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## REMOVAL OF DIMETHOATE CONTAMINATION FROM WATER: EXTRACTION CONDITIONS

M. I. Awaad\*, M. T. Haridi \* and A. Elbaz Elsaiid \*.

## ABSTRACT

Dimethoate (o,o-dimethyl-S-methyl-carbamoyl-methyl phosphorodithioate) is an organophosphorous insecticide used for the control of houseflies, as well as a wide range of insects and mites on a variety of fruit, vegetable, field and forestry crops. Water contamination by dimethoate is problematic in factories, stores and end user sites. Seven of the most common non-polar solvents have been tested as water decontaminating agents. Stock solution containing 152  $\mu$ g/ml of dimethoate in deionized water was prepared. 50 ml samples were mixed with known volumes of the selected extracting agent. The mixture was then stirred for a prescribed time interval at a fixed temperature. The phases were then separated and analyzed using a high performance liquid chromatograph. The extraction efficiency was calculated, and effect of each of the operating parameters on its value was determined. 1,2 dichloroethane was found to be the most efficient extracting agent. The favorable mode of extraction and operating conditions are presented.

#### **KEY WORDS**

Water pollution, Dimethoate, and Solvent extraction.

## INTRODUCTION

Water pollution is the contamination of fresh or saline water with materials, which are toxic, or otherwise harmful to human beings. The liquid wastewater is essentially the water supply of the community after it has been fouled by a variety of uses. If this wastewater is allowed to accumulate, decomposition of the organic contaminants may lead to production of large quantities of the malodorous gases [1-2].

The large-scale application of pesticides and insecticides in agriculture areas can contribute to the presence of these toxic materials in the surface ground water and ultimately in water supplies. Contamination can also occur through draining from surrounding terrain, precipitation from atmosphere or accident spills of pesticides in the watershed area [3].

Dimethoate is an organophosphorous insecticide used for the control of houseflies, as well as a wide range of insects and mites on a variety of fruit, vegetable, field and forestry crops.[4].

Dimethoate main biochemical action is on the nerve system. It acts as cholinesterase inhibitor, as are the most organophosphorous compounds [5].

A number of techniques are available for removal and/or recovery of the organophosphorous insecticides from wastewater. Solvent extraction is one of these techniques, which based on the relative solubility of an analyte in two immiscible liquids. [6-7].

#### EXPERIMENTAL

#### Chemicals

The used chemicals are all of the HPLC grade as given in Table 1. Deionised water was used for preparation of the standard solutions and the eluenting agents. Table 2 provides information about structures, grades, physical properties, and the suppliers of the employed dimethoate.

#### Instrumentation and Operating Conditions

Organophosphorous pesticides show normally typical absorption bands in the UV-Vis region, hence UV detectors are suitable for the performed chromatographic analysis.

Absorbance of dimethoate in UV-Vis region was measured by a Shimadzue UV-Vis double beam spectrophotometer model 1700.

An Agilent 1100 series HPLC was employed for analyzing the contaminated samples before and after extraction

The following operating conditions were adopted:

- Stationary phase: 150 mm \* 10 mm Id reversed phase column Zorbax SB C-18.
- Mobile Phase: Acetonitrile (30%) Water (70%).
- Flow rate: 0.3 ml / min.
- Injection volume: 3 µl.

- Detector: UV Detector at 300 nm.
- Post run: 30 minute.
- Column temperature: 37 °C
- External standard method (EST) was employed

The stock solution of dimethoate was prepared by dissolving 152 mg of dimethoate in deionized water and completed to 1000 ml.

## Extraction of Dimethoate

Batchwise extraction was employed for removal of dimethoate from the artificially contaminated water samples; 50 ml of the polluted sample was placed in a 250 ml Erlenmeyer flask. 6 ml of the selected solvent were then added, the extraction time was selected from 1 to 20 minutes depending upon the temperature during extraction. Magnetic stirrer was employed to accelerate the extraction process. Separation of the phases was done by a bench top centrifuge.

Effects of the solvent type, solvent quantity, extraction time and temperature were investigated.

#### **RESULTS AND DISCUSSION**

The maximum absorbance of dimethoate in UV-visible region according to their structure was 300 nm.

#### Effect of the Solvent Type

Fig. 1, shows the percent of dimethoate removed by different organic solvents at the following extraction conditions:

- Stirring time 6 minutes
- Amount of solvent 6 ml
- Temperature 25 °C

Its clear from the figure that, 1,2 dichloroethane is the most suitable solvent for extraction of dimethoate from water while mixed ether (petroleum ether, diethyl ether) have the lowest extraction efficiency.

#### Effect of the Solvent Quantity

Fig. 2, shows the relationship between the solvent volume and the residual concentration of dimethoate in  $\mu$ g/ml. As the amount of 1,2 dichloroethane increases, the residual concentration of dimethoate decreases and consequently the efficiency of extraction increases.

The minimum amount of 1,2 dichloroethane required for complete extraction of 7.6 mg of dimethoate from 50 ml contaminated water sample was found to be 6 ml.

#### Effect of Extraction Time

Fig. 3, shows the relationship between the extraction time in minutes and the residual concentration of dimethoate in  $\mu$ g/ml. As the stirring time increases, the residual concentration of dimethoate decreases and consequently the efficiency of extraction increases.

The minimum time required for complete extraction of dimethoate from contaminated water was found to be 6 minutes. These values express the rapidity of decontamination and consequently its economic feasibility.

## Effect of Temperature on the Extraction Process.

Fig. 4, shows the relationship between the solvent amount in ml and the residual concentration of dimethoate at different temperatures. As the temperature increases the amount of solvent required for extraction slightly decreases and consequently the efficiency of extraction increases. The normal temperature can therfore be adopted regarding the effeciency and economy of the carried out extraction process.

Fig.5 shows the relationship between the extraction time in minutes and the residual concentration of dimethoate at different temperatures. As the temperature increases, the time required for extraction slightly decreases consequently, the efficiency of extraction slightly increases ( in the studied range 18 - 35 ° C).

But once more the normal temperature can be adopted for rationalizing the energy consumption without losing much of the extraction efficiency.

## CONCLUSION

- 1,2 dichloroethane was found to be the most efficient solvent compared with the other selected ones.
- Six milliliter of 1,2 dichloroethane was found to be capable of decontaminating completely about 7.6 mg of dimethoate from 50 ml contaminated water sample .
- The extraction was rapid even at the normal temperature.
- The optimum time required for extraction of dimethoate using 1,2 dichloroethane was (6) minutes.
- The optimum temperature in the extraction of dimethoate was (35)  $^{\circ}$ C. ( in the studied range 18 35  $^{\circ}$  C).

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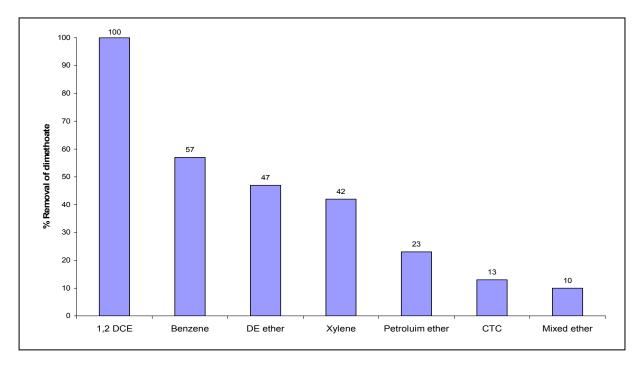


Fig.1. Percent of dimethoate removed by different organic solvents at the same extraction conditions

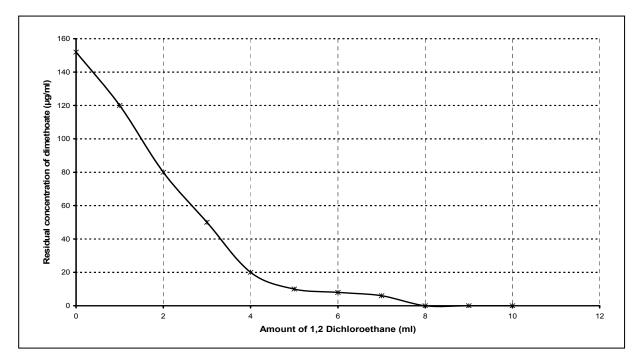


Fig.2 Variation of residual concentration of dimethoate with the 1,2 dichloroethane volume

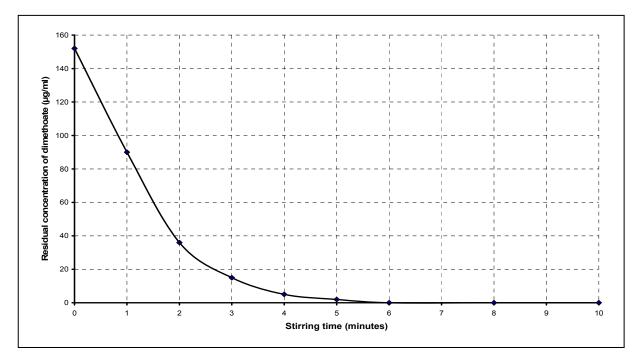


Fig.3. Variation of residual concentration of dimethoate with the stirring time

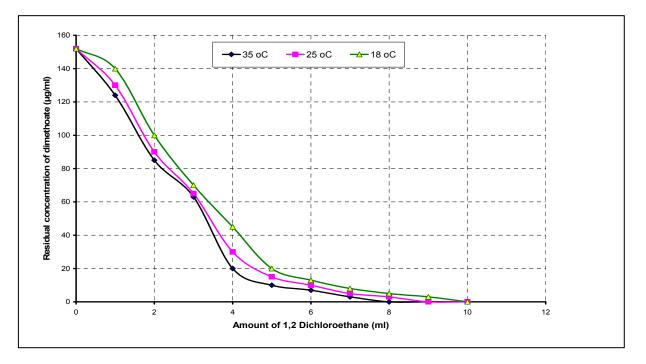


Fig.4. Variation of residual concentration of dimethoate with the 1,2 dichloroethane volume at different temperature

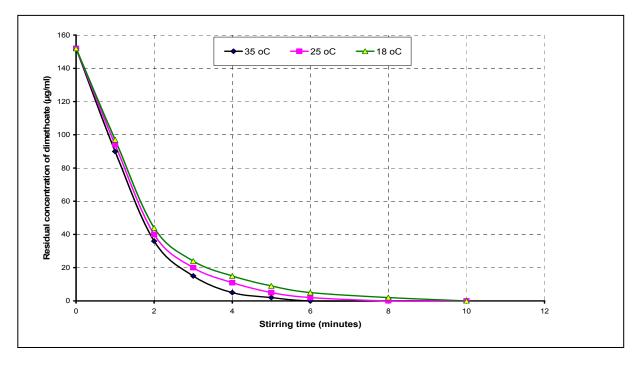


Fig.5. Variation of residual concentration of dimethoate with the stirring time at different temperature.

No.	Chemicals	Chemical formula	symbols	Grade	supplier
1	Deionized water	H <sub>2</sub> O	DI	HPLC	Fulka
2	Benzene	C <sub>6</sub> H <sub>6</sub>	В	HPLC	Fulka
3	Diethyl ether	$C_2H_5-O-C_2H_5$	DEE	HPLC	Aldrich
4	Carbon tetrachloride	C-CL <sub>4</sub>	СТС	HPLC	Aldrich
5	Acetonitrile	CH₃CN	AcN	HPLC	Aldrich
6	Toluene	$CH_3-C_6H_5$	Т	HPLC	Riede -Dettaen
7	Petroleum ether	Benzin, ether,	PE	HPLC	Honil
8	Xylene	CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub> -CH <sub>3</sub>	Х	HPLC	Aldrich
9	1,2 dichloroethane	CI-CH <sub>2</sub> -CH <sub>2</sub> CI	1,2 DCE	HPLC	Aldrich

Table (1) The Chemicals Used in Analytical Techniques

# Table (2) Properties of Dimethoate

Chemical formula	$C_5H_{12}NO_3PS_2$	Melting point °C	49
Solubility in water at 20 °C	23.8 gm/l	Boiling point °C	117
Solubility in organic solvents	very soluble	Flash point °C	> 163
Molecular weight	229.2	Specific Gravity at 25 °C	1.277
Vapour pressure at 30 °C	1.1 mPa	Grade	95 %
Octanol:Water partition coefficient	0.704	Source	NCIC