

## Monitoring of Organochlorine Pesticide Residues in Groundwater Samples from Sana'a City (Yemen) by Gas Chromatography Using Solid Phase Extraction Method

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### ABSTRACT

Residue values of organochlorine pesticides (OCPs) in groundwater samples from Sana'a, the capital of Yemen, are reported. A total of 60 water samples from 10 main sampling sites in Sana'a City were extracted with the extraction solvents (Dichloromethane : hexane; 3:1) and Solid Phase Extraction (SPE) method was used for the extraction of residues in water sample. The analysis of the 18 OCPs (4,4-DDD, 4,4-DDE, 4,4-DDT, a-endosulfan, a-HCH, aldrin, b-endosulfan, b-HCH, d-HCH, dieldrin, endosulfan sulfate, endrin, endrin aldehyde, endrin ketone, heptachlor, heptachlor epoxide, lindane and methoxychlor) was carried out by capillary gas chromatography equipped with an electron capture detector ( $^{63}\text{Ni}$ ), split/splitless mode. Three residues of 18 targeted OCPs were found in 3 samples only. Among the various pesticides analyzed only, aldrin (0.0002  $\mu\text{g/l}$ ), dieldrin (0.0007  $\mu\text{g/l}$ ) and 4,4-DDT (0.0005  $\mu\text{g/l}$ ) were found in the groundwater samples. For all compounds examined, all concentration levels of residues were found to be within WHO and European Economic Commission Standards (EEC) for drinking water quality.

**Keywords:** Organochlorine, pesticide residues, groundwater, Sana'a gas-chromatography, solid phase extraction

### INTRODUCTION

Organochlorine pesticides (OCPs) have an extended history of widespread use worldwide. In spite of their proscription and restrictions, many countries are yet using them for various intentions (public health, agriculture), because of the low cost, ease of use, versatility in controlling various insects (Afful *et al.*, 2010). Yemen still utilizes considerable quantities of some organochlorine pesticides, such as DDT, HCB, dieldrin, heptachlor and polychlorinated biphenyls (PCBs). Therefore, they were generally found in shore sediments and marine organisms in the Gulf of Aden coast in trace quantities (DouAbul and Al-Shwafi., 2000). OCPs are among the oldest agrochemicals introduced to control insect pests; they first introduced in the 1940s, and were used extensively until most of them were banned worldwide in the 1970s and 1980s due to their health risks. According to the pesticides manual for the Republic of Yemen, the Yemeni government has banned use of most of persistent OCPs (aldrin, chlordane, dieldrin, endrin, heptachlor, mirex, toxaphene and DDT) since 1990. However, it is believed that smuggling of OCPs still taken place (Ministry of Water and Environment MWE 2003). Organochlorine pesticides are very persistence liposoluble compounds and are capable of bio-accumulating in the fatty parts of biological beings such as breast milk, blood and fatty tissues via the food chains (William *et al.*, 2008). Several studies have indicated the presence of OCPs in water (David *et al.*, 2011; Farshid *et al.*, 2011 and Narasimha *et al.*, 2012), human breast milk (Ennacer *et al.*, 2008 and Okonkwo *et al.*, 1999), soil (Getenga *et al.*, 2004), foodstuffs (Ahmad *et al.*, 2013; Hajslova and Zrostlikova., 2003 and Qin *et al.*, 2011), wildlife (Tanabe *et al.*, 1993) sediments (Fatoki and Awofolu., 2003 and Safina *et al.*, 2011), air (Aydin and Yurdin., 1999), fish (Farshid *et al.*, 2012), serum (William *et al.*, 2008) and in mussels (Prudente *et al.*, 1999). Many of these OCPs and their metabolites have been involved in a wide range of toxic and adverse human and environmental effects including reproduction and birth defects (Hileman, 1994; Edwards, 1987 and Bouman, 2004), immune system malfunction (Kolpin *et al.*, 1998), endocrine disruptions (Adeyemi *et al.*, 2008 and Izelyamu *et al.*, 2007),

testicular tumors and lower sperm counts in humans (Davies and Barlow., 1995; Cocco *et al.*, 1997), human breast and liver cancers (Garabrant *et al.*, 1992), altering the metabolism of xenobiotics and steroid hormones (Colborn and Smolen., 1996). OCPs have also been linked to chronic convulsions (Belta *et al.*, 2006), Parkinsons diseases (Fleming *et al.*, 1994). Information of OCPs in sources of drinking water from Sana'a City is limited, no single research paper was found to describe the situation of groundwater contamination by OPCs, this study describe the organochlorine pesticide result in groundwater in Sana'a City. The objective of this study was to evaluate the spectrum of organochlorine pesticides residues in groundwater samples of Sana'a City by solid phase extraction method and analysis with GC-ECD.

### MATERIALS AND METHODS

#### 1- Chemicals and reagents

OCPs Certified Reference Material (4,4-DDD, 4,4-DDE, 4,4-DDT, a-endosulfan, a-HCH, aldrin, b-endosulfan, b-HCH, d-HCH, dieldrin, endosulfan sulfate, endrin, endrin aldehyde, endrin ketone, heptachlor, heptachlor epoxide, lindane and methoxychlor) were purchased from Sigma-Aldrich (Germany). Stock solutions (1000  $\mu\text{g/ml}$ ) were prepared by dissolving the pesticide standards in a mixture of toluene and n-hexane in the ratio (1:1) and a fresh working solution containing a mixture of each analyte was prepared by stepwise dilution of the stock solution in n-hexane according to the standard practices. Organic solvents (Dichloromethane, Ethylacetate, Methanol, Toluene &, n-Hexane) were of pesticide residue analysis and were obtained from Merck (Darmstadt, Germany). Solid Phase Extraction (SPE) cartridges were obtained from International Sorbent Technology.

#### 2- Study area and sampling

The sampling sites were composed of ten sources of drinking water wells in Sana'a City, a total of 60 water samples from 10 main sampling sites were collected. After collection of the water samples, the water sample was filtered through watt man filter paper and analyzed by validated method. All the water samples were collected in high purity glass bottles and immediately transported to the

laboratory. The samples were stored at +4°C and extraction of the OCPs was performed within 48 h. Global positioning system was used to identify the location of collected samples (Table 1).

**Table 1. Name and location of collected samples**

	Site	Coordinate	
		longitude	Latitude
1.	Al-Saleh mosque	15°19'18.69"N	44°12'26.01"E
2.	Sana'a University	15°21'58.91"N	44°10'57.99"E
3.	Maeen district	15°22'10.19"N	44°11'06.50"E
4.	Mathbah	15°23'01.27"N	44°10'14.33"E
5.	Al-andulos zoon	15°22'41.89"N	44°10'30.02"E
6.	Sawan	15°22'49.04"N	44°14'19.45"E
7.	Al-hasabah	15°23'03.48"N	44°12'47.76"E
8.	Fifty street	15°21'41.28"N	44°09'19.78"E
9.	Al-asbahi	15°17'45.15"N	44°13'56.06"E
10.	Bani-huat	15°28'27.77"N	44°13'57.73"E

### 3- Sample preparation

The extraction procedure, recovery and quality control was completed as described by (Ashok and Aman., 2013): Solid Phase Extraction (SPE) was used for the extraction of organochlorine pesticide residues in water sample. The collected water samples were pre-filtered through a 0.45 µm nylon filter (Wattman) to remove particulate matter and were acidified with hydrochloric acid (6N) to pH 2.5. Methanol modifier (10 mL) was added to water sample for better extraction. SPE using pre-packed reversed phase octadecyl (C-18 bonded silica) contained in cartridges was used for sample preparation. Prior to the extraction, the C-18 bonded phase, which contains 500 mg of bonded phase, was optimized with 5 mL of ethylacetate, 5 mL of dichloromethane, 10 mL of methanol and 10 mL of organic free water before use. The sample was mixed well and allowed to percolate through the cartridges with flow rate of 10–15 mL/min under vacuum. After sample extraction, suction continued for 15 min to dry the packing material and pesticides trapped in the C-18 bonded phases were eluted by passing 10 mL extraction solvent (Dichloromethane : hexane; 3:1) and fraction was evaporated in a gentle steam of nitrogen. Final volume was made up into 1 ml by n-hexane and injected 1 µl in gas liquid chromatograph equipped with an electron capture detector (Ni<sup>63</sup>).

**Table 2. Concentrations of organochlorine pesticides detected in the groundwater samples**

Organochlorine pesticide	Concentrations (µg/l)									
	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
a-HCH	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
b-HCH	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
lindane	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
d-HCH	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Heptachlor	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
aldrin	nd	nd	nd	nd	nd	0.0002	nd	nd	nd	nd
heptachlor epoxide	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
a-endosulfan	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
4,4-DDE	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
dieldrin	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.0007
endrin	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
b-endosulfan	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
4,4-DDD	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
endrin aldehyde	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
endosulfan sulfate	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
4,4-DDT	nd	nd	nd	nd	nd	nd	nd	nd	0.0005	nd
methoxychlor	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
endrin ketone	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd

nd: not detected

### 4- Instrumental and operating conditions

The analysis of the 18 selected OCPs was carried out by capillary gas chromatography using Shimadzu 2010 (Shimadzu, Kyoto, Japan) chromatograph equipped with an electron capture detector (<sup>63</sup>Ni), split/splitless injection port, a DB-5 (5 % phenyl polysiloxane) fused silica capillary column by J&W Scientific (30 m x 0.25 mm i.d., 0.25 µm film thickness) and auto-injector model AOC-20i. Helium was used as the carrier gas while nitrogen was used as the make-up gas. The temperature program was as follows: 80 °C for 1 min, 80–218 °C at 8 °C min<sup>-1</sup>, 218 °C for 18 min, 218–250 °C at 4 °C min<sup>-1</sup> and 250 °C for 10 min. A 1 µl volume was injected in a splitless mode at 250 °C. The residues of organochlorine pesticides were determined by comparing the peak areas of the samples and the calibration curves of the standards.

### Quality Assurance

For every set of 10 samples, a procedural blank and spiked sample consisting of all reagents was run to check for interference and cross-contamination. The recovery experiment was performed at three concentrations and each concentration was analyzed in triplicate. All residues are expressed as µg/l. minimum, maximum and geometric means were used to express residue levels. Values below detection limits were assigned as a Not-Detectable (ND). The World Health Organization 1993, and European Economic Commission (EEC) 1980, has set allowable residual limit of individual and total concentration in drinking water set at 0.1 and 0.5 µg/l respectively. The method should be able to extract the pesticides from water samples at the guideline levels. Hence the method is validated by spiking the doubled distilled water with 0.5 ppb level of mixture of pesticides of different group, and analysed. The method's efficiency concerning 18 ECD-sensitive compounds has been validated and found satisfactory.

## RESULTS AND DISCUSSION

Results of sample analyses are summarized in Tables (2). These analyses were carried out on samples from different localities and various surface waters in Sana'a City.

Three residues of 18 targeted OCPs (4,4-DDD, 4,4-DDE, 4,4-DDT, a-endosulfan, a-HCH, aldrin, b-endosulfan, b-HCH, d-HCH, dieldrin, endosulfan sulfate, endrin, endrin aldehyde, endrin ketone, heptachlor, heptachlor epoxide, lindane and methoxychlor) were found in 3 samples only. Among the pesticides analyzed only, aldrin (0.0002 µg/l), dieldrin (0.0007 µg/l) and 4,4-DDT (0.0005 µg/l) were found in the groundwater samples. The mean total concentration of 0.0007 µg/l observed for dieldrin was the highest while mean total concentration of 0.0002 µg/l observed for ardrin was the lowest. Most of the published research work used the conventional extraction and clean-up methods, in the current work a SPE was used because it is: affordable, convenient, simpler, efficient extraction and less solvent consumption (Albanis and Hela., 1995; Crescenzi *et al.*, 1997; Miliadis 1998; Doong 1999 and David *et al.*, 2011). For all compounds examined, all concentration levels of residues were found to be within WHO Guidline (World Health Organization 1993) and European Economic Commission Standards (EEC Council Directive 1980/778/EEC) for drinking water quality. These results demonstrate that there are no existed certain potential health risks to drinking water consumer in Sana'a city. Table (2) showed that only three groundwater sampling areas (Sawan, Al-asbahi and Bany huat) was found to contain pesticide residues. It is well known that aldrin and dieldrin are closely related organochlorines, when aldrin is applied in the field; it is rapidly broken down to dieldrin (Turgut 2003), that is explaining the presence of dieldrin in some sampling areas. Since no information from earlier studies regarding OCPs residue in Yemen, it will be difficult to compare our results. The present results are the first of their kind for Yemen and should serve as background information.

Factors influencing pesticide movement to groundwater include properties of the pesticide, properties of the soil, rainfall and depth of ground water, method and rate of application of pesticide (Buttler *et al.*, 1993). The Organochlorines solubility in water is low; their mobility in soil is generally limited, although it is greater in sandy soil. They tend to be bound in clay soils with limited leaching. It is well known that a high clay content and organic matter content exhibit the high adsorption capacity, it was also noted that the relatively high organic matter content and clay content of soil restricted the leaching of the soil. However, the key factors in determining whether pesticide residues will be leached to groundwater are primarily the mobility of the compound and secondarily its persistence (Foster and Chilton., 1998). There are several reasons for Organochlorine pesticides contamination of groundwater: due to their molecular features (Ahmed *et al.*, 1998); persistent nature and translocation from agricultural land (Matin *et al.*, 1998); the abundant application and or long half-life period in a neutral environment (Tomlin 1994); faults in the well casings allowing surface water into the wells, also there could be large fractures in the bedrock which result in relatively fast flow of groundwater and recharge from the surface (Eitzer and Chevalier., 1999).

The ground water level in Sana'a basin is very deep, most borehole depths in the Tawilah Sandstone aquifer vary from 100-400 m. Depth to water is between 50-150 m in most boreholes. The thick, unsaturated zone, resulting from deep groundwater levels (100-170 m below ground level) suggests that groundwater pollution is unlikely. However, the complex geological structure and the presence of rock fractures could reduce the travel time of pollutants through this layer. The use of pesticides and chemical fertilizers in agriculture in Yemen is, however, still at a relatively low level and therefore groundwater contamination from this source is not of major concern at present (Richard *et al.*, 1997).

Ground water quality at Sana'a city wasn't within the concern of the related parties, that is may be due to the absence of well-equipped accredited laboratories in the country. Only two earlier studies were found to describe the quality of groundwater: (Al-Hamdi, 1994) investigated the quality of groundwater in the city of Sana'a, they concluded that no immediate risk was thought to exist for the wellfields. (Al-Eryani *et al.*, 1991) concluded that industrial wastewater in the Sana'a area was not presenting an immediate threat to the quality of the groundwater; their focus was on heavy metals and inorganic pollutants.

## CONCLUSION

This method has been developed for the simultaneous extraction and cleanup of 18 OCPs in groundwater samples. SPE with DCM:hexane technique showed to be viable and reliable method, it also has been shown to be rapid alternative for the quantitative and qualitative analysis of OCPs in groundwater samples. The method is inexpensive simple, precise and reproducible for the analysis of wide range of OCPs pesticides. The proposed method can be applied to the fortified and real groundwater samples contaminated with OCPs at the µg/l level. We recommend that on-going monitoring be continued to determine the OCPs residue levels and further studies to be carried out to determine the pathways of OCPs contamination in groundwater. The method may serve as a screening protocol for the determination of OCPs in groundwater samples on a routine basis.

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### رصد متبقيات المبيدات العضوية الكلورية في عينات المياه الجوفية في مدينة صنعاء - اليمن بواسطة الكروماتوجرافي الغازي و الاستخلاص باستخدام طريقة الطور الصلب ( SPE ) جلال عوض و محمد هاشم قسم وقاية النبات، كلية الزراعة، جامعة صنعاء، اليمن

نورد في هذا البحث قيم متبقيات المبيدات العضوية الكلورية في عينات المياه الجوفية من مدينة صنعاء، اليمن. تم استخلاص ما مجموعه ٦٠ من عينات المياه الجوفية من ١٠ مواقع رئيسية مستهدفة لأخذ العينات في مدينة صنعاء بواسطة مذيبات الاستخلاص (ثنائي كلورو ميثان : الهكسان؛ ٣ : ١). كما استخدمت طريقة الطور الصلب (SPE) لاستخلاص متبقيات المبيدات في عينات المياه الجوفية. استخدم جهاز الكروماتوجرافي الغازي ذو الخاصية الشعرية والمزود بكاشف اللاقط الإلكتروني (ECD) في تحليل متبقيات المبيدات العضوية الكلورية وتم الكشف عن ثلاث مركبات فقط من بين ال ١٨ مبيد قيد الدراسة. من بين المبيدات المختلفة التي حلت، تم فقط العثور على مبيد الألدرين (٠.٠٠٠٢ ميكروغرام / لتر)، مبيد الديلدرين (٠.٠٠٠٧ ميكروغرام / لتر) و مبيد ٤،٤-دي دي تي (٠.٠٠٠٥ ميكروغرام / لتر) في عينات المياه الجوفية. لكل المركبات قيد الدراسة، وجد ان كل مستويات التركيزات للمتبقيات تقع ضمن الحدود المسموح بها من قبل كلاً من وكالة حماية البيئة في الولايات المتحدة الأمريكية و منظمة الصحة العالمية ومعايير اللجنة الاقتصادية الأوروبية (EEC) لجودة مياه الشرب.