43

Vol. 40, No. 3, Autumn 2014

# Determination of Organochlorine Pesticides in River Waters by GC-ECD after Solid Phase Extraction, Mazandaran

# Shiva Dehghan Abkenar<sup>1\*</sup>, Morteza Hosseini<sup>2</sup>, Hosein Esmaili Gholzoom<sup>3</sup>, Ghorban Ali Khoda Bakhshi<sup>4</sup>, Naser Mehrdadi<sup>5</sup>

- 1. Assistant Professor, Department of Chemistry, Islamic Azad University, Savadkooh Branch, Mazandaran, Iran
- 2. Assistant Professor, Department of Life Science Engineering, Faculty of New Sciences and Technologies, University of Tehran, Tehran, Iran (hosseini\_m@ut.ac.ir)
- 3. Ph.D Candidate, Islamic Azad University, Savadkooh Branch, Mazandaran, Iran (Amir1352hs@yahoo.com)
- 4. Expert on Surface Water, Mazandaran Regional Water Company, Mazandaran, Iran (Khodabakhshi84@yahoo.com)
- 5. Associate Professor, Faculty of Environment, University of Tehran, Tehran, Iran (mehrdadi@ut.ac.ir)

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## **Expanded Abstract**

# Introduction

Several hundred pesticides of different chemical compositions are currently used for agricultural and vector control purposes all over the world. Because of their extensive use, they are detected in various environmental matrices, such as soil, water, and air. Due to their lipophilic nature, hydrophobicity, and low chemical and biological degradation rates, organochlorine pesticides (OCPs) have led to their accumulation in the biological tissues and subsequent magnification of concentrations in the organisms due to the progress up the food chain.

The organochlorine pesticides group includes DDT (dichlorodiphenyl trichloroethane), methoxychlor, aldrin, dieldrin, chlordane, toxaphene, endrin, heptachlor, and lindane (gamma isomer of benzene hexachloride (BHC)). These are trade names for closely related hydrocarbon compounds to which several chlorine atoms have been joined.

Residues of OCPs were detected in almost all environmental compartments, including water bodies, food, fish, and milk as well as in human beings.

#### Materials and Methods

In this study, the residue levels of 20 organochlorine pesticides (OCPs) were found in the river water samples obtained from the different regions of Mazandaran province, Iran. Total water samples, from 6 sampling sites, were collected every two months between 2010 and 2011.

For this study, a total of 56 water samples from 6 sampling sites were collected. All the water samples were collected in high purity glass bottles and immediately transported to the laboratory. After this, the samples were stored at  $+4^{\circ}$ C and extraction of the OCPs was performed within 48 h.

Some instruments were used for this experiment. 5.0 mL LiChrolut® EN cartridges were purchased from Merk, Germany. The vacuum assembly was homemade and the vacuum was generated by the homemade vacuum pump. The analysis of the reported OCPs after extraction was carried out by gas chromatography. Gas chromatograph used was of Agilent GC 6890 N with an electron capture detector (GC-ECD). The column used was HP-5 (30 m x 0.32 mm, IP 0.25  $\mu$ m) and obtained from Sigma Chemical Co., USA.

Solid Phase Extraction (SPE) methodology was developed by spiking of 1.0 mL of organochlorine pesticides mixture of 1.0 mg/mL concentration each (in methanol-water) in 499.0 mL tap water. This mixture was shaken for about 30 minutes. The spiked water sample was kept at room temperature overnight. C18 Cartridge was pre-conditioned by using methanol (10.0 mL) followed by water (10.0 mL). After equilibrium, 0.5 L of the spiked water was passed through this cartridge at 10.0 mL/min flow rates. The elution of OCPs was carried out by using ethyleacetate at different flow rates 1.0 mL/min. This methodology was applied to the natural conditions by replacing spiked water by Telar and Tajan rivers water and the results were compared.

\* Corresponding Author: Tel: +98 9111174767

## Determination of Organochlorine ...

Shiva Dehghan Abkenar, et al.

# **Results and Discussion**

44

The separations of these pesticides in a water sample are shown in Fig. 1.

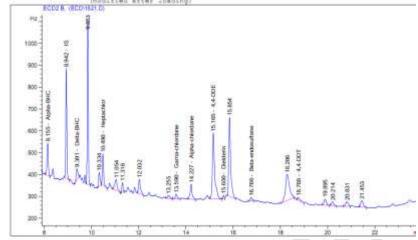


Fig. 1. GC Chromatograms of organochlorine pesticides in a water sample

The developed SPE and GC methodologies were applied for the analysis of organochlorine pesticides in the Telar and Tajan rivers. For this purpose, river water samples were collected from the Telar and Tajan rivers at 6 different sites. The values of concentration of the organochlorine pesticides observed are given in Table 1.

pesticides Amount (ng/ml)							
Station number	1	2	3	4	5	6	maximum residue limit established by FAO/WHO
α-BHC β-BHC γ-BHC δ-BHC	0. 180	0.195	0.160	0.103	0.099	0.108	$\sum_{BHC} 4 \text{ ng/ml}$
Heptachlor heptachlor epoxide	0.038	0.036	0.034	0.031	0.038	0.041	$\sum_{hep} 10 \text{ ng/ml}$
aldrin dieldrin	0.012	0.014	0.008	0.010	0.010	0.009	$\sum_{di+al} 30 \text{ ng/ml}$
$\gamma$ -chlordane $\alpha$ -chlordane	0.008	0.011	0.018	0.006	0.007	0.025	$\sum_{\gamma + \alpha} 30 \text{ ng/ml}$
endosulfan I, II , sulfate	0.022	0.040	0.027	0.021	0.037	0.018	$\sum_{en\Pi+I} 30 ng/ml$
4,4'-DDE 4,4'-DDD 4,4'-DDT	0.060	0.056	0.055	0.082	0.146	0.034	$\sum_{DD} 100 \text{ ng/ml}$
endrin endrin ketone endrin aldehyde	0.060	0.034	0.038	0.065	0.064	0.076	$\sum_{en} 20 \text{ ng/ml}$
methoxychlor	0.003	0.005	0.007	0.003	0.015	0.002	40 ng/ml

Table 1. Concentration of organochlorine pesticides in the Telar and Tajan rivers

# Conclusions

The developed SPE and GC methods were used for the separation, identification, and quantification of organochlorine pesticides in the water of Telar and Tajan rivers. The reported values of pesticides in the Telar and Tajan rivers water indicates that the rivers aren't polluted. Besides, these methodologies are rapid, selective

#### Journal of Environmental Studies

45

Vol. 40, No. 3, Autumn 2014

and reproducible. The percentage extractions of organochlorine pesticides are quite good. Therefore, these methods can be used for the analysis of organochlorine pesticides in waste, surface, ground and mineral water samples.

Keywords: chromatography, organochlorine pesticide, pesticide, solid phase extraction.

