

Sensitive Spectrophotometric Methods For Determination Of Temefos In Water Sample

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ABSTRACT—In modernistic agriculture as appreciably extended productivity by using chemical substances but it also notably will increase the concentration of pesticides in our environment with negative effect on man or women. In this study we develop an analytical method for the analysis of temefos either in pure form or in dose form by means of Spectrophotometrically. It's a type an organophosphate pesticide used to treat disease carrying insects which includes mosquitoes. On this approach we studied pesticide is oxidized with recognized excess of Cerium (IV) sulphate in acidic medium and the un reacted oxidizing reagent are anticipated through including a regarded quantity of methyl orange in the same solution is detected by using UV- visible model LT-31 (labtronics) spectrophotometer. The consequent absorbance for at λ_{max} 511nm. Beer's law obey in 2-15 $\mu\text{g/ml}$ concentrations. The proposed work is applied for the analysis of examined pesticide in natural or dosage form with excellent precision.

Keywords— Pesticide residue, Cerium (IV) Sulphate, Methyl orange, Spectrophotometer, Water sample

concentration of a chemical increases at higher level in the food chain is known as bioconcentration and biomagnification.

The well-known Organophosphate group of pesticides are effective against a wide range of insects and they are not persistent. However, they much more absorbed through the skin, lungs, and gastrointestinal tracts. Human exposed excessive amounts have shown range of symptoms including tremor, confusion, slurred speech muscle twitching and convulsions. Organophosphates are basically organic esters of phosphoric acid, thiophosphoric acid. The most well-known organophosphate pesticide Temefos is a larvicide used to treat water infested with disease-carrying insects including mosquitoes, midges, and black fly larvae. They are anticholinesterases that disable the cholinesterase, an enzyme for the central nervous system.

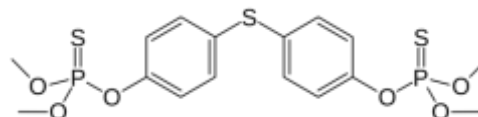


Fig.1 The chemical structure of Temefos.

I. INTRODUCTION

The term pesticide is used to cover a range of chemicals that kill organisms that humans consider undeniable and includes the more specific categories of insecticides herbicides, rodenticides and fungicides. There are three main groups of synthetic organic pesticides: organochlorine, organophosphate and carbamates. Increased concentration of artificial fertilizers, pesticides and salts in various environmental samples create many food problems. These chemicals on mixing with water used for irrigating the crops, also make food poisonous. The phenomenon in which the

These compounds are broadly used and display severe toxicity so several methods had been pronounced for its assay like LC-MS [5-6], titrimetry, voltammetry [7], GC [8], HPLC, atomic absorption spectrophotometry (AAS) [11] and spectrophotometry [4] have been stated inside the literature for the determination of organophosphates. Most spectrophotometric approaches involve the analysis of organophosphorus pesticides through total phosphorus measurement, based on the formation of molybdenum blue using various reducing agents.

A number of those techniques be afflicted by bad sensitivity, instability of color or contain extraction in which as others suffer from the interference with arsenic and copper, blank absorption or longer time required for color improvement. Conquer those drawbacks, a speedy and sensitive technique has been proposed for the analysis of the studied organophosphorus pesticide.

The goal of this work is to increase facile, fast and low price, correct, sensitive and proven spectrophotometric approach for the analysis of Temefos based on oxidation of the studied pesticide with moderate extra of Ceric (IV) Ammonium Sulphate in acidic medium and the unreacted oxidizing reagent are expected by adding a known amount of methyl orange within the same sample is detected by using UV- visible version LT-31 (labtronics) spectrophotometer. The proposed method is applied for the estimation of tested pesticide in natural or dosage form with desirable precision.

II. EXPERIMENTAL

Apparatus

All the absorbance spectral measurements have been made using UV- Visible model LT-31 (labtronics) spectrophotometer furnished with matched 1-cm quartz cells and temperature controller become used for all spectrophotometric measurements.

Materials and Reagents

All the reagents and chemicals have been exceptional availability fine and Analytical grade. Spectroscopic grade solvents had been used for evaluation and all solutions have been prepared via double distilled water in the course of the experiment.

Reagents:

Cerium (IV) Sulphate (250 $\mu\text{g ml}^{-1}$)

A stock solution of cerium (IV) sulphate turned into organized via dissolving 0.5 g of cerium (IV) sulphate in 1.0 M Sulphuric acid and transfer into 50-ml volumetric flask. The volume became finished to the mark with the same acid. Stock solution changed into diluted as it should be with 1 M Sulphuric acid to yield 250 $\mu\text{g ml}^{-1}$ Cerium (IV) Sulphate solution.

Methyl Orange (500 $\mu\text{g ml}^{-1}$)

The solution of methyl orange is prepared by dissolving 50 mg of methyl orange in water and transfer right into a 100-ml volumetric flask. The quantity became finished to the mark with water.

Sulphuric acid (5 M)

By adding 274 ml of concentrated Sulphuric acid to 726 ml water with cooling solution of Sulphuric acid was prepared.

Preparation of stock and sample solutions

Stock Standard Solutions:

By weighing accurately 10 mg of pure pesticide sample and transferring separately into volumetric flask of 10 ml, with the addition of methanol to up to volume and stock solution of temefos (1 mg ml^{-1}) was prepared.

Temefos Sample Solution:

A stock solution with concentration of 1 mg ml^{-1} was prepared by adding 0.2 ml of temo larvae into 100 ml volumetric flask, completed to volume with methanol.

Recommended procedure:

In 10 ml volumetric flask to give final concentration of 2-15 $\mu\text{g ml}^{-1}$, respectively, aliquots of temefos stock solution were added. Then 1 ml of Cerium (IV) Sulphate solution (250 $\mu\text{g ml}^{-1}$) and 1 ml of Sulphuric acid (5 M) were added to the flask. Flask were allowed to stand at room temperature for 10 minutes with occasional shaking after proper mixing. 0.5 ml of methyl orange solution (500 $\mu\text{g ml}^{-1}$) was added finally. After that solution was diluted to the up to the mark by water and mixed. After 5 minutes, UV experiment was executed and the absorbance of the sample was tested and measured at 511 nm in opposition to a reagent blank prepared within the identical way the usage of 1 ml water in place of 1 ml methyl orange solution.

III. RESULTS AND DISCUSSION

It is an indirect spectrophotometric method which is based on the ability of cerium (IV) sulfate to oxidize the studied pesticide and to bleach the color of the indicator methyl orange. The studied pesticide is allowed to react with a known excess amount of Cerium (IV) Sulfate in acidic media. The unconsumed Cerium (IV) Sulphate found in excess over the pesticide is quantified by adding methyl orange and monitoring the absorbance of the solution at $\lambda_{\text{max}} = 511 \text{ nm}$. The absorbance of indicator solution decreases due to the presence of oxidant. Assuring that the oxidation reaction is complete, the unreacted Cerium (IV) amount is proportional to the drug amount in the sample.

According to Scheme 1 increasing concentration of drug in the sample, the amount of unreacted Ce (IV) bleaching the dye is decreases

and thus the absorbance of the sample solution is measured at $\lambda_{max}=511$ increases linearly with the pesticide concentration (Fig. 1,2).

Scheme 1: Reaction scheme of the indirect determination of OPP by oxidation with cerium (IV) sulfate.

- (i) Organophosphate + Ce (IV) \rightarrow Organophosphate oxidation product + Ce (III) + Ce (IV) unreacted
- (ii) Ce (IV) un reacted +MO \rightarrow Oxidation Product of MO + Unreacted MO
- (iii) * Measured spectrophotometrically at $\lambda_{max}=511$ nm

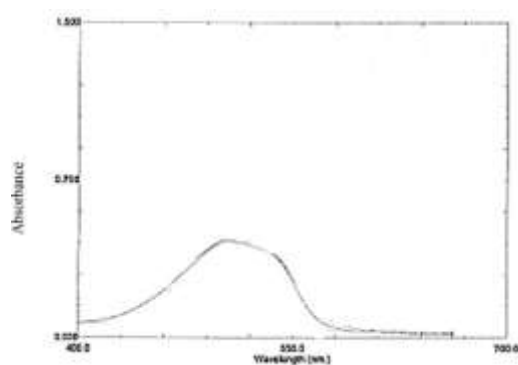


Fig. 2 Absorption spectra of solutions containing temefos 0.5M H₂SO₄ and 10 µg ml⁻¹ temefos

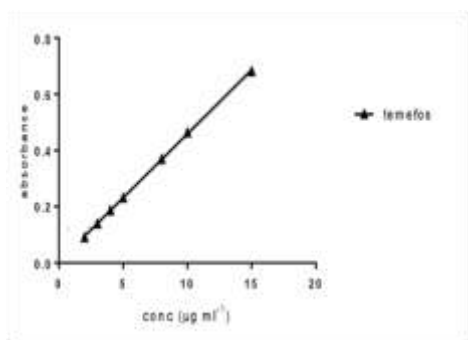


Fig. 3 Calibration curve of temefos

Optimization of the reaction conditions:

The optimum conditions for the assay procedure and color development of each method have been established by varying parameters one at a time, keeping the other fixed and observing the effect produced on the absorbance of the colored species.

Effect of the reagents

By the regardless addition of organophosphate amount, methyl orange is almost bleached if the order of addition reagents was dye+ drug+ oxidant or dye+ oxidant +drug. This remark is going on because the cerium (IV) sulphate did now not have enough time to oxidize the drug

rapidly .It easily bleaches the methyl orange. After the observation it is concluded that drug and oxidant solutions respectively must be added first ,their order of addition does not affect the reaction and methyl orange have to be added after a given period of time when the drug is completely oxidized by Ce(SO₄)₂.

Effect of time on the oxidation reaction of Organophosphate by Ce(SO₄)₂:

When methyl orange is added to the solution containing OPP and Cerium (IV) Sulphate in acidic medium then immediately solution get bleached and low absorbance observed. From the observed absorbance it can be explained that the oxidation of drug by Cerium (IV) Sulphate is a time developing reaction .Thus the effect of reaction time was studied. Then Temefos is allowed to react with 25 µg ml⁻¹ Cerium (IV) Sulphate at darkness different times before adding the indicator and measuring the absorbance. It was observed that the absorbance of this solutions increases with the time up to 10 minutes remaining then constant (Fig. 5). Thus, for further measurements a reaction time of 10 minutes was selected.

Effect of Sulphuric acid concentrations:

Sulphuric acid is for the acidic medium and the reaction was carried out in acidic medium. At 5M concentration of H₂SO₄ , highest absorption intensity was obtained for temepfos (Fig .6) .In the analysis 1 ml sulphuric acid was used.

Effect of Cerium (IV) Sulphate:

The absorbance of solutions containing a fixed concentration of MO (25 µg ml⁻¹) and different concentrations of cerium (IV) sulfate comprised in the range 2.5-25 µg ml⁻¹Ce(IV) in 5 M H₂SO₄ have been measured and it was observed that a concentration of Ce(IV) higher than 25 µg ml⁻¹Ce(SO₄)₂ bleaches completely the solution containing 25 µg ml⁻¹(Fig. 4).

IV. CONCLUSION

The present study described the successful development of new, simple, sensitive, selective, accurate and rapid spectrophotometric method for the accurate determination of temefos; forms using cerium (IV) sulphate as the oxidimetric reagent. All the analytical reagents are available in laboratory and they are inexpensive with having excellent shelf life. Therefore, the method is practical and valuable for routine analysis in quality control laboratories for analysis of investigated pesticide.

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