

**MONITORING OF PESTICIDE RESIDUES IN
DRAINAGE, CANAL AND TAP WATERS AT
DAMANHOUR, EL-BEHIRA GOVERNORATE, EGYPT.**

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ABSTRACT

The study was carried out to monitor the organochlorine (OC) and organophosphorus (OP) insecticide residues in drainage, canal and tap waters of Damanhour city, Egypt. Pesticides monitored were selected among organochlorine and organophosphorus insecticides. Results revealed that, the recovery efficiencies of the tested compounds yielded more than 85.5% at a fortification level 5 ng/ml. Detection limits of each compound ranged between 10 to 30 ng/L. Also results confirm the presence of eight pesticide residues representing the OC compounds in drainage, canal and tap waters. Heptachlorepoide (0.16 µg/l), p,p-DDE (1.9 µg/l), dieldrin (0.039 µg/l) and p,p-DDD (1.55 µg/l) residues were found to be higher in drainage water than the permissible limits of these compounds. Also, the OP pesticide residues (ethoprophos) were found only in drainage water. In general pesticide residues in summer water samples were relatively higher than that of winter water samples. In conclusion, drainage water was higher polluted and contains more pesticide residues than canal and tap waters. This means that the discharging of agricultural wastes in the water supplies must be controlled.

Key words: OP pesticides; OC pesticides; SPE; MLLE; GC/MS.

INTRODUCTION

In Egypt, the common and extensive use of pesticides in agriculture, usually involving a wide broadcast application and spread out in large crop field areas, is recognized to be a major source of diffuse contamination, typically by leaching to the underlying aquifers or by runoff to the surface waters. Of particular concern are later ones, since they are often used as supply resource for human drinking. Monitoring pesticides in water samples has been an issue of great relevance in the last decades. The EEC Drinking waters directive (ECC, 1988&1998) establishes a concentration threshold of 0.1 µg/l for each individual pesticide or 0.5 µg/l for total amount of them, including their main metabolites. In order to reach the levels of sensitivity for the determination of the residues of pesticides allowed in drinking water, an extraction and enrichment steps are always necessary prior instrumental analysis. Gas chromatography (GC) has been one of the most often employed analytical techniques for determination of pesticides. The coupling of GC with mass spectrometry (MS) has been extensively used, as it increasing the selectivity and certainly of identification, avoiding false positive multiresidue determination in water (Pablos-Espada et al., 1999). As agricultural activities are the principle source of pesticides in El-Behira Governorate, it is expected that concentrations in agricultural drains (El-Khairi drain) and the drinking water supply are even higher. Therefore, the present study was carried out to monitor the level of organochlorine and organophosphorus pesticide residues in El-Khairi drain water, El-Khandk canal water and tap water of Damanshour, El-Behira Governorate in winter and summer of 2008.

MATERIALS AND METHODS

Sampling :

Surface water samples (each of 5 L) were collected in glass bottles fitted with Teflon-lined caps from the surface water of EL-

Khairy draining canal, El-Khandk canal and tap water. Collection of water samples was repeated at January and July in 2008 .In the laboratory, the collected water samples were extracted quickly after filtration through a filter paper.

Pesticide standards and chemicals:

A technical grade samples of pesticides were obtained from Riedel-de Haen, Seelze, Germany. Stock standard solutions of 500 ng/ml were prepared by dissolving each pesticide in methanol and storing at -4°C . Working standard solutions were obtained by diluting the stock solutions in methanol. All reagents were of analyticalreagent grade from Merck (Darmstadt, Germany) unless stated otherwise.

Extraction procedures:

Two different extraction procedures were adopted in order to compare extraction efficiencies of the organochlorine and organophosphate pesticide residues from water as follows:

Solid phase extraction (SPE):

Solid phase extraction cartridge (Sep-pack plus) packed with 500 mg of C18, obtained from (Milford) MA, USA., were conditioned by successive elution with 10 ml of a mixture acetonitrile:dichloromethane (1:1) V/V, 5 ml of methanol and 3 ml of distilled water (three times) . All the solvents were passed by gravity throughout the cartridges. Then, an aliquot of 500 ml water sample was aspirated through the cartridge under vacuum. The sample flow rate was controlled at ca. 8-10 ml /min . The cartridge was not allowed to dry completely during the extraction process. Before eluting the pesticides, the cartridge was dried by passing air for 15 min. Pesticides were eluted by gravity with 5 ml of dichloromethane: acetonitrile (1:1) v/v followed by 2 ml of n-hexane. The extract was dried over anhydrous sodium sulfate (Na_2SO_4). The extract is evaporated in rotary evaporator using a 40-50°C water – bath. The final volume is adjusted to 2 ml with n- hexane (US EPA 199 and Vinuesa et al., 1989).

Micro Liquid Liquid extraction (MLLE):

In this method, a hand made extraction apparatus which consists of a container to push deionized water, was used. This container has a valve at its end and is connected by a Teflon tube which measure 40-60 cm to a separatory funnel which has a volume of 500 ml and has a valve at its end the other end is occupied by a connection piece has an inverted Y shape. Five hundred ml of water sample were transferred into a 500 mL separatory funnel and then 0.5 mL of n- hexane pure was added. The mixture was mechanically shaken for 2 min. The supernatant organic phase was raised up to the bottleneck of the separatory funnel by raising a communicating vessel filled with deionized water. Raising of the level ensures the collection of the liquid with a Pasteur micropipette, the extract being thus ready to inject into the GC-MS according to the technique developed by (Fernández *et al.*, 1998 and Tahbboub *et al.*, 2005).

Analysis of Organochlorine Pesticide Residues:

Extracts of water samples (1-2ul) were analyzed for 18 OC's utilizing a GCECD (Variation 3400, Walnut Creek, CA, USA). The GC was equipped with a varian 8200 auto sampler and split / splitless part which was operated in splitless mode (Purge time set at min) and maintained at a temperature of 250° C chromatographic separation of OC's. were achieved using HP-608 fused silica capillary column (30 m X 0.53 mm i.d.S 0.5 um film thickness). Helium was used as the carrier gas and nitrogen as the make up gas. Separation conditions for GC- ECD were as follows: Initial column temperature set at 80° C for 6 min. It was increased to 215° C (hold for 1 min.) at a rate of 15° C / min., then to 230° C at a rate of 5° C / min. and finally to 290° C (hold for 2 min.) at a rate of 5° C /min. The GC detector autosampler was programmed for injection 1-2µl . For quantification of OCs in extracts of water samples, a 1-2 ul were injected into GC- ECD operated under conditions as described before. The concentration of each OC compound was determined from calibration curve (internal standard method of calibration). The calibration curves (5 points) for all OCs under investigation were constructed using an OCs fortified ultra pure water at concentration levels, 0.01-10 ug/L of each

compound used applying the extraction and the analytical procedure as described before. The integrated peak areas obtained from the Reconstructed Ion Chromatogram (RIC) corresponding to the quantitation ion were plotted versus the concentration which were used for fortification. The median detection limit MDL and limit of quantification LOQ were determined. The MDL was determined using the fortified water samples with OCs standards mixtures. The fortified water samples were extracted and analyzed as before mentioned. The MDL was calculated as the lowest concentration of OC which provides a chromatographic peak height 3 times the average base line noise (at the same retention time) obtained from the GC-ECD of the extract of non- fortified water sample (Blank).

The limit of quantification (LOQ) was determined corresponding a value of ten times the base line noise in GC- ECD chromatogram of blank sample (PAM,1994 and U.S.EPA, 1994).

Analysis of Organophosphorus Pesticide Residues:

Extracts of water samples (1-2ul) were analyzed for ten OP pesticides utilizing a bench top Gas Chromatography-Mass Spectrometry 9 Gas Chromatography-Ion Trap Detector, GC-ITD) which consisted of a Varian 3800 series Gas Chromatograph interfaced to a Saturn 2000 equipped with a split/split less part which was operated in split less mode (purge time set at 1 min) and maintained at a temperature of 250°C. All chromatographic separation were achieved using an HP- 5 MS capillary column, 30m x 250 um ID and 0.25 um film thickness. The carrier gas was helium at a constant flow rate of 1.1 ml/min. The temperature at the column was initially set at 85° C for 0.3 min. It was increased to 150° C (hold for 4 min) at a rate of 30° C / min., then to 185° C at a rate of 2° C / min. and finally to 290° C (hold for 5 min) at a rate of 4° C/min. The ITD was operated in Electron Impact ionization mode (EI) at 70 eV and temperature at 220° C. EI spectra were monitored by scanning ions within the range of 50-500 a. m. u. The GC-ITD was controlled by a computer system which have EI-MS libraries (Willey spectral library of more than 140000 compound). The target compounds were identified by their full scan mass spectra and retention time using the

total ion current as a monitor to give a Total Ion Chromatogram (TIC). The use of the full scan mode allows to compare the spectrum obtained for interested compounds with the EI- MS libraries. In addition, Selective Ion Monitoring (SIM) mode was used for identification (mass spectra in elution time window were searched for 3 selective specific ions for the compound of interest. For quantification of OPs in extracts of water samples, 1-2 ul were injected into GC- ITD operated under conditions as described before. The concentration of each OP compound was determined from calibration curve (Internal Standard method of calibration). The calibration curves (5 points) for all OPs under investigation were constructed using an OPs fortified ultra pure water at concentration levels, 0.01-10 ug/L of each compound used applying the extraction and the analytical procedure as described before. The integrated peak areas obtained from the Reconstructed Ion Chromatogram (RIC) corresponding to the quantification ion were plotted versus the concentration which were used for fortification. The MDL and LOQ were determined (PAM ,1994 and U.S.EPA,1994) using the fortified water samples with Ops standards mixtures. The fortified water samples were extracted and analyzed as before mentioned. The MDL was calculated as the lowest concentration of OP which provides a chromatographic peak height in the GC-ITD-RIC, 3 times the average base line noise (at the same retention time) obtained from the GC-ITD-RIC of the extract of nonfortified water sample (Blank). The limit of quantification (LOQ) was determined corresponding a value of times the back noise in GC- ITD-RIC of blank sample.

RESULTS AND DISCUSSION

Results in Tables 1 and 2 show the percent recovery values (Rec.%) and percent relative standard deviation (RSD) obtained from fortified samples for both methods of extraction SPE, MLLE. The average recovery percentages for the OCs ranged from 89.9 ± 6.5 to 100.5 ± 6.5 % and from 85.5 ± 8.1 to 98.1 ± 6.5 % for MLLE and SPE methods,

respectively (Table 1). The corresponding average recovery percentages for OPs ranged from 92.3 ± 7.5 to 103.4 ± 7.5 % and from 89.2 ± 5.5 to 98.2 ± 7.3 % for MLLE and SPE methods, respectively. In general, these values are approximately similar to the reference values reported in the EPA methods (U.S.EPA,1991 & 1984) , and can be considered acceptable according to them for the compounds analyzed (recoveries of 70 to 130 % and RSD below 30 %) . In determination of pesticide residues in samples of drainage and drinking waters, SPE method was actually applied because of its greater operation simplicity. All data of residue analysis were corrected according to these obtained recovery percentage values. Minimum detection limits were determined using the two methods of extraction, for both OC and OP compounds. Results in Table 1 show that the minimum detection limits for the OC compounds were ranged from 10 to 25 and from 12 to 30 ng / l by using MLLE and SPE methods, respectively . The corresponding values for the OP compounds were ranged from 12 to 22 ng / l for MLLE method and from 10 to 20 ng / l for SPE method (Table 2).

Analysis of water samples of El- Khairy drainage canal revealed the presence of eight OC insecticides; B-HCH, heptachlorepoxyde, p,p'-DDE, dieldrin, p,p'-DDD, endrin aldehyde, endosulfan sulfate and endrin ketone (Table 3). Also, only three OP insecticides, ethoprophos, diazinon and fenthion, were detected in drain water (Table 4).

Results in Table 3 revealed the presence of heptachlorepoxyde , dieldrin , p,p'- DDE and p,p'-DDD in winter drain water with concentrations of 0.16 , 0.039 , 1.9 , 1.55 $\mu\text{g} / \text{L}$, respectively . The corresponding values for summer water samples were 0.235, 0.046, 2.073 , 1.586 $\mu\text{g} / \text{L}$ respectively. The concentration of these OCs were relatively higher in summer - than in winter water samples. On comparison of these results with that of water pollution rule of (WHO,1995 and EMHR,2007) , it is clear that heptachlor epoxyde , p,p DDE , dieldrin and p,p DDD concentrations were more than the permissible limits of these compounds.

Table 1 Average recovery percentage (Rec.%) , relative standard deviation (RSD) and minimum detection limits (MDL) of OCs by using the two methods of extraction.

OCs	MLLE method		SPE method	
	(Rec.%)±RSD	MDL ng/ L	(Rec.%)±RSD	MDL ng/ L
Alpha – HCH	100.5±6.5	10	98.1±6.5	12
Gamma- HCH	99.5±7.012	18	89.2±7.5	22
Beta – HCH	93.4±7.1	20	88.2±6.7	24
Heptachlor	99.6±10.1	12	91.2±4.5	14
Delta HCH	98.9±8.1	10	95.2±6.3	12
Aldrin	92.8±8.9	12	90.5±6.5	14
Heptachlorepoide	93.3±7.1	12	90.1±6.3	15
Endosulfan I	90.1±5.5	20	89.5±7.8	25
P,P' - DDE	100.1±4.5	25	95.1±5.5	30
Dieldrin	100.2±4.5	10	95.5±7.1	12
Endrin	94.6±7.1	15	90.1±7.5	20
P,P' DDD	89.9±6.5	15	85.5±8.1	21
Endosulfan II	94.6±5.5	12	90.2±8.5	14
P,P' DDT	95.1±6.1	10	90.5±5.5	12
Endrin aldehyde	99.2±7.0	12	91.5±5.6	15
Endosulfan sulfate	98.4±7.2	13	90.3±5.1	15
Methoxychlor	97.1±7.2	10	91.2±6.2	12
Endrin Ketone	98.2±6.5	11	89.3±7.3	15

Table 2 Average recovery percentage (Rec.%) , relative standard deviation (RSD) and minimum detection limits (MDL) of OPs by using the two methods of extraction.

OPs	MLLE method		SPE method	
	Rec(%)±RSD	MDL ng/ L	Rec(%)±RSD	MDL ng/ L
Mevinophos	99.1±6.5	12	90.1±6.5	14
Ethoprophos	92.3±7.5	13	89.2±5.5	12.5
Diazinon	95.4±6.5	18	90.1±6.5	16
Parathion - methyl	99.1±7.3	20	91.2±7.5	18
Tetrachlorvinphos	102.3±6.5	22	95.6±6.7	19
Fenthion	103.4±7.5	14	96.2±7.1	10
Fensulfothion	100.1±6.5	12	98.2±7.3	10
Azinophos – methyl	99.2±7.5	20	95.3±8.2	15
Sulprofos	101.2±6.5	18	97.2±7.2	15
Coumaphos	100.3±8.5	20	98.1±6.5	20

Table 3 Mean ± SD of organochlorine insecticides residue levels (ug/L) in drain water samples.

Name	Mean ± SD	
	Winter samples	Summer samples
Beta – HCH	0.104 ±0.062	0.119 ±0.078
Heptachlorepoide	0.16* ±0.061	0.235* ±0.129
P,P' - DDE	1.9* ±0.417	2.073 *±0.585
Dieldrin	0.039* ±0.013	0.046 *±0.015
P,P' DDD	1.55* ±0.575	1.586 *±0.618
Endrin aldehyde	0.117 ±0.031	0.122 ±0.032
Endosulfan sulfate	0.044 ±0.025	0.051 ±0.023
Endrin Ketone	0.151 ±0.045	0.163 ±0.055
Total	4.065	4.395

* Values are exceeding the permissible limits.

Table 4 Mean \pm SD of organophosphorus insecticides residue levels ($\mu\text{g/L}$) in drain water samples.

Name	Mean \pm SD	
	Winter samples	Summer samples
Ethoprophos	3.163 \pm 1.704	3.288 \pm 2.241
Diazinon	3.033 \pm 0.768	3.633 \pm 1.013
Fenthion	2.533 \pm 0.586	3.1 \pm 1.015
Total	8.729	10.021

Analysis of water samples of El- Khandk drinking water canal and tap water revealed the presence of eight OC compounds. The concentration of these OCs were relatively higher in summer water samples than that in winter water samples. The concentrations of heptachlorepoide and p,p'-DDE were 0.14 and 3.15 $\mu\text{g/l}$., respectively, in El-Khandk drinking water and 0.155 and 1.25 $\mu\text{g/l}$., respectively, in tap water of Damanhour (Table 5). These detected OC residues in drinking water were relatively higher than the maximum residue levels (MRL) for these two OCs (ECC,1998, WHO,1995 and EMHR, 2007).

Data based on the grand total concentrations of OC and OP insecticide residues showed that the most polluted water were collected at summer season of 2008. Mean of OCs was 4.065 in winter and 4.395 in summer. However, mean of OPs was 8.792 in winter and 10.021 in summer, as shown in Tables (3 and 4). These results can be explained, in general trends , taking into account their physiochemical properties , such as persistence half life , hydrolysis and mobility (solubility and adsorption), that govern their environmental behavior, together with the extent of their use .On the other hand, concentrations of alpha HCH , heptachlor , delta HCH , aldrin , endosulfan I, endrin , endosulfan , p, p -DDT and methoxychlor did not detected in water samples. Despite the absence of DDT , heptachlor , aldrin . endrin and endosulfan insecticides, their degradation products such as DDE , DDD , heptachlor epoxide , dieldrin , endrin aldehyde , endrin ketone and endosulfan sulfate were dominant in water samples . This may be

due to the different mechanisms such as microbial effect and oxidation (Matsumura, 1972), photoionization, electron transfer (Bowma and Sans, 1980 and Laymann *et al.*, 1990) and aerobic biodegradation, hydrolysis and photodegradation in surface water (Veloguleti *et al.*, 2002).

As regards to the organophosphorus insecticides , ethoprophos , diazinon and fenthion were detected only in drain water samples. Results in Table 4 revealed the presence of ethoprophos, diazinon and fenthion in winter drain water samples with concentrations of 3.163 , 3.033, 2.533 $\mu\text{g/L}$.,respectively. The corresponding concentration in summer drain water samples were 3.288, 3.633 and 3.1 $\mu\text{g/L}$., respectively. Thus, the concentrations of these OPs in summer water samples were relatively higher than that in winter water samples. However, the rest of the OP insecticides, mevinophos , methylparathion , tetrachlorvinphos , fensulfothion , sulprofos and coumaphos did not detected in all of the tested drain water samples.

Organophosphorus insecticides are quickly degraded in aquatic environment where the alkaline media accelerates their degradation (Saad *et al.*,1982). Ethoprophos and diazinon were found at highest levels of OP insecticides detected in drain water. Diazinon was also found at the highest level in San Joaquin river in California (Domagalski *et al.*,1997), and in surface water of Imathia in North Greece (Albanis *et al.*,1998) and in Qarun lake in Egypt (Mansour *et al.*,2001). As regard to drinking water, canal and tap water , analysis of the collected water samples revealed the absence of any detectable residues of OP insecticides.

Table 5 Organochlorine insecticides residue levels (ug/L) in water samples of El-Mahmoudia canal and tap water obtained from Damanhour, El-Behira area Governorate.

OCs	Canal water		Tap water	
	Winter samples	Summer samples	Winter samples	Summer samples
Gamma- HCH	0.09	0.09	ND	ND
Beta – HCH	0.08	0.09	0.1	0.15
Heptachlorepoxyde	0.13*	0.15*	0.15*	0.16*
P,P' - DDE	3.1*	3.2*	1.2*	1.3*
Dieldrin	0.01	0.012	0.011	0.012
Endrin aldehyde	0.12	0.14	0.11	0.11
Endosulfan sulfate	0.01	0.01	0.01	0.11
Endrin Ketone	0.11	0.13	0.13	0.15

*= Values are exceeding than the permissible limits.

The overall results of the study revealed the presence of organochlorine residues in both drain and drinking waters and the OPs residues in drain water only. The concentrations of these OC compounds were more higher in drain water than in drinking water and tap water. This could be attributed to the drainage canal receives industrial and productive waste waters. Thus, the agricultural pesticide runoff surface water supplies must be controlled.

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الملخص العربي

رصد متبقيات المبيدات الكلورينية العضوية والمبيدات الفوسفورية في ماء الصرف ، ماء الترغ وماء الصنبور لمدينة دمنهور – محافظة البحيرة – مصر

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أجريت هذه الدراسة لرصد متبقيات المبيدات الكلورينية العضوية والفوسفورية في ماء مصرف الجبيري ، ماء ترعة الخندق وماء الصنبور بمدينة دمنهور وأوضحت نتائج كفاءة الاسترجاع للمركبات المختبرة أكثر من 85.5% عند مستوى تقوية 5 نانوجرام/مل . كان الحد الأدنى لاكتشاف المركبات الكلورينية العضوية والمركبات الفوسفورية العضوية يتراوح ما بين 10 إلى 30 نانوجرام / لتر. وأثبتت النتائج أيضاً وجود متبقيات ثمانية مبيدات هيدروكربونية كلورينية في مياه الصرف ، مياه ترعة الخندق وماء الصنبور وكانت متبقيات مبيدات الهبتاكلور إيبوكسيد ، بارا – بارا DDE ، الديلدرين ، البارا – بارا DDD أعلى في ماء الصرف عن الحدود المسموح بها دولياً . كما وأن متبقيات المبيدات الفوسفورية وجدت فقط في مياه الصرف. وكانت

متبقبات المبيدات في المياه المختبرة أعلى في مياه الصيف عن مياه الشتاء . عامة أوضحت النتائج أن مياه الصرف كانت أعلى تلوثاً وتحتوي متبقبات المبيدات بتركيزات أعلى عن مياه الترعة والصنبور وهذا يعني وجوب التحكم في صرف متبقبات المبيدات الزراعية في مصادر المياه.

