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Determination of Organochlorine and Organophosphorous Pesticide Residues in Irrigated Water from Gubi, Waya Dams and Gudum Fulani Irrigation Sites in Bauchi LGA, Bauchi State,Nigeria Using Composite Sampling.

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ABSTRACT

Due to the vital role of water for humanity, it is necessary to improve and maintain its quality. Environmental and global changes especially industrial wastes, domestic and agricultural activities are the main sources of water pollution.This research seeks to investigate organochlorine and organophosphorous pesticides residues from irrigated water samples collected from Gubi, Waya Dams and Gudum Fulani irrigation sites in Bauchi, Bauchi State. Water samples were collected and extracted by liquid –liquid extraction using dichloromethane (DCM) solvent. Water extracts were quantitatively analyzed using GC-MS analysis. GC-MS analysis revealed that: Heptachlor (9.887±0.01 mg/L) was detected from WS1. p,p^1–DDT (0.293±0.26 mg/L) was detected in WS3. Dichlorvos (2.682±0.041 mg/L, 1.894±0.009 mg/L) were obtained from WS1 and WS3 respectively. The results obtained exceeded their Maximum Residue Limit (MR) when compared with USEPA and Codex Alimentarious FAO/WHO. The order of contamination of water samples is WS2 < WS1 < WS3 with WS3 most contaminated. The results showed that these pesticides may show some health concern and the relevant regulatory Agencies of Government should urgently regulate and reinforce the proper use of hazardous pesticides.

1. Introduction

Water plays an essential role for life on earth. It is one of our most important natural resources. Due to fast growing of our civilization, the demand for water has increased dramatically and its uses have become more expensive. Large volume of water is used in industry, agriculture and for personal consumption, with varied impurity. The common harmful impurities of water include bacteria, viruses and different chemical substances such as pesticides. Nowadays, the contamination of water by pesticides is very important ecological problem especially where there is an intensive agricultural practice that involves releasing of highly toxic substances into the water supply which may cause serious effect on human and animal health [1]. Due to the vital role of water for humanity, it is necessary to improve and maintain its quality. Environmental and global changes especially industrial, domestic wastes and agricultural activities are the main sources of water pollution [2].

1.1Pesticides

Pesticides are any substance or mixture of substances used for preventing, destroying, or killing of pest. A pest is an organism that attack and destroy our farm produce. When a pesticide is applied to a Farm land, certain reactions follow. It is first stick to leaves where it is absorbed, then rain fall inevitably washes some of the chemicals off leaf surface onto the soil below and some may be transformed by sunlight [3].

Every fresh water body such as rivers, lakes and wells may likely contain pesticide residues as pollutants which leached out from soil surfaces or through aquifers. The residues enter natural water from direct application for control of aquatic weeds, trash fish, aquatic insects, percolation and run off from agricultural production fields, drift from agro-allied industrial waste water and discharge from waste waters from clean-up equipment for pesticides formulation application. The residues of these pesticides that are present in water should not exceed extreme limits as this may cause a threat to human health [4]. Organochorine pesticides have long residual action and persist in the environment for long period without losing toxicity [5]. Even though they have been banned since 1984 to 1988, but their residues are still found in the environment [6]. They have lives ranging from months to years and in some cases decades [7]. Organophosphorous are less persistent in the environment and have high acute toxicity compared to organochlorines. High levels of pesticide residues are responsible for the poisoning and several health's' hazard in both rural and urban areas in Nigeria [8].

1.2 Classification of Pesticides

Pesticides are broadly classified into insecticides, fungicides and herbicides. Insecticides are further classified into Organochlorines, Organophosphorus, Carbamates and Pyrethroids [9]. There are also inorganic pesticides such as copper, sulfur and arsenicals [10]

1.2.1 Organochlorine Pesticides

These pesticides contain carbon, hydrogen and chlorine. They were developed in the mid-1940s through 1950s and were banned for sale in the U.S by the late 1980s due their detrimental effect [10]. Organochlorine pesticides consist of two different groups based on their molecular structures, namely; the cyclodiene or diene and the DDT groups. They are considered to be eco-toxic and have long term effects on the environments and human health [3].They are generally categorized as chlorinated cyclodienes, chlorodiphenylethanes, chlorinated benzene and cyclohexanes [14].

1.2.1.1 Cyclodiene Organochlorine Pesticides

These are cyclic compounds possessing the characteristics of "endo-methylene bridged" structure. Usually, they are product of Diels-alder reaction of hexa-chloropentadiene and a saturated compound. Examples are heptachlor, chlordane and aldrin [3] as shown in figure 1.1.

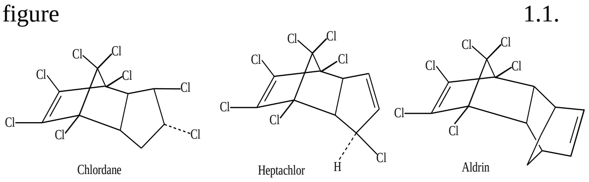
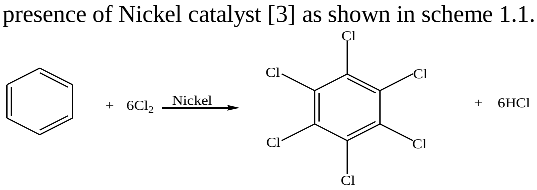


Figure 1.1: Some Structures of Cyclodiene Organochlorine Pesticides

1.2.1.2 Hexachlorobenzene (HCB) Organochlorine Pesticides

Hexachlorocyclohexane (HCH), also known as benzene hexachloride (BHC), is an organochlorine insecticide that is available in two formulations - technical grade HCH and lindane. Technical grade HCH is a mixture of different isomers: a-HCH (60-70%), b-HCH (5-12%), g-HCH (10- 15%), d-HCH (6-10%), and e-HCH (3-4%).They are used to protect the seeds of onion, sorghum, wheat and other grains against fungus. They are prepared through chlorination of Benzene in the presence of Nickel catalyst [3] as shown in scheme 1.1.



Scheme 1.1: Preparation of Hexachlorobenzene

1.2.1.3 DDT Organochlorine Pesticide and its Analog

DDT is a very effective insecticide used in public health for the eradication of mosquitoes. They contain two aromatic rings and represent the major organochlorine pesticides (OCPs).Insects sprayed with DDT exhibit hyper activity and convulsion consistent with the interaction of the DDT with the nervous system. Example of DDT and its analog include: Methoxychlor, p, p[1] – DDT, α -p-DDT. [3].as shown in figure 1.2.

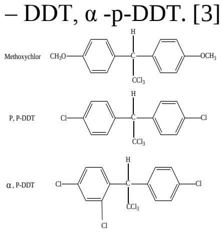
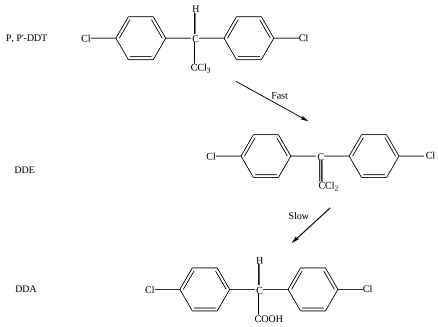


Figure1. 2: Some Structures of DDT Derivatives

1.2.1.4 Environmental Degradation of DDT

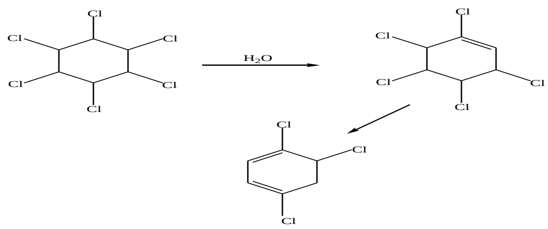
In the environment, p,p[1]-DDT (Para, Para Dichloro Diphenyl Trichloro Ethylene) undergoes relative degradation by the elimination of HCl group to yield Dichloro-Diphenyl Dichloro-Ethylene (DDE) when heated in water. Subsequent hydrolysis of the DDE to 2, 2-bis-(4-chloro phenyl) acetic acid (DDA) is extremely slow. Since there are only unreactive vinyl and aryl chloride in DDE, consequently, DDE is the principal DDT degradation product in the environment. [3] as shown in scheme 1.2.



Scheme1.2: Environmental Degradation of DDT

1.2.1.5 Lindane Organochlorine Pesticides

This is the gamma-isomer (<99% pure) of hexachlorohexane (HCH), it was one of the most widely used insecticides in the world in controlling a wide range of sucking and chewing insects, it is used in the control of insects such as insect borers, beatles and hornet, its main uses include treatment of seed, on crops, in warehouse, in forestry, on domestic and agricultural animals and for pest control of scabies and lice on human. It undergo slow environmental degradation by the elimination of the HCl group in the aqueous phase [3] as shown in scheme 1.3



Scheme1.3: Environmental Degradation of Lindane

1.2.1.6 Endosulfan Organochlorine Pesticides

This was introduced in 1954 and used as a contact and stomach insecticides and arcaricides in a great number of food and non-food crops. This compound has been formulated to be used in commercial agriculture, home gardening and wood preservation. The technical grade endosulfan contains at least 94% of two isomers: α-endosulfan and β-endosulfan [3] as shown in figure 1.3.

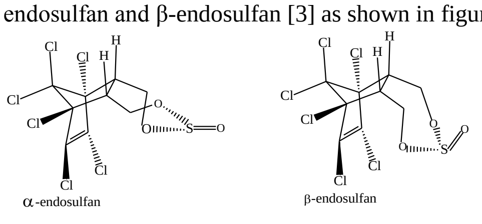


Figure1. 3: Some Structures of Endosulfan

1.2.2 Organophosphate Pesticides

This is a generic term referring to insecticides containing phosphorous, they were developed during the early 19th century, but their effects on insects which are similar to their effects on human, were discovered in 1932. Some of the compounds are quite toxic, the widely used organophosphates are glyphosate, Dichlorvos,

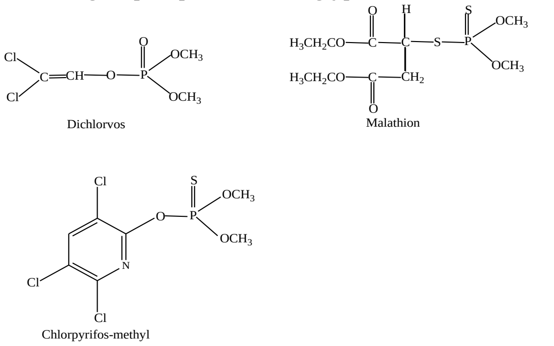


Figure 1. 4: Some Structures of Organophosphorous Pesticides

1.2.3 Carbamate Pesticides

Carbamates are class of pesticides that are structurally and mechanistically similar to organophosphate insecticides. They are derived from carbamic acid and cause carbamilation of acetyle cholinesterase at neuronal synapses and neuromuscular junction. They were introduced in 1950s.The common examples of carbamates include carbaryl, propoxur, oxamyl, methomyl, pirimicarb and aldicarb. They are toxic and can persist in the environment from weeks to months. They also act through similar mechanism as organophosphates by blocking an enzyme essential to the transmission of nerve impulse. [10]

1.2.4 Pyrethroids Pesticides

Pyrethroids are organic compounds similar to the natural pyrethrins, which are produced by the flowers of pyrethrum. They are used as commercial and household insecticides. Synthetic pyrethroids developed in 1970s are more stable in the presence of light and have a higher insecticidal activity than organophosphate or carbamates, an example of synthetic pyrethroids is permethroids which have been used on fruits, vegetables and corn [10].

1.2.5 Inorganic Pesticides

These are chemicals that are carbon free based on its chemical structure, this form the basis for differentiating it from organic pesticides which are based in carbon. The minerals used in inorganic pesticides are extracted from the earth. An example of inorganic pesticide include Cu,

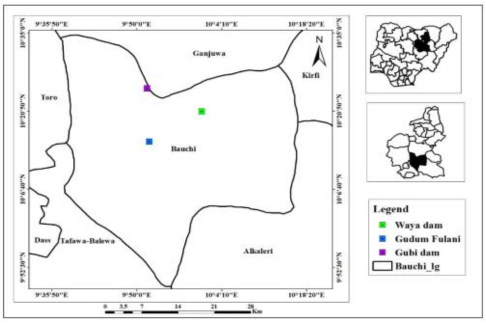


Figure 2.1: Map of Study Area and Sampling Sites

2.1 Extraction of Organochlorine and Organophosphorous Pesticides Residues from Water Samples

Liquid – liquid extraction method was used to extract pesticide residues from the water samples. The extraction

Malathion, chlorpyrifos-methyl etc. (Camarata, 2006) as shown in figure 1.4.

S and As. The major Arsenic-bearing compounds were Paris green and London purple [10].

1.2.6 Removal of Pesticide Pollutants from the Environment.

Owing to the detriment effects of the pesticide pollutants to human and the environment at large, researches are being conducted to ensure they are completely removed from the environment. It was reported that one of the promising and currently used method is heterogeneous catalysis, the method is relatively new and cheap because it involves the use of solar energy which is abundantly available and it can remove the pesticide completely from the environment without transferring them to another medium leading to the secondary pollution problem. The photo catalysts that are mostly used for the construction of solar cell include TiO_2, ZnS, gC_3N_4 and graphene oxide and their Nano composites. [11] Graphene and C_{2O} Fullerene were used to remove Cyanogen chloride pollutant in the environment which shows the possibility of using them in the removal of organochlorine pesticides. [12] Also N_4B_4 clusters were used for the removal of CO and CO_2 pollutants which in turn could be tried for gaseous pesticide pollutants. [13]

This research was aimed to investigate organochlorine and organophosphorous pesticides residues from irrigated water samples collected from Gubi dam, Waya dam and Gudum Fulani irrigation sites in Bauchi Local Government Area, Bauchi State, Nigeria.

2.0 Materials and Methods

2.1 Sample Collection

Samples of water were collected at 3 irrigation location from three different Dams (Gubi Dam, Waya Dam and Gudum Fulani) in Bauchi, Bauchi State, as shown in figure 2.1 based on the procedure described by [15] and [8]. The areas were rural settlements, where irrigation is often carried out in the Dams. The sampling was carried out late April, 2020. The sampling procedure used was composite sampling. Ten (10) samples were collected at each sampling location, and then mixed and homogenized to give one composite sample which is the representative of each location. Therefore, a total of 3 homogenized and mixed samples of water were collected from the 3 locations. The water samples were labeled and stored in a refrigerator till further use.

was carried out using the method reported by [15] as described by method 3510, [15]. 50.0 cm^3 of dichloromethane (DCM) was introduced into the separating funnel containing 100.0 cm^3 of the water sample and shake vigorously for 2 minutes. The sample was then allowed to settle for 30 minutes to ensure proper separation of the phases. After separation, the organic layer was filtered into a 250 cm^3 conical flask through anhydrous sodium sulphate (Na_2SO_4) that has been prewashed with Dichloromethane (DCM). The extraction was repeated twice using a 50.0 cm^3 portion of dichloromethane and later combined. The combined organic extracts were concentrated using a rotary evaporator at 45 ^{0}C and low pressure. 5.0 cm^3 of n-Hexane was added to the extract in DCM to exchange the solvent. The extracts were further concentrated to 1.0-2.0 cm^3 using a rotary evaporator at 45 ^{0}C until no further DCM remain in the extract. The extracts were then transferred into 2 cm^3 GC vials before analysis with Gas Chromatography.

2.2 Gas Chromatography –Mass Spectrometry Analysis for the Water Extracts

2.2.1 Gas Chromatographic Conditions for OCPs

The extracts (water) were determined with the aid of a Gas Chromatography equipped with a Mass- selective detector (GC-MS), an auto–sampler and a split–split less injector .The DB-5 fused silica capillary column of 30m × 0.25 µm i.d ×0.25µm, film thickness was coated with cross linked 5% phenyldimethyl polysiloxane. The carrier gas was Helium (99.999% purity) at a flow rate of 1.0 ml|min. Oven Temperature was maintained initially at 100 ^{0}C for 1 min, increased at 12 ^{0}C | min to 200 ˚C, then at 20 ˚C|min to 215 ˚C , at 10 ˚C|min to 265 ˚C for 7min , and then finally at 20˚C |min to 280˚C and held for 4min. Injection volume was 1 µL, injected split less mode at injection temperature 250 ˚C .The Mass Spectrometer was operated in electron impact (EI) ionization with detector voltage 70 eV ,ion source temperature of 200 ˚C ,GC interface temperature of 320˚C and emission current of 150 µV , Acquisition mode was Selected Ion Monitoring . This method was adopted from [8].

2.2.2 Gas Chromatographic Conditions for OPPs:

A gas chromatography model Agilent 7890B equipped with mass selective detector, model Agilent 5977A, auto sampler and flame photometric detector (FPD) was used for Organophosphorus (OP) determination. The carrier gas was Helium gas (99.999% purity), flow rate was 2.7 ml/min. The injection mode is split less while flow control mode was in constant flow. The temperature of injector and flame photometric detector was held at 250 ^{0}C respectively. The oven temperature was programmed at 60 ^{o}C for 1 minute, ramped to 200 ^{0}C at 10 ^{0}C/min (held for 2 minutes) and to 280 ^{0}C at 10 ^{0}C/min (held for 3 minutes). The total run time was 28 minutes. The column type is HP5 MS (30 m X 0.25µm and 0.32mm). [15].

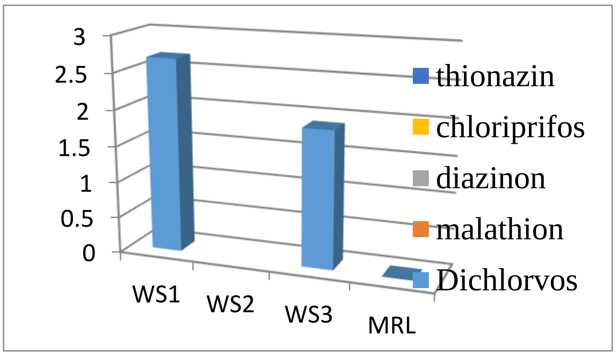
2.3 Preparation of Blank Solution:

3.1.3 Organophosphorous Pesticide Residues' s Concentration from Water Sample

Organophosphorous pesticide residues were detected in some of the water samples collected from the 3 irrigation site. Figure 3.2 shows the concentration of organophosphorous Pesticides detected.

Figure 3.1 : Mean Concentration (mg /L) of Organochlorine Pesticide Residues in Water Samples

Keys: WS1=Water Sample 1,WS2= Water Sample 2, WS3= Water Sample 3, MRL= Maximum Residue Limi



Similar volume of solvents (n-hexane/acetone) and anhydrous sodium sulphate which were used in the extraction were subjected to similar extraction and cleanup procedures as the examined samples to detect any possible traces of the studied pesticide.

2.4 Preparation of Calibration Curves:

Stock solutions of pure standards of organochlorine (α-BHC, γ-BHC, γ-lindane, heptachlor, endosulfan II, dieldrin, p, p^1–DDD, p, p–DDT, and 1,1-dichloropentane) and Organophosphorus (Dichlorvos, chlorpyrifos , Diazinon, Malathion and thionazin) were prepared and then serially diluted to produce different concentrations of pesticides. Stock solutions were stored in amber coloured bottles at 4 ^{0}C in a refrigerator while working standard solutions were prepared fresh before use. Standard solutions of OCPs and OPPs were run in GC- ECD and GC-FPD under set chromatographic conditions and the main peak areas were plotted against concentrations to obtain calibration curves of individual pesticides. The retention times for standard samples were used for confirmation of the pesticides. Retention time windows were constant for the standard samples and were therefore relied upon for component identification. Calibration curves were produced with four different standard concentrations.

3. Results and Discussion

3.1 Results

3.1.1 GC–MS Analysis

The water samples were extracted and analyzed using GC–MS (GC 7890B, MSD 5977A, Agilent Tech.) at Yobe State University Damaturu, Yobe State, Nigeria.

3.1.2 Organochlorine Pesticide Residues's concentration in Water Samples

Organochlorine pesticides were detected in some of the water samples collected from the irrigation sites. Figure 3.1 shows the concentration of organochlorine pesticides residues detected.

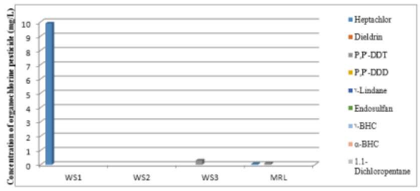


Figure 3.2: Mean Concentration (mg/L) of Organophosphorous Pesticide Residues in Water Samples

Keys: WS1=Water Sample 1, WS2=Water Sample 2, WS3= Water Sample 3, MRL= Maximum Residue Limit.

3.2 DISCUSSION

The results of the mean concentration of organochlorine and organophosphorous pesticide residues in water samples collected from 3 irrigation sites (Gubi dam, Waya dam and Gudum Fulani) in Bauchi local Government area of Bauchi state were presented in Figures 3.1-3.2. Figure 3.1 showed the organochlorine pesticide residues in water samples from the 3 irrigation sites. The results showed that Heptachlor and p, p^1–DDT were detected in Gubi dam and Gudum Fulani water samples respectively. The mean concentration of Heptachlor was 9.887±0.01 mg/L, which was higher than the maximum residue limit (MRL) of 0.05 mg/L according to USEPA and Codex Alimentarious [16]. The high level of Heptachlor might be due the long term application of Heptachlor which persisted in the environment, because the pesticides usually applied in the site are sharp shooter, imidaclopride and rich soil which are also organochlorine pesticides. Also, the mean concentration of p, p^1-DDT was found to be 0.293±0.26 mg/L which was relatively high when compared to MRL of 0.10 mg/L [17].The high level of P,P[1]-DDT might be due to the unregulated application which also persisted in the environment.

The high level of these pesticides may cause skin sensitization, allergic reaction and rash, contact dermatitis, enzyme induction and kidney tumor [4]. It can be neurotoxic, carcinogenic, endocrine disruptor; this implies that they can affect human growth and reproduction [8]. The situation is evident as some of the farmers around the areas were found to be suffering from neurological diseases, hypertension, loss of consciousness, frequent headache and as a result, many health problems could be discovered if detailed studies on the health status of the famers around the areas are to be carried out.

Most of the famers are not aware of the side effects of these pesticides, believing that whatever health problem they have is from Allah, not knowing that, there is cause and effect for everything. Therefore, awareness on the site effects of these pesticides and health problem associated with them should be carried out regularly.

Figures 3.2 showed the mean concentration of organophosphorous pesticide residues in water samples from the irrigation sites. The results showed that dichlorvos was detected in Gubi dam, and Gudum Fulani water samples. The mean concentrations of dichlorvos in the 3 samples were 2.682±0.041 mg/L, 1.894±0.009 mg/L and 3.900±0.015 mg/kg obtained from Gubi dam and Waya water samples respectively. The results are higher than MRL of 0.0005 mg/kg [16] which might be attributed largely to the farmer's practices in the use of these insecticides. High level of dichlorvos may exert toxic effects in human by inhibiting the enzyme, cholinesterase. Effects from acute exposure include perspiration, nausea, vomiting, diarrhea, drowsiness, fatigue, headache and at every high concentration, convulsions and coma.

Most of the famers are ignorant about the proper method of spraying the pesticides, personal protecting equipment (PPE) such as hand gloves, socks, gown, nose mask, face mask, eye glass should be used while spraying, after spraying, and hands should be washed thoroughly before eating food. Milk especially liquid type greatly help in

4. Conclusion

From the analysis, it can be clearly concluded that Heptachlor,1,1-dichloropentane,p,p1-DDT organochlorine and dichlorvos organophosphorous pesticide residues were detected in the three irrigation site (Gubi dam, Waya dam and Gudum Fulani) with their concentrations exceeded their maximum residue limits and are therefore among the major sources of contamination in water in Gubi dam, Waya dam and Gudum Fulani irrigation sites in Bauchi, Bauchi state, as a result of unregulated application of the pesticides by the famers or long term application of the pesticides, non-compliance or ignorant on the preventive measures and above all carelessness from the Government and its stakeholders which consequently, makes these pesticides persisted in the environment, posing serious health problems to the populace.

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