatory funnel; spiked with standard and was swirled to mix, then 50 ml of phosphate buffer, 50g of NaCl and 80 ml dichloromethane (DCM) were added to the water. The funnel was vigorously shaken 5-6x to allow phase-

separation (approximately 5 minutes). The procedure of extraction steps was repeated with DCM [10]. For sample clean up, the extracted sample in the organic layer was collected and filtered through anhydrous sodium sulfate into a round bottom flask. The extract was evaporated to 1 - 2 ml using the rotary vacuum evaporator at 37°C. Quantitatively, the extract was transferred to a test tube (minimum 1 ml marking), the round bottom flask was rinsed 2-3 x with ethyl acetate (EA) and transferred to a capped test tube, which was inverted several times. The EA solution was evaporated to 0.3-0.5 ml using the rotary evaporator at 35°C; the volume was made up to 1 ml with EA and mixed. The solution was transferred in to a 2 ml amber vial; 5 µl was injected on to GC-MS [10].

### Preparation of calibration standards

Working standard solutions were prepared by diluting the stock solutions to 10 µg/ml in EA. Appropriate aliquots were taken and further diluted with EA to give calibration standard solutions with concentration of 50 ng/ml. The calibration curve for each pesticide was plotted using peak area (PA) against concentration of pesticide in both the water matrix and aqueous solvent [1].

### Data analysis

Data were subjected to ANOVA and all values from chemical analyses were presented as mean ± SD and coefficient of variation (C.V. %).

### Results

The results of GC-MS analysis showed 38 different peaks, of which some were identified based on their retention time (Rt), according to the available standards and MS library, molecular formula, molecular weight and peak area (PA%); Table 1; Figure 1-3). The Rt are shown in the tables. The detected pesticides and their metabolites were the carbamate insecticide carbaryl (1-naphthylmethyl carbamate), its metabolite 1-naphthanol, the herbicide 2,4-D (2,4-dichlorophenoxy acetic acid), acetic acid, propionic acid and the OC Lindane (γ-HCH). Formulations of 2,4-D used in Sudan include esters, acids, and several salts, which vary in their chemical properties, environmental behavior, and to a lesser extent, toxicity.

Azaza Haffir				
	Carbaryl (Rt = 14.2)	1-Naphthanol (Rt = 13.8)	Propionic acid (Rt = 17.7)	Acetic acid (Rt = 8.4)
<b>Water</b>				
July	1.000	8.050	3.06	ND
Sept	0.258	ND	17.52	ND
Nov	0.254	0.891	ND	ND
Jan	ND	7.147	ND	5.230
<b>Soil</b>	2.400	4.410	ND	1.846
Elkafay Haffir				
<b>Water</b>				
July	1.599	36.120	ND	4.980
Sept	5.453	18.230	5.23	2.940
Nov	0.250	19.199	5.16	2.244
Jan	4.269	27.651	ND	ND
<b>Soil</b>	0.04	7.715	ND	0.810

Tarfa Haffir				
<b>Water</b>				
July	1.810	2.60	ND	1.780
Sept	4.269	ND	13.06	4.200
Nov	1.600	ND	ND	1.130
Jan	0.950	ND	ND	1.129
<b>Soil</b>	5.230	1.13	0.40	2.135

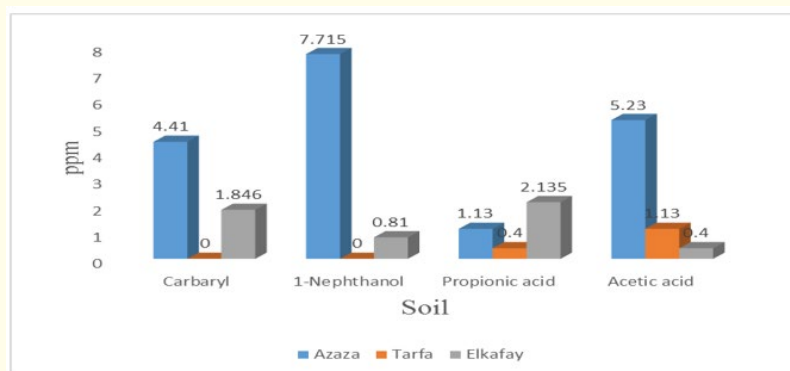
**Table 1:** Concentration (ppm) of the detected pesticides in the tested haffirs water and soil from July to January.

ND = Not Detected.

## Soil

### Carbaryl and 1-Naphthanol

The data (Table 1; Figure 1) showed that the concentration of carbaryl in the soil was high in Tarfa (5.23 ppm), followed by Azaza haffir (2.4 ppm), and the least concentration was that of Elkafay haffir (0.04 ppm). The degradation product 1-Naphthanol concentration was highest in Elkafay haffir soil (7.715 ppm), followed by Azaza soil (4.41 ppm), and the lowest concentration was detected in Tarfa soil (0.4 ppm).



**Figure 1:** Concentrations of the detected chemicals in the haffirs soil.

### 2,4-D and its metabolites

2, 4-D (acetic acid) herbicide concentrations were 1.846, 2.135 and 0.81ppm in Azaza, Tarfa and Elkafay, respectively. Propionic acid was only detected in Tarfa haffir soil at 0.4ppm (Table 1 and figure 1).

## Water

### Carbaryl and 1-Naphthanol

The concentration of this parent compound in Azaza during July was 1 ppm, in Elkafay was 1.599 ppm, whereas in Tarfa was the highest, i.e. 1.81 ppm. Two months later (September), the concentrations became 0.258, 5.453, and 4.269 ppm, following the same order of haffirs. By November, the concentration dropped to 0.254, 0.25 and 1.6 ppm, respectively. The last reading during January, resulted in ND in Azaza, 4.269 ppm in Elkafay, and 0.95 ppm in Tarfa haffir (Table 1). For the degradation product 1-naphthanol the highest

concentration detected throughout the study was that of Elkafay during July (36.12 ppm), followed by Azaza (8.05 ppm), the lowest was that of Tarfa (2.2 ppm). The concentrations during September were ND for both Azaza and Tarfa, while in Elkafay it was 18.23 ppm. Tarfa continued to be clean from this product during November, whereas Azaza showed 0.891 ppm and Elkafay increased to 19.199 ppm. Data for January, showed that Elkafay haffir water registered the 2<sup>nd</sup> highest throughout the study, i.e. 27.65, and Azaza concentration increased dramatically from 0.89 to 7.147 ppm. Tarfa showed again ND levels (Table 1).

#### 2,4-D and its metabolites

The acetic acid concentration in Azaza haffir water was ND for July, September, and November. However, 5.23 ppm was detected from January samples. Elkafay started with 4.98 ppm from July samples, dropped to 2.94 ppm for September samples, continued to drop to 2.244 ppm in November and disappeared in January samples. Tarfa haffir water started with 1.79 ppm for July, increased to 4.2 ppm for September, then dropped to 1.129 ppm for November samples, and did not significantly change for January samples (1.129 ppm; Table 1).

In terms of propionic acid (Table 1), Azaza started with 3.6 ppm for July samples, increased to 17.52 (September), then disappeared from November and January samples. Elkafay samples started with ND level (July), jumped to 5.23 ppm (September), did not significantly change (5.16 ppm) for November samples and disappeared from January samples. Also, Tarfa started with ND levels, suddenly increased to 13.06 ppm in September samples, and then disappeared in November and January samples.

#### Lindane

This insecticide was only detected in Azaz water during September (1.17 ppm) and January (0.51 ppm; Figure 2).

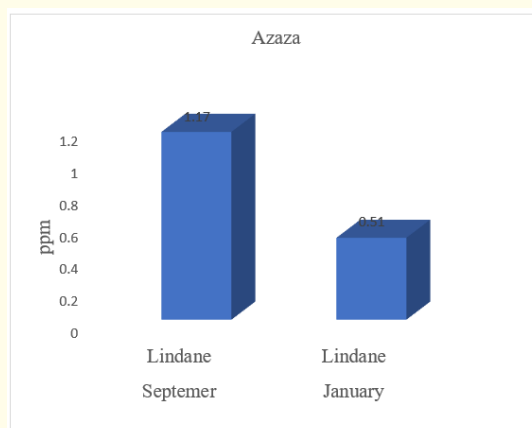


Figure 2: Concentrations of Lindane in Azaza haffir water.

#### Hydrocarbons (HCs)

The following HCs were detected (Table 2): benzene-1,2,3-trimethyl; benzene-1-ethyl 2-methyl; benzene-1,2,4-trimethyl, and benzene 1,2,4,5-tetramethyl, in addition to the 9-Octanol; Hexanol-2-ethyl, and 1-Octanol in Azaza water. Regarding Tarfa haffir water the HCs detected were benzene -1- ethyl, 3,5- dimethyl and benzene-1,2,4,5-tetramethyl. However, in Elkafay haffir water only hexadecane and undecane were found. These chemicals appeared during different months. For example, benzene-1,2,3-trimethyl was detected in Azaza water during July at 0.871 ppm and was not detected in any other sample taken later. Benzene-1-ethyl-2-methyl at 4.3 ppm was found in

September, while in November the benzene-1,2,4-trimethyl at 2.8 ppm was the only one found. These are all isomers. 9-Octanol (4.8 ppm), and hexanol-2-ethyl (0.87 ppm) 1-octanol (0.3 ppm) were detected in July, September, November and January, respectively in Azaza haffir.

The Tarafa haffir water benzene-1-methyl 1,4-dimethyl was 0.23 ppm, and benzene-1-ethyl-3,5-dimethyl was 0.29 ppm in July and September, respectively. There were no significant differences between hexadecane and undecane compounds in Elkafay haffir ( $P \leq 0.05$ ). The concentrations were 0.55, and 0.42 ppm for hexadecane and undecane (Table 2).

Azaza			
Month	Oil (Hydrocarbon)	Concentration	RT
July	Benzene-1,2,3-Trimethyl	0.871	9.94
September	Benzene-1-ethyl 2-methyl	4.3	8.4
November	Benzene-1,2,4-Trimethyl	2.8	9.12
January	Benzene 1,2,4,5-tetramethyl	0.26	12.5
July	9-Octanol	4.8	15.5
September	Hexanol-2-ethyl	0.87	10.16
November	1-Octanol	0.3	11.2
Tarafa			
July	Benzene-1-ethyl, 3,5-dimethyl	0.23	11.53
September	Benzene-1,2,4,5-tetramethyl	0.29	12.25
Elkafay			
July	Hexadecane	0.55	8.15
September	Undecane	0.42	17.7

Table 2: Water concentration (ppm) for oil (hydrocarbons) in the 3 haffirs.

## Discussion and Conclusion

Persistent pesticides, such as OCs, were used for cotton and sugarcane production prior to their severe restriction and later banning for agricultural use in the Sudan since 1983 except endosulfan in cotton Lindane for public health use (disease vector control). However, a few years ago the former was not introduced to the country [5-8]. In the Sudan, measurable levels of  $\gamma$ -HCH and heptachlor were detected in surface runoff and well-water samples surrounding pesticide stores in central Sudan [11]. In the present work the water and soil showed that Azaza haffirs water samples contain high concentrations of Lindane. The lowest concentration was found in Nov, while the highest was detected in July. Farmers used to buy smuggled Lindane and that secured for public health to control the stored products and to prevent the sesame seed bug (*Elasmolomus sordidus* Forskal; Hemiptera: Lygaeidae) from attacking the cut sesame stalks (hilla) from being attacked by this pest before collecting the seeds. However, Lindane has long-term effects on human health, including anemia, as well as liver, testicular, bone marrow, and kidney damage [12,13].

The soil concentrations of Azaza and Tarfa haffirs were significantly higher than Elkafay haffir for the carbaryl (Sevin). Carbaryl have been used for decades to control the same pest and still used. The concentrations were 2.40, 0.04, and 5.25 ppm for Azaza, Tarfa and Elkafay haffir soil, respectively. Tarfa 1-naphthanol, i.e. the degradation product of carbaryl, and carbaryl itself water concentration were significantly higher than those of Azaza and Elkafay haffirs. The result showed that the concentrations of 1-naphthanol were ranging from ND- 9.199 ppm from July to January. The high concentrations of the metabolite during July and September might reflect the intensive application of carbaryl in the previous seasons. However, the high levels of the parent compound during November and January are

reflecting the recent use during this season. Carbaryl concentrations ranged between in Azaza haffir water were 0.25 and 4.27 ppm. Acute occupational exposure of humans to carbaryl has been observed to cause cholinesterase inhibition, which impairs CNS function, resulting in nausea, vomiting, bronchoconstriction, blurred vision, convulsions, coma, and respiratory failure. Acute carbaryl exposure in humans may also cause eye and skin irritation [9,14,18-21].

Several formulations and trade name of 2,4-D are now available in the Sudanese markets, especially Gedarif State to control broad weeds as pre- and post-emergence herbicide in sorghum and several other crops. In the aqueous environment, 2,4-D is most commonly found as the free anion [22-24]. The amine salt formulations and ester formulations dissociate to the anion and, usually within one day [22]. The rate of hydrolysis is pH-dependent, with the hydrolysis half-life at pH 9 much shorter than the half-life at pH 6 [24]. Therefore, the persistence of the 2,4-D anion is of primary concern. Residues of 2,4-D can enter ponds and streams by direct application or accidental drift, which is not applicable in our case; by inflow of herbicide previously deposited in dry stream beds, pond bottoms, or irrigation channels, also not applicable to the area since it is a rainfed agricultural area; runoff from soils, which is the main source; or by leaching through the soil column. The last two reason could be behind detection of the herbicide in the study haffirs. 2,4-D residues into ponds and streams is dependent upon soil type, with coarse-grained sandy soils with low organic-content expected to leach 2,4-D into groundwater [23,25]. In soil, 2,4-D esters and salts are first converted to the parent acid prior to degradation [25]. The rate of the ester hydrolysis decreases with decreasing soil moisture and with increasing molecular weight of the alcohol portion of the ester. The fate of 2,4-D may be affected by several processes, including runoff, adsorption, chemical and microbial degradation, photodecomposition, and leaching. Water solubility and the soil adsorption coefficient (K<sub>oc</sub>) indicate the potential mobility of a chemical in soil; while the aerobic and anaerobic soil metabolism, hydrolysis half-lives, and field dissipation rate indicate the persistence of a chemical in soil [23,27].

In the present study, only Elkafay haffir showed significantly higher levels of acetic acid over of the other haffirs, and Azaza haffir was significantly higher than Tarfa haffir. However, for 2,4-D (acetic acid) herbicide the concentration was not detectable for July, September, and November, 1.85 ppm in Azaza haffir, and 4.98, 2.94, 2.24 ppm and ND, respectively, for the same order of Months, and 1.78, 4.2, 1.13 and 1.29 ppm, in Elkafay haffir.

In terms of propionic acid, Tarfa haffir concentrations were significantly higher than the acetic acid butyl. The concentrations of propanoic acid in Azaza haffir were 3.06, and 17.52 ppm, for July and September and ND in November and January. Tarfa haffir showed 5.25, and 5.16 ppm in September and November only, whereas Elkafay showed 13.03 ppm during September only. These trends were expected since the herbicide is usually applied before sowing. Exposure to this herbicide might result in fatigue, weakness, anorexia, perhaps nausea, vomiting and diarrhea. Chronic exposure may lead to CNS defects in the control of motor function [24-29].

HCs [30,31], like petroleum and its products, have become a major energy source in the century. Its use in industry and daily life activities have increased *ca.* 10x to that used previously, leading to HC contamination of both soil and water. Contamination of soil with oil spills is another major concern. It is now commonly known that contaminated soil is a serious, often lethal hazard to the health of humans, and polluting ground water, environment, and consequently decreases overall productivity of agricultural land. HCs concentration in Azaza haffir water (benzene compounds), were 0.871, 4.3, 2.8 and 0.26 ppm, and of the alcohols was 4.8, 0.87, 0.3 and 0.0 ppm, for July, September, November and January, respectively. As for Tarafa haffir, the of concentrations was 0.23, and 0.29 ppm for Elkafay haffir. No significant differences between hexadecane and undecane compounds were detected. These levels might pose severe immediate and long-term influence, since many HCs constituents are toxic in nature. These pollutants persist in soil and water for a very long time often decades. The effects of all detected contaminants in these haffirs in this series of publications [1-4] will be reported in the next publications in terms of different types cancer in Gedarif State and kidney problems.

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