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Extraction of monocrotophos from stream water using solid phase extraction method

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Abstract
This research is a continuation of a study on the extraction of organophosphorus insecticides from water using solid phase extraction column loaded with granulated activated carbon. In this study, a wavelength of 220nm was used to monitor its disappearance from water. Prior to extractions processes, the half-life of 0.032, 0.30 and 0.031 hours of monocrotophos was determined in pH6,7 and 8 respectively. The target analyte was extracted from well characterized stream water collected from the middle belt region of Nigeria, distilled water and deionized distilled water using a packed SPE column and eluted with n-hexane. The eluant was analysed using uv-visible spectrophotometer and the percentage recovery of spiked samples were found out to be 68.69%, 85.68% and 80.73% respectively. The percentage recovery from SPE system were compared with recoveries from liquid –liquid extraction method with a percentage recovery from stream water, distilled water and deionized distilled water were 74.50%, 97.54% and 93.65% respectively. Subsequently, the second order rate constant for the extraction of monocrotophos in pH 6,7 and 8 were calculated to have values of $6.0x10^{-3}$, $6.42x10^{-2}$ and $6.4x10^{-3}$ $m^{-1}s^{-1}$ respectively. The SPE system has a great potential for the removal of organophosphorus insecticides from water.

Keywords: Organophosphorus insecticides, activated charcoal, Water, Removal

1. Introduction
When many organochlorine pesticides became banned in 1970's due to their high persistence in the environment, the agrochemical industries turned to short-term environmental persistence organophosphorus pesticides (OPPs) which have high toxicity to humans and mammals. OPPs because of their high efficiency and broad spectrum, they have higher degradation rates, widely used for protecting crops against pests, thereby increasing the productivity of the harvest. Despite being susceptible to relatively rapid degradation, organophosphorus insecticides have been found in varying concentrations in ground and surface water including that used for drinking. However, the massive use of pesticides has already contributed to the current levels of environmental pollution especially in water systems [1]. Therefore, the development of accurate and sensitive analytical methods for the simultaneous determination of trace levels of OPPs that facilitate the assessment of risk is in increasing demand [2]. When organophosphorus insecticides are applied to the environment, they tend to move through the soil, water and air, sometimes over long distances through photo-oxidation, plant uptake, volatilization, runoff, leaching, chemical decomposition, sorption, biological degradation while others persist [3, 4]. The solubility of organophosphorus insecticide is important to its fate and mobility primarily because highly soluble chemicals tend to be rapidly distributed within soil and the hydrosphere [5]. In general, the solubility of pesticides in water is a function of temperature, pH, ionic strength (concentration of soluble salts) and the presence of other organics. It is generally conceded that organophosphorus insecticide residues in surface water supplies, under normal conditions, cannot be present in toxic range capable of producing violent illness or death [1]. Rather, some must have viewed in terms of hazardous materials because of their possible subtle, long-term effects [6]. Among the chemicals identified have been some with effect upon the central nervous system and others with carcinogenic or tumor causing properties. Also, the possibility exists with teratogenic (disfiguring) or mutation characteristic may be present. Nevertheless, we should bend every effort to determine their presence in water [7]. It is as a result of these that there have been many approaches to extract pesticides from water such as Liquid-Liquid Extraction (LLE), Solid-Phase Extraction (SPE), Solid Phase Micro-Extraction (SPME), Supercritical Fluid Extraction (SFE) [8, 9].
SPE is a form of Liquid Chromatography which is a separation process that is used to remove organic compounds from a mixture of impurities based on their physical and chemical properties. Analytical laboratories use SPE to concentrate and purify samples for analysis. It can also be used to isolate analytes of interest from a wide variety of matrices, including urine, blood, water samples, beverages, soil, animal tissue, environmental and consumer products. The separation ability of a SPE

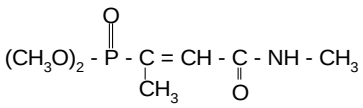
system is based on the preferential affinity of desired or undesired solutes in a liquid mobile phase to be partitioned on a solid stationary phase through which the sample is passed. Impurities in the sample are washed away while the analyte of interest is retained on the stationary phase or vice-versa. Analytes that are retained on the stationary phase can then be eluted from the SPE adsorbent with the appropriate solvent [8]. The present research continues a series of studies on the extraction of organophosphorus insecticides from water using granulated activated carbon as an adsorbent and finally their quantification by uv-spectrophotometry. The aim of the study was to carry out a laboratory study on the efficacy of using SPE column loaded with granulated activated charcoal to extract monocrotophos as one of the model organophosphorus insecticides from water.

2. Materials and Methods

The materials used in this study include reagents, apparatus, equipments and instruments.

2.1 Reagents

The reagents used in this study were crystalline $CH_3COONa.3H_2O$, $Na_2HPO_4.12H_2O$, HCl, CH_3COOH), CH_3CH_2OH), CH_3OH, n-haxane, ammonia buffer solution, acetone, Eriochrome black T (EBT), H_2SO_4), EDTA. All solvents, including n-hexane were of HPLC grade and purchased from Sigma Chemical Company. All the chemicals used were analytical grade reagents (AnalaR) except commercial monocrototphos (O-2-diethylamino-6-methylpyrimidin-4-yl phosphorothioate) bought from Novatis crop protection AG Basel Switzerland (Pesticide Data Handbook, 1994). The structure of monocrotophos is shown below.



Molecular Formula: $C_7H_{14}NO_4P$. Molecular weight: 223

Properties. It is a colourless solid at room temperature and has a melting point of 54-55ºC in the physical state with a mild ester odour. The technical product is a reddish brown semi-solid. It is soluble in water, acetone, and ethanol, sparingly soluble in xylene but almost insoluble in diesel oil and kerosene. It is corrosive to black iron, drum steel, brass and does not attack glass and aluminum; it is incompatible with alkaline pesticides [10]. It is extremely toxic to birds and used as a bird's poison [11]. The EPA classifies monocrotophos as a class one toxicity (a highly toxic) compound; products containing monocrotophos bear the Label "Danger". Monocrotophos is metabolized and excreted rapidly and does not appear to accumulate within body. In mammals, 60-65% are excreted within 24 hours especially in urine. The pesticide was selected because it is widely used in Benue State to control pests on beans and is likely to pose residue problems in environmental media, including water. The pesticide was used directly as provided by the manufacturer without further purification.

3. Instruments

A Jenway 6305 UV-visible spectrophotometer (Jenway 600) was used to measure the absorbance of monocrotophos in both aqueous and buffer media. Laptop Computer (Zinox) was used for graph plotting and rate determinations. Digital weighing balance (Galaxy 400) for weighing samples. SPE column (23cm long with internal diameter of 1.73cm) was used for packing granulated activated charcoal. Separatory funnel was used as a reservoir for holding spiked samples. Retort Clamp and stand was used to clamp the SPE column and the separatory funnel. Hi 9024 microcomputer pH meter was used to measure the pH of water. Hamilton gas tight syringe (10mL and 0.1mL) used for measuring volumes. Brown vials (10mL) used for collecting the extracted analyte. Silica glass cuvettes used for holding spiked samples. Polypropylene frits used for supporting the granulated activated charcoal. Granulated activated charcoal was bought from Gunsdong Guandgea Chemical Co. Ltd, China. Oven Box was used for drying glass wares. All experiments were carried out at room temperature.

Preparations of buffer solutions

Preparation of sodium ethanoate (CH_3COONa): A solution of 0.1M sodium ethanoate was prepared by adding 13.6g of crystalline sodium ethanoate ($CH_3COONa.3H_2O$) to 200cm^3 of distilled water in a 1L volumetic flask to dissolve and finally adding distilled water to make up to the mark. Stoppered and inverted several time to homogenize the solution. Labeled and stored at room temperature.

Preparation of sodium hydrogen tetraoxophosphate (v) ($Na_2HPO_4.12H_2O$): A solution of 0.1M sodium hydrogen tetraoxophosphate (v) was prepared by dissolving 35.8g of crystalline $Na_2HPO_4.12H_2O$ in 200cm^3 of distilled water in 1L volumetric flask and finally adding distilled water to make up to the mark.

Preparation of hydrochloric acid (HCI): 0.1M HCI was prepared by diluting 8.60cm^3 of concentrated HCI in 700cm^3 of distilled water in 1L volumetric flask and finally adding distilled water to make up to mark, stoppered and homogenized. The solution was standardized with Sodium hydroxide (NaOH) using methyl orange indicator.

Preparation of ethanioc acid (CH_3COOH): 0.1M ethanoic acid was prepared by diluting 5.70cm^3 of concentrated ethanioc acid in 500cm^3 of distilled water in 1L volumetric flask and finally adding distilled water to make up to the mark, stopper and homogenized. Labeled and stored.

Preparation of pH 6, 7 and 8

Preparation of pH 6: CH_3COOH/CH_3COONa buffer solution, 52.20cm^3 of 0.1M CH_3COOH was added to 947.80cm^3 of 0.1M CH_3COONa in a 1L volumetric flask.

Preparation of pH 7: Na_2HPO_4/HCl buffer solution, 244.00cm^3 of 0.1M HCI was added to 956.00cm^3 Na_2HPO_4 in a 1L volumetric flask.

Preparation of pH 8: HCl/Na_2HPO_4 buffer solutions, 44.90cm^3 of 0.1MHCl were added to 955.10cm^3 Na_2HPO_4 in a 1L volumetric flask.

Preparation of stock solutions

The preparation of stock solution was done by methods described by Guenzi [2]. A standard solution of monocrotophos (2 x 10^{-4}M) stock was prepared in 20cm^3 ethanol by measuring 3cm^3 of stock solution with a 10cm^3 graduated Hamilton gas-tight syringe and transferred into a 1dm^3 volumetric flask containing 977cm^3 of distilled water This was homogenized, stopperred with a glass stopper and stored in a refrigerator to maintain its stability.

Spiking monocrotophos with distilled deionised distilled and stream water

A mixture of monocrotophos and distilled, deionised distilled and stream water respectively were prepared by transferring 100cm^3 of monocrotophos (2 x 10^{-4}M) solution into 1000cm^3 volumetric flask containing 900cm^3 of distilled water, homogenized and kept for use.

3.1 Sampling of stream water

Three water samples were used for this study namely; distilled water (DW), deionized distilled water (DDW) and stream water (SW). 10L each of DW and DDW was bought from Benue State Environmental Protection Agency Laboratory, Makurdi. The DW and DDW were maintained at room temperature during transportation and storage in the laboratory. The stream water (SW) was selected on the basis of it been a real water sample. The stream water was collected from one point in Agu stream in Daudu (latitude 7^o 44′ N and longitude 8^o 31′E) Lafia-Makurdi road in Guma Local Government Area of Benue State, Nigeria with a glass bottle, covered with screw-top Teflon to avoid contamination during transport and storage in the laboratory. Immediately after the arrival of the sample to the laboratory some physic-chemical parameters of the water samples were examined. Before the experiment, the stream water sample was filtered through 0.45 µm filter paper and stored in amber colored bottles at 4 °C in the refrigerator. Plastic containers were not used in this study because polyethene contains traces of plasticizers which may leach from the plastic to water or organics from water may adsorb onto the plastic.

Solid phase extraction system

The SPE system consists of a cleaned column of 23cm long with an internal diameter of 1.73cm. The granulated charcoal was activated at 50^{o}C in an oven for 24 hours and cooled in a desiccator before use. Then, the column was loaded with polyethylene frits below followed by loading 1.0g of granulated activated charcoal and finally loaded with polyethylene frits above. This was to prevent the activated charcoal from passing out of the column and acts as a support. This gave a bed height of 5.5cm to ensure good retention of the desired analyte. The column and the separatory funnel were clamped on the retort clamp and stand connected to a vacuum pump.

3.2 Methods

Determination of maximum wavelength.

The determination of maximum wavelength of monocrotophos was carried out by using the wavelength scanning method over 200-400nm in distilled water measured at room temperature to choose the maximum wavelength to monitor its disappearance. Approximately 2.50cm^3 of DW was transferred into a silica glass cuvette, R and was kept in the reference compartment of the UV-visible spectrophotometer also; 2.50cm^3 of monocrotophos (2x10^{-4}M) was transferred into another silica glass cuvette, P and was placed in a compartment in the UV-visible spectrophotometer. The absorbance was manually measured in the wavelength range mentioned above and the maximum wavelength was chosen from the highest peak on the spectra.

The degradation of monocrotophos in buffer solutions

The degradation of monocrotophos was determined in buffer solutions using the following procedure: Approximately 2.0cm^3 of buffer solution of pH 6 was first injected into the silica glass cuvette and placed in the compartment of the spectrophotometer to equilibrate the solution for 10 minutes. Then the reaction was initiated by the addition of 0.5cm^3 of monocotophos (2 x 10^{-4}M) using Hamilton gas-tight syringe. The final volume in the cuvette was 2.50cm^3 and the wavelength of 220nm was set on the UV-visible spectrophotometer. The absorbances were manually measured at five minutes interval that lasted 60 minutes and each absorbance measured in duplicates. The data was used to obtain pseudo-first order plot using B+ log ($A_t - A_i$) on the vertical y-axis and time on the horizontal x-axis using a computer, Rate constants (K_{obs}) = - 2.303 x slope, Rate constants (K_{obs}) V_s buffer solution was plotted to obtain second order rate constants and the Half-lives ($t_{1/2}$) was calculated using $t_{1/2}$ = ln2/k, where $t_{1/2}$ is the half- life of pesticide and K = rate constant obtained from the second order plot. The same procedure was applied for pH 7 and 8.

Quantitative Recovery Study

Determination of absorbance before extraction

Approximately 2.50cm^3 solution of monocrotophos spiked with distilled, deionised distilled and stream water were added separately to three silica glass cuvettes and kept in three compartments of Jenway 6305 UV-visible spectrophotometer and distilled water was used as a reference and their absorbance's were measured at a wavelength of 220 nm before extraction.

SPE procedure

The granulated activated charcoal in the column was conditioned by a method described by Jiping et al, 2009 [12] Approximately, 10cm^3 of methanol was added to the SPE column followed by 10cm^3 n-haxane and finally 10cm^3 of distilled water was added to promote wetting and to enhance uniform flow of analyte passing through it. Then, 100cm^3 of monocrotophos spiked with distilled water was quantitatively transferred into a separatory funnel, its tap opened and allowed to pass through the column at a flow rate of approximately 3cm^3/min. without allowing it to get dry with the aid of a vacuum pump. Separation efficiency was maximized by having a continuous partitioning of analyte on the granulated activated charcoal as the spiked sample pass through the column. After, all the solution in the reservoirs was emptied; it was washed with 5cm^3 of DW. 10cm^3 of n-hexane was introduced into the column and allowed to soak for 15 minutes to dissolve the analyte that have been adsorbed onto the sorbent before elution with 10cm^3 hexane and concentrated by a rotary evaporator. The absorbance of the concentrate was measured using the procedure earlier mentioned and the percentage recovery calculated. The same procedure was used for monocrotophos spiked with DDW and SW. Two extractions were carried out.

Procedure for LLE

100cm^3 of monocrotophos spiked with distilled water was quantitatively transferred into a 250cm^{3}separatory funnel that had been washed with detergent rinsed with distilled water and finally with acetone to remove all forms of contaminants. 10cm^3 of n-hexane was added and shaken intermittently and vented out after 5 minutes. The phases were allowed to separate out in 15minutes. The organic phase was extracted and concentrated by a rotary evaporator. The absorbance after extraction of the concentrate was measured at the above

wavelength earlier mentioned and the percentage recovery calculated. The same procedure was used for monocrotophos spiked with DDW and SW. Two extractions were carried out.

$$\text{Percentage recovery from water samples} = \frac{\text{Absorbance after extraction}}{\text{Absorbance before extraction}} \times 100$$

Results and Discussion

In this study, a wavelength scanning method over 200-400nm for monocrotophos in distilled water was measured at room temperature to choose the maximum wavelength to monitor the disappearance of the reactant. The chosen wavelength in this study was 220nm (figure 1) while the literature value was reported to be 218 nm [10]. The difference in the literature value may be as a result of additives or impurities since the commercial monocrotophos was not purified.

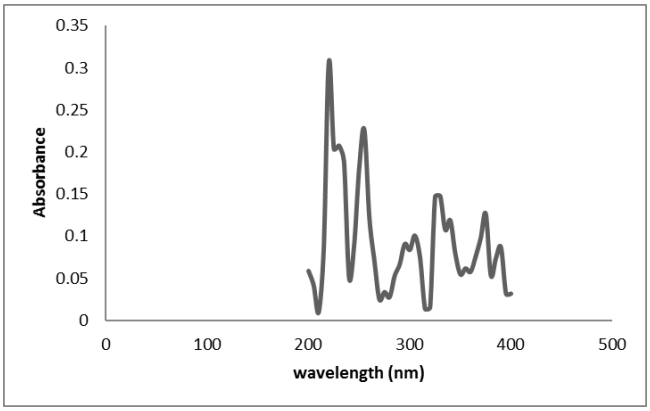


Fig 1: UV-visible spectra of monocrotophos interacting with distilled water from 200-400nm

The degradation of monocrotophos in buffer solutions

We also investigated the pH effect on the pesticide. Firstly, the degradation status of monocrotophos was studied in range of pH6-8. The degradation of monocrotophos in the environment including water [13] is one of the most important factors in evaluating their fate in water as well as their possible adverse effect [14]. Thus, the ability to measure or reliably estimate the possible degradation process or pathway is of critical importance to the environmental risk assessment of this chemical [15].

The kinetic studies

A kinetic study was carried out using UV-visible spectrophotometer prior to extraction, to determine the half life of monocrotophos in water. A graph of mean absorbance against time for the degradation of monocrotophos in the range of pH 6, 7 and 8 are shown on figure 2a-c. The data obtained lead to pseudo-first order plots on figure 3a-c. The second order rate constant K_{obs} was obtained by plotting the rate constant K_{obs} against buffer solution of pH 6-8 (figure 4). From the second order, the rate constant K_2 was obtained from the slope. The second order rate constants (K) for the degradation of monocrotophos in buffer solution of pH 6, 7 and 8 were calculated to be 0.0060, 0.0642 and 0.0064 $M^{-1}S^{-1}$ respectively. These values were used to calculate the half-live of monocrotophos in the buffer solutions pH 6, 7 and 8 using the formula $t_{1/2} = \ln 2/K$. Where $t_{1/2}$ is the half life of monocrotophos, K is the rate constant obtained from the second order plot as shown on figure 4. The half-life of monocrotophos in pH 6, 7 and 8 were 0.032, 0.30 and 0.031 hour respectively. The values of the half - live of monorotophos obtained in this study decreases as the solution become either more acidic or more basic, relative to the rate observed at neutral pH. This is in agreement with the studies carried out by Luther, *et al.* [13] that, the degradation of pesticides exhibit marked pH dependence. Eneji [16], Momoh et al. [17] carried out extensive study on the degradation of organophosphorus insecticides and they reported that, their degradation decreases in buffer solution as the solution gets closer to pH 7 and increases in mildly acidic and basic medium. The half - live in pH 7 has a higher value so the extraction was carried out in pH 7.

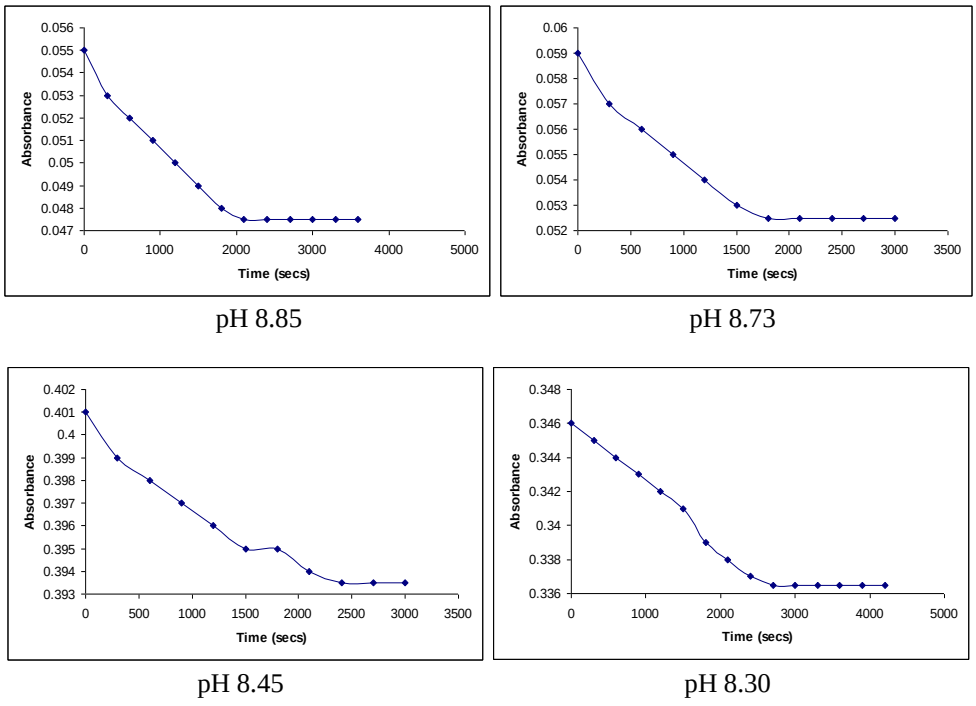


Fig 2a: A typical plot of absorbance versus time for the degradation of Monocrotophos ($2x10^{-4}M$) in pH 8

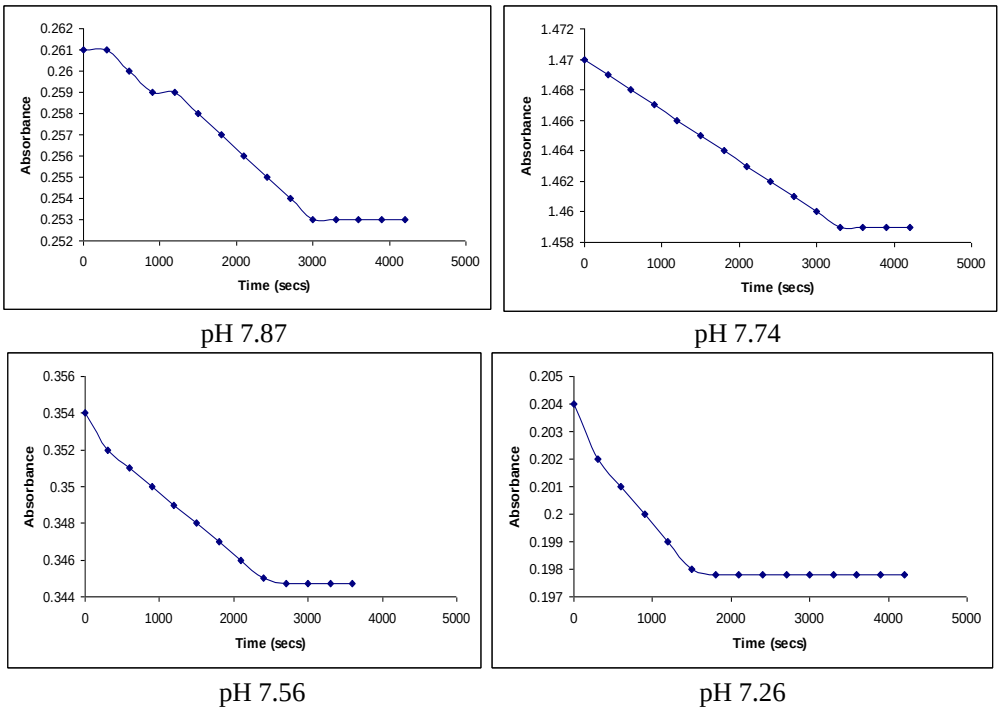


Fig 2b: A typical plot of absorbance versus time for the degradation of Monocrotophos ($2x10^{-4}$M) in pH 7

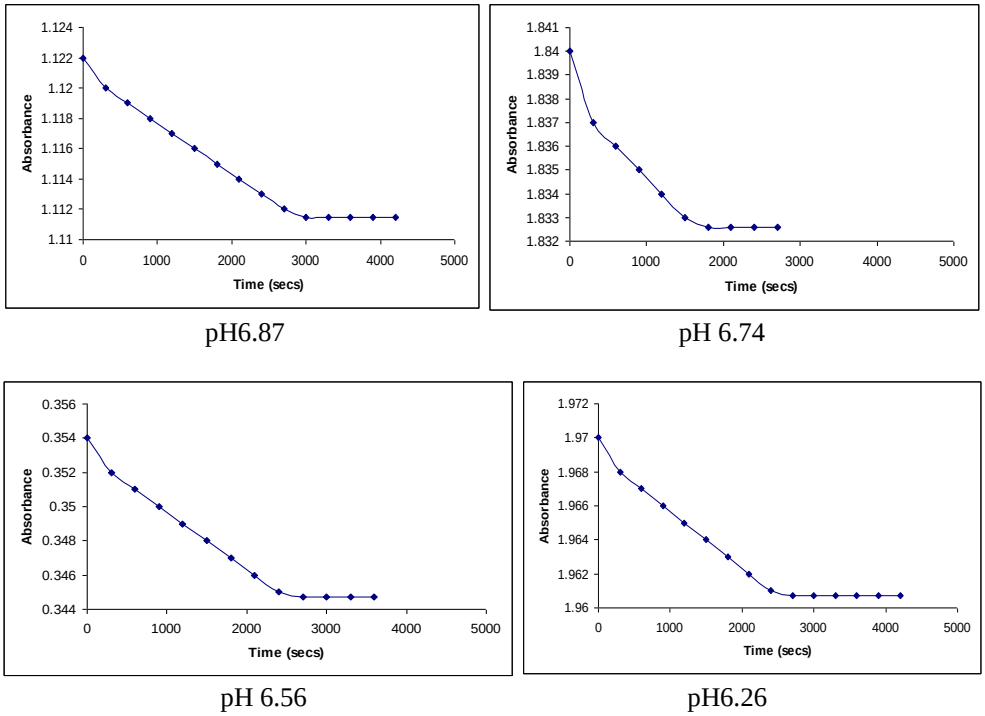
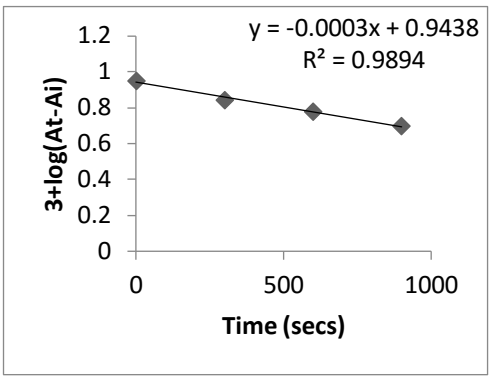
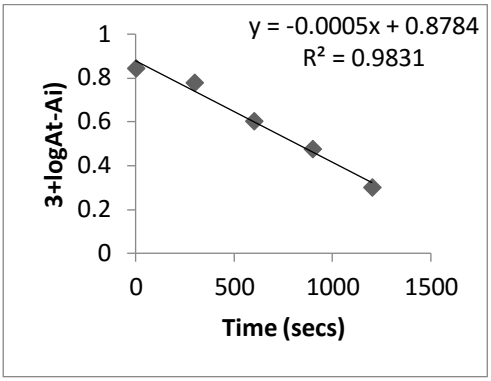


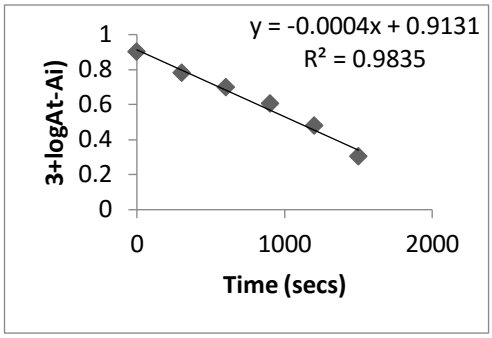
Fig 2c: A typical plot of absorbance versus time for the degradation of Monocrotophos ($2x10^{-4}$M) in pH 6



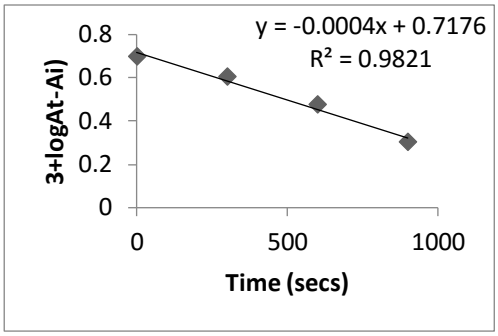
pH 8.85



pH 8.73

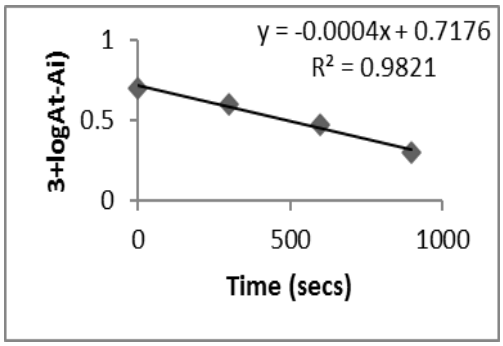


pH 8.45

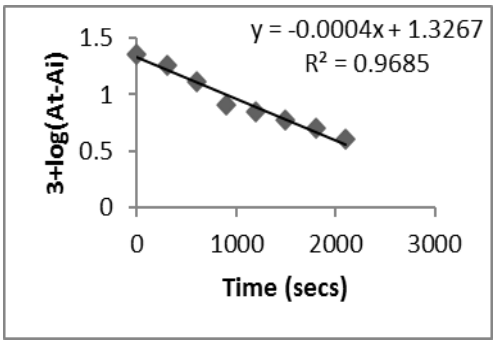


pH 8.30

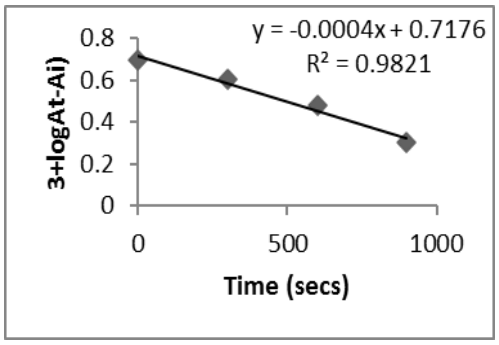
Fig 3a: Pseudo-first order plot for the degradation of Monocrotophos ($2x10^{-4}$M) in pH 8 at 220 nm



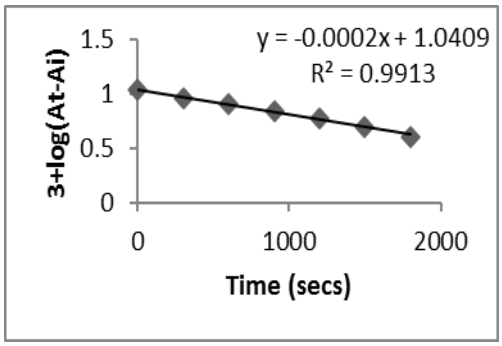
pH 7.87



pH 7.74

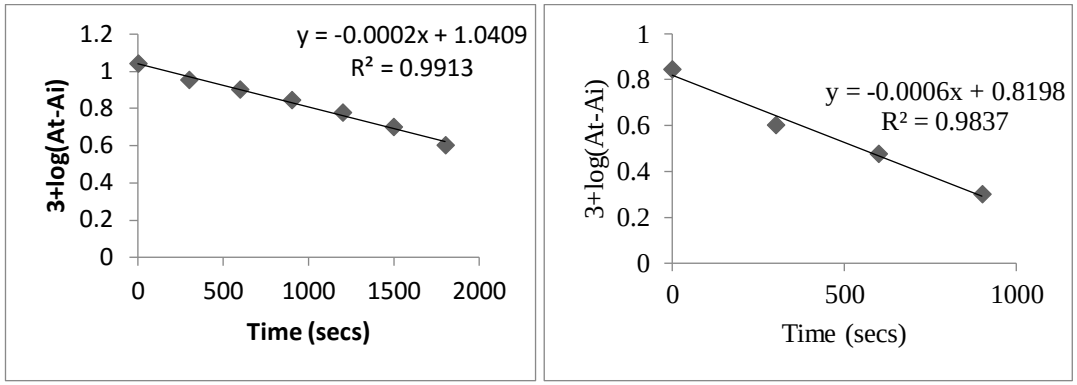


pH 7.56

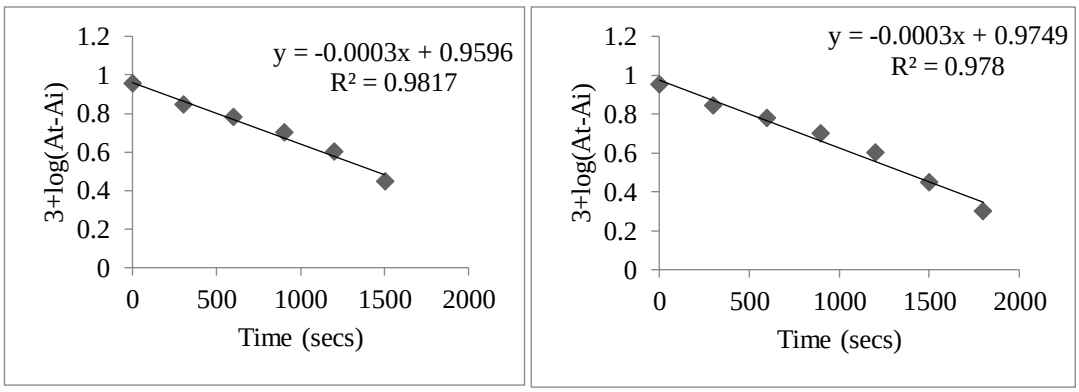


pH 7.87

Fig 3b: Pseudo-first order plot for the degradation of Monocrotophos ($2x10^{-4}$M) in pH 7 at 220 nm



pH 6.87 pH 6.74



pH 6.56 pH 6.25

Fig 3c: Pseudo-first order plot for the degradation of Monocrotophos ($2x10^{-4}$M) in pH 6 at 220 nm

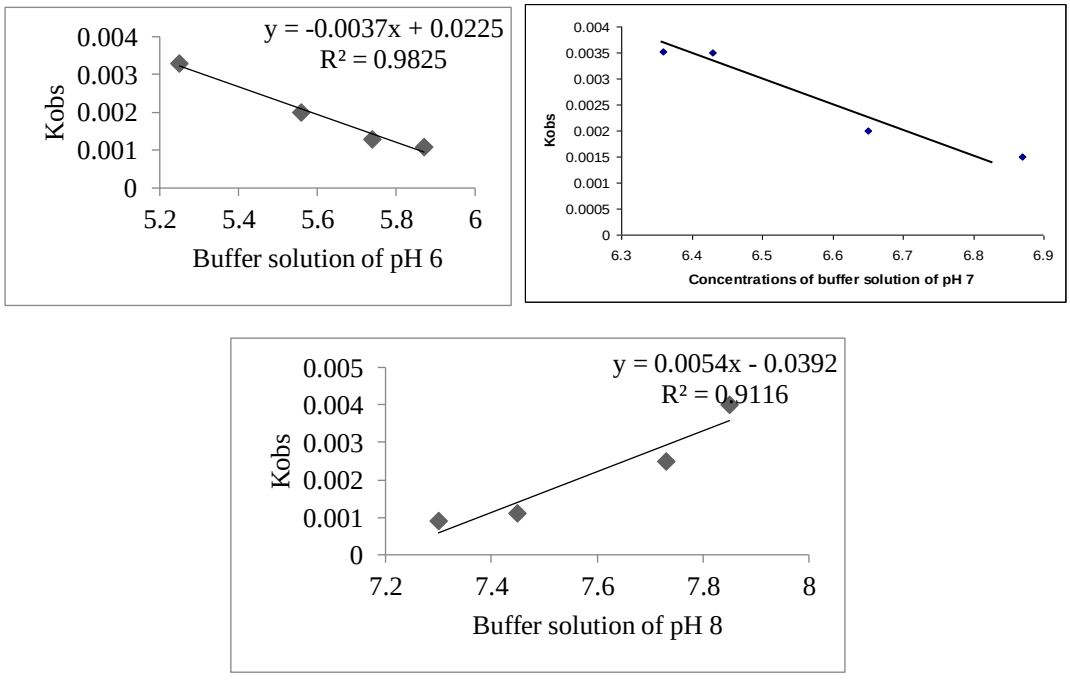


Fig 4: A Second-order plot for the degradation of Monocrotophos in pH 6,7 and 8

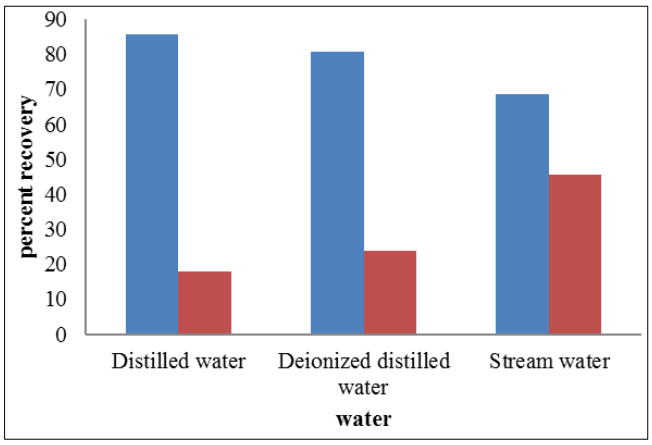


Fig 5: Mean percentage recovery and analyte loss of monocrotophos from water using SPE technique. Eluted with 10cm^3 n-Hexane. Results are the average of two determinations

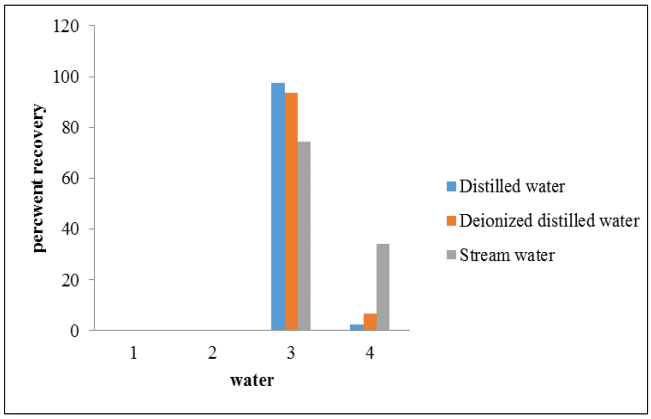


Fig 6: Mean percentage recovery and analyte loss of monocrotophos from water using LLE technique. Eluted with 10cm^3 n-Hexane. Results are the average of two determinations.

The mean percent recovery of monocrotophos from the DW, DDW and SW ranges from 68.69% to 85.68% for SPE and 74.50% - 97.54% for LLE technique as indicated on figure 5 and 6. Sapna et al. [18] reported the mean percent recovery of 98.79% for monocrotophos from soft drinks using LLE. The low percent recovery for monorotophos from this work as compared to the work of Sapna et al may be due to the use of different extraction solvent and the chemical interaction of other organic substances in the water with monocrotophos. We did not investigate the nature of organic substances in this study. However, Aranda and Kruus [19] reported the presence of fulvic and humic acids are unbiqoutous in stream water and they interact with organophosphorus insecticides reducing their recovery from 60%-46% during extractions. This may be the reason why the recovery of monocrotophos from DW and DDW were higher than the recoveries obtained from SW. This also indicates that monocrotophos have significant interactions with the fulvic/humic acids, clay and silt (matrices) in the stream water. This is in agreement with the study reported in Ohio University Bulletin [20] that, clay, silt hold water and dissolved chemicals longer because of their greater surface area to increase absorption and cannot be easily eluted.

The lower mean percent recovery from SPE systems as compared to LLE techniques may be due to the adsorption of analytes on to the granulated activated charcoal (GAC) and interaction of the pesticides with the mobile phase. The GAC may have trapped the pesticides and other matrices from water before elution such that even after elution some of the analytes were not eluted due to intra-pesticide diffusion into its interior of the GAC and was not able to be eluted. This agrees with the study reported by Edward *et al.* [15] where C_{18} and C_8 was used in SPE columns to extract seven agrochemicals in water with a percent recovery of 72-98%, that the agrochemicals were strongly adsorbed to be extracted. Sadek, et al. [21], confirmed that, absorption of phenoxylalkanoic acids on graphitized carbon as a sorbent is assumed to occur initially at the surface of the sorbent particles which may subsequent diffuse into the interior or become covalently bonded (intrapesticdes diffusion or chemosorption respectively) which eluting solvent may not be able to penetrate into the interior and dissolve the analyte and elute completely resulting in tailing. Oscik and co-workers [22] reported that interaction and molecular associations in the mobile phase can occur resulting in degradation of the analyte and were independent of the nature of the sorbent used. This may be the reason why there was low recovery from stream water. This also, is in agreement with the studies carried out by Sadek *et al.* [21] and Smith *et al*, [11] in which they reported that any strong interaction of the analyte with residual hydroxyl sites or complexation with trace metals present in the water can lead to tailing and irreproducitibility of recoveries. They also investigated the influence of trace metal retention of analytes on sorbent and concluded that, they affect retention and elution. David *et al.* [23] reported that any extraction method developed on spiked samples will not necessary yield high recoveries of spiked analyte when significant analyte interaction exist and does not elute.

LLE technique was used to extract monocrotophos from various types of water to determine whether there was analyte loss during the extraction and that SPE systems were in control. Soren, et al, [24] affirms that in spiked recovery studies including SPE techniques, LLE technique should be performed to demonstrate collection efficiences and to demonstrate that no loss of target analyte occur during extraction. The results obtained from this study using LLE technique to extract monocrotophos from DW, DDW and SW indicates that the percentage analyte loss values for monocrotophos extracted from DDW and DW was less than the value obtained in SW (figure 5 and 6). The higher values obtained in SW may be due to the presence of different matrices which may interact with monocrotophos thereby reducing their recovery from the stream water while the lower values of percentage analyte loss may be due to the fact that DDW and DW are significantly free from these matrices. Figure 5 and 6 show an increase in percent analyte loss from SW as compared to DDW and SW for monocrotophos using both techniques. This may be that these pesticides did adsorb but did not elute from the GAC solid phase extraction column. The mean analyte loss from extraction using SPE systems were approximately twice that of LLE techniques. Although, similar mean analyte loss were obtained for monocrotophos using LLE technique as compared to their loss in SPE system. The difference in mean percent loss may be due to diffusion of the analyte into the interior of the GAC and its interaction with water matrices and difficult to be eluted by n-hexane as compared to LLE techniques which has no sorbent but can only interact with only water matrices. However, the comparison revealed that there was a significant loss of analyte during SPE systems and the systems were in control.

In conclution granulated activated charcoal as an adsorbent can be used to extract organophosphorus from water. Granulated activated charcoal can be used as absorbent for the extraction of monocrotophos from water and eluted with n-hexane. It is hereby recommend that, there should be more researches on the usage of SPE systems for monitoring water pollution and for regulatory purposes using granulated activated charcoal as an absorbent. Similarly, other solvents such as dichloromethane and acetonitrile should be used for elution of monocrotophos from granulated activated charcoal. Other extraction techniques be experimented for their extractions and compared with SPE.

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