Academy Journal of Multidisciplinary Doctoral Research [AJMDR]
Volume 3, Issue 1, 2025 ISSN: 2992-4669 || eISSN: 1116-3321

Determination of Organochlorine and Organophosphorous Pesticide Residues in Irrigated Soil from Southern Parts of Bauchi State, Nigeria.

Sallau S[1]*, Sani U[1], Abubakar A[2], Zango AM[1], Muhammad AA[2] and Idris SY[2]

[1]*Department of pure and applied chemistry, Bayero University kano, Kano State, Nigeria*
[2]*Department of chemistry Abubakar Tafawa Balewa University Bauchi, Nigeria*

Corresponding Author's Email: sallaushaaban@gmail.com
Received: 17-01-25
Accepted: 19-03-25
Published: 30-04-25

Abstract

Chemical compounds or mixes known as pesticides are intended to prevent, eliminate, or kill pests which are organisms that cause harm to crops, lower agricultural yields, or contaminate products that are kept. This study examines the pesticide residues of organochlorine and organophosphorous materials in soil samples that have been watered. Using a mortar and pestle, a 25.00 g soil sample was combined with 50.00 g of granular sodium sulfate and ground into a powder. Subsequently, the powdered material underwent normal methods for extraction, concentration, cleaning, and GC-MS analysis. Heptachlor (12.278±0.023 mg/kg) was detected in SS1 and 1,1-dichloropentane (0.5296±0.0012 mg/kg and 0.797±0.014 mg/kg) was found in SS2 and SS3, respectively, according to the GC-MS analysis. In SS2, dichlorvos (3.900±0.015 mg/kg) was found. The Maximum Residue Limits (MRL) established by the USEPA and Codex Alimentarius FAO/WHO are exceeded by these results. Samples are contaminated in the following order, SS3 < SS1 < SS2 with SS2 being the most contaminated. Based on the findings, which point to possible health dangers, government regulatory bodies should take immediate action to ensure that dangerous pesticides are used and regulated properly.

Keywords: Organochlorine, Pesticides, Organophosphorus, Soil, GC-MS.

1.0 Introduction

Chemical compounds or mixes intended to prevent, eliminate, or kill pests are known as pesticides. Organisms known as pests degrade crops, decreasing agricultural productivity and posing a risk to items that are processed, marketed, or stored [1]. Applying pesticides on farms is the main cause of pollution in the environment because runoff from rains frequently carries the chemicals from leaf surfaces into the soil and waterways. Sunlight can also change certain insecticides. Pesticides sprayed on soil have an initial interaction with soil moisture and subsequently with soil particles, affecting the environment's chemical processes. Various chemical events, including sorption processes (transfer), microbiological and chemical breakdown (transformation), volatilization to the atmosphere, leaching into different soil profiles, and and transportation by overland movement, take place in soil and regulate the environmental fate of pesticides.[1]. Pesticide residues are frequently found in freshwater bodies including rivers, lakes, and wells as a result of contaminants that seep through aquifers or soil surfaces. In addition to direct application for the purpose of controlling aquatic weeds, trash fish, and aquatic insects, these residues can also find their way into natural waters through percolation and runoff from agricultural fields, drift from agro-industrial

wastewater, and discharge from pesticide formulation and application equipment. Because pesticide residues are harmful to human health, their levels in soil and water shouldn't be allowed to go beyond certain bound s [2]. Organochlorine insecticides exhibit a prolonged duration of action and can withstand prolonged environmental exposure without diminishing in toxicity [3]. Although they have been outlawed since 1984 and 1988, their remnants are still present in the surroundings [4]. The duration of these pesticides can vary from months to years, and in certain situations, even decades [5]. In Nigeria, both rural and urban regions face numerous health dangers and poisoning due to high levels of pesticide residues, despite being less persistent in the environment than organochlorines [6].

1.1 Adverse Effects of Pesticides on Humans

Pesticide contamination of food and its build-up provide serious health hazards to the general public, resulting in both acute (short-term) and chronic (long-term) damage, with the liver and kidneys being the most commonly affected organs. issues of acute toxicity from pesticides including organochlorines and organophosphorous compounds include headaches, nausea, dizziness, weakness, eye problems, rashes, and neurological issues. Prolonged exposure can cause cancer development, infertility problems, genetic mutations, and other major health problems [7]. Numerous organochlorines and their metabolites are extremely toxic and linked to a number of harmful health outcomes, including as immune system dysfunction, cancer, neurological impairment, abnormalities of the reproductive system, and birth defects [3]. Pesticides containing organochlorines are extremely hazardous to the majority of aquatic life and extremely damaging to humans and other organisms. Even at low doses, they can have detrimental short- and long-term impacts. Furthermore, non-lethal consequences like immune system and reproductive harm can be very subst antial[8].

Non-biodegradable pesticides, such as those with organochlorine and organophosphorous content, are prohibited, yet they nonetheless provide a serious risk to people, animals, aquatic life, and the environment. Since farmers still use these pesticides, it is necessary to continuously evaluate and monitor them as well as the environmental residues they leave behind.

1.2 Natural Remediation of Pesticide Contamination

Research is being done to make sure that pesticides are totally eliminated from the environment because of the serious threats they represent to both human health and the environment.

Heterogeneous catalysis is one of the most successful, promising, and widely used techniques at the moment. With the use of solar energy, which is widely available, this relatively new and economical method removes pesticides from the environment without letting them enter another medium, therefore avoiding the risk of secondary contamination. In the creation of solar cells, photocatalysts such as TiO_2, ZnS, gC_3N_4, graphene oxide, and their nanocomposites are frequently employed [13].

Investigating the pesticide residues of organochlorine and organophosphorous materials in irrigated soil samples taken from Bauchi State, Nigeria's southern regions, is the goal of this study.

2. Methodology

2.1. Sample Collection

As indicated in Figure 1, soil samples were gathered using the procedure outlined by [9] and [6] from three irrigation zones connected to three distinct dams (Gubi Dam, Waya Dam, and Gudum Fulani) in the southern regions of Bauchi State, Nigeria. These are rural communities where dam water is frequently used for irrigation. In late April of 2020, sampling was conducted. Composite sampling was the technique employed for sampling. A composite sample typical of each location was created by combining ten samples from each site and homogenizing them. As a result, three homogenized composite soil samples were taken from each of the three locations. The soil samples were labeled, allowed to air dry, and kept in the proper containers pending additional examination.

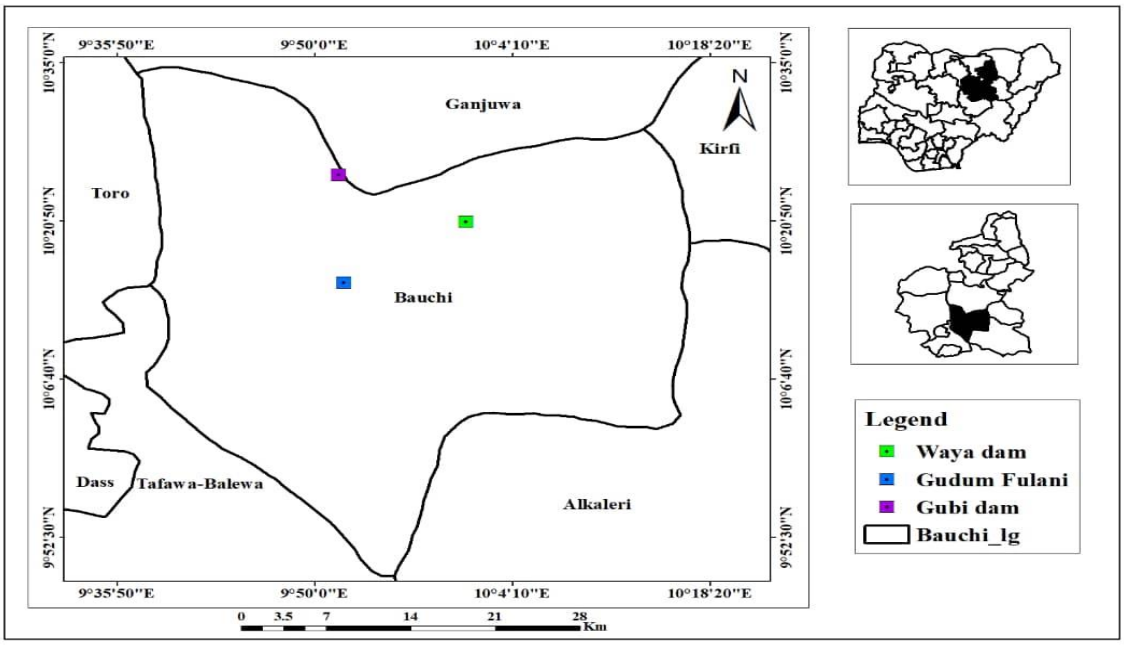

Fig. 1: Map of Study Area and Sampling Site (Sallau *et al*., 2022)

2.2 Extraction of Organochlorine and Organophosphorous Pesticide Residues from Soil

Using a mortar and pestle, 25.00 g of soil sample and 50.00 g of granular sodium sulfate were combined and mashed into a powder. After that, the powdered sample was extracted using 150.00 cm^3 of an n-hexane:acetone (1:2) mixture. After being moved to a flask with a circular bottom, the extract was concentrated to a volume of around 20.00 cm^3 in a water bath that was kept between 50°C and 55°C. Using a rotary evaporator, the residual solvent in the concentrated extract was further evaporated to a volume of roughly 5.00 cm^3. Before being subjected to GC-MS analysis, the concentrated extract was quantitatively transferred to a centrifuge tube, further concentrated to 0.50 cm^3 using a nitrogen evaporator, and diluted to 2.00 cm^3 in hexane [10].

2.3 Extract Clean-up

Pesticides and large molecular elements like lipids, proteins, pigments, and residues frequently coexist in soil extracts. These materials, called "dirt," need to be taken out of the extracts in order to prepare them for chromatographic examination. Their existence may harm the GC apparatus in addition to interfering with the chromatographic system and detection procedure [6]

2.4 Clean-up Method for the Soil Extracts

Silica gel that has been activated was used to clean soil samples. Two grams of deactivated silica gel and one gram of anhydrous Na_2SO_4 (an adsorbent) were placed inside a column that measured roughly fifteen centimeters in length and one centimeter in internal diameter. Before cleaning, each column received 15.00 cm^3 of n-hexane conditioning. After adding the extracts to the column, 20.00 cm^3 of n-hexane and diethyl ether (1:1 v/v) were used to elute the mixture. Using a rotary evaporator, the eluate was dried out and recovered into 2.00 cm^3 of n-hexane. For GC-MS analysis, the extracts were put into glass GC vials [6].

2.5 Gas Chromatography–Mass Spectrometry Analysis for the Soil Extracts

2.5.1 Gas Chromatographic Conditions for OCPs

A Gas Chromatography-Mass Spectrometry (GC-MS) system with an autosampler and a split-splitless injector was used to analyze the extracts

from the soil samples. Cross-linked 5% phenyl-dimethylpolysiloxane was deposited on the DB-5 fused silica capillary column (30 m × 0.25 mm i.d. × 0.25 µm film thickness). The carrier gas was helium (99.999% pure), flowing at a rate of 1.0 milliliter per minute.

After being kept at 100°C for one minute, the oven temperature was raised to 200°C at a rate of 12°C per minute, and then to 215°C at a rate of 20°C per minute. After that, the temperature was raised by 10°C/min to 265°C and held there for 7 minutes. Afterward, it was raised by 20°C/min to 280°C and held there for 4 minutes. 1 µL of the injection was made in splitless mode at 250°C injection temperature.

With a detector voltage of 70 eV, an ion source temperature of 200°C, a GC interface temperature of 320°C, and an emission current of 150 µA, the Mass Spectrometer ran in electron impact (EI) ionization mode. SIM, or selected ion monitoring, was the acquisition mode. This procedure was modified from [6].

2.5.2 Gas Chromatographic Conditions for OPPs

An Agilent 7890B gas chromatography system with a mass selective detector (Agilent 5977A), an autosampler, and a flame photometric detector (FPD) was utilized to determine the presence of organophosphorous pesticides (OPPs). The carrier gas, helium (99.999% pure), was pumped at a rate of 2.70 milliliters per minute. Splitless injection was the method of operation, while continuous flow was the flow control mode. Temperatures of the injector and flame photometric
detector were kept constant at 250°C.

Following a one-minute initial setting at 60°C, the oven temperature was ramped up to 200°C at a rate of 10°C/min (held for two minutes), and then increased to 280°C at a rate of 10°C/min (held for three minutes). There were 28 minutes throughout the run. An HP-5 MS (30 m × 0.25 µm × 0.32 mm) column was employed [9].

2.6 Preparation of Standard Solution

To find any potential traces of the targeted pesticides, the identical volumes of solvents (n-hexane/acetone) and anhydrous sodium sulfate used in the soil extraction were put through the same extraction and clean-up processes as the sampled tests.

2.7 Preparation of Calibration Curves

First, stock solutions of pure standards of organophosphorus pesticides (dichlorvos, chlorpyrifos, diazinon, malathion, and thionazin) and organochlorine pesticides (α-BHC, γ-BHC, γ-lindane, heptachlor, endosulfan II, dieldrin, p,p'-DDD, p,p'-DDT, and 1,1-dichloropentane) were made and then serially diluted to produce various concentrations. Working standard solutions were made fresh before use, and stock solutions were kept in amber-colored bottles in a refrigerator at 4°C.[13]

GC-ECD and GC-FPD were used to examine standard solutions of OCPs and OPPs under predetermined chromatographic conditions. Plotting the obtained peak regions against concentrations allowed calibration curves for each insecticide to be created. The pesticides were

verified to be present using the retention periods for standard samples. For the standard samples, retention time frames were reliable and could be depended upon for compound identification. Calibration curves were generated with four different standard concentrations. [13]

3.1 Results

3.1.1 GC-MS Analysis

The water and soil 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 Residue Concentration in Soil Samples

Certain soil samples that were taken from the irrigation sites included pesticides with an organochlorine content. The concentrations of the found organochlorine pesticide residues are shown in Figure 2.

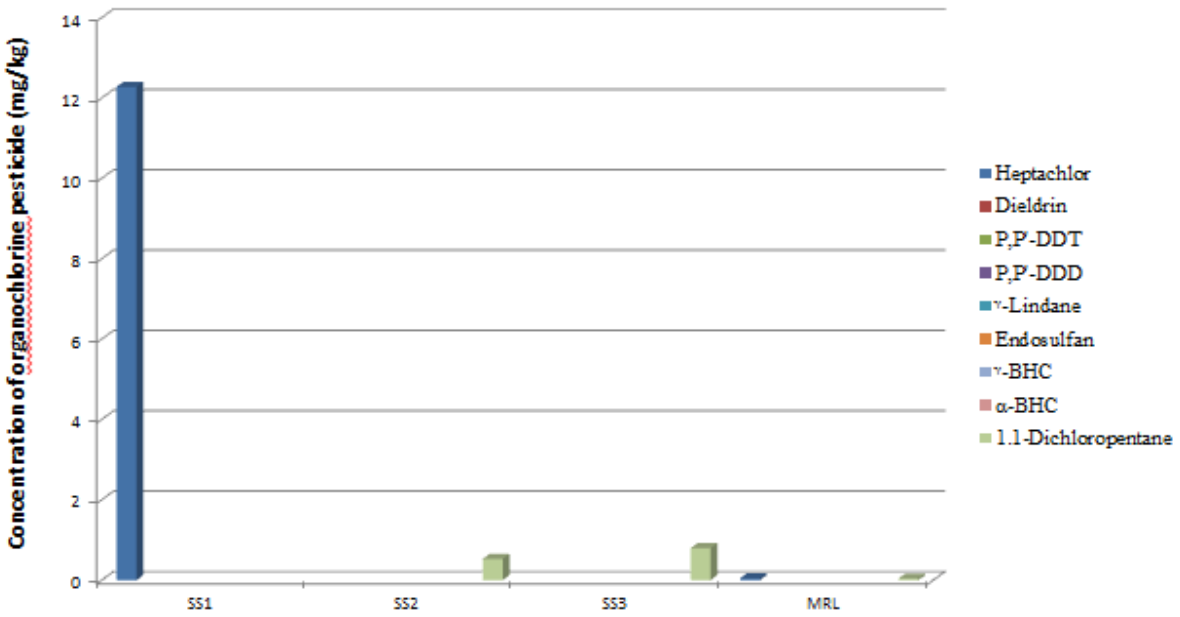

Fig. 2: Mean Concentration (mg/kg) of Organochlorine Pesticide Residues in Soil Samples

Keys: SS1=Soil Sample 1, SS2= Soil Sample 2, SS3= Soil Sample 3, MRL= Maximum Residue Limit.

3.1.3 Concentration of Organophosphorous Pesticide Residues in Soil Samples

Organophosphorous pesticide residues were found in a number of soil samples taken from the three irrigation sites. Figure 3 shows the concentrations of various insecticides.

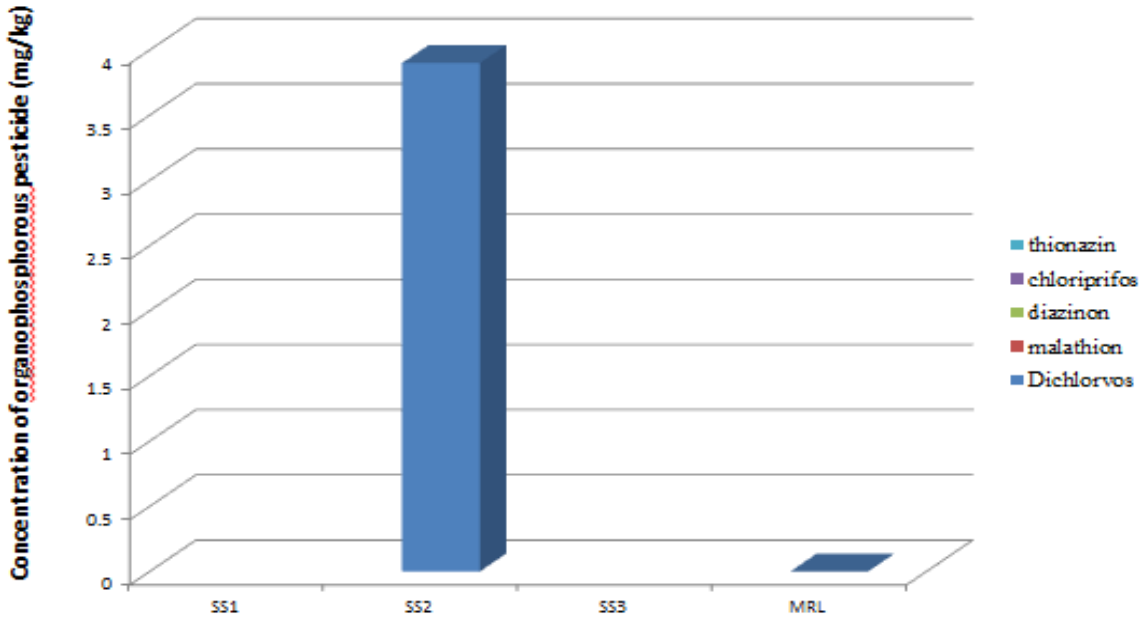

Fig. 3: Mean Concentration (mg/kg) of Organophosphorous Pesticide Residue in Soil Samples

Keys: SS1=Soil Sample 1, SS2 = Soil Sample 2, SS3= Soil Sample 3, MRL= Maximum Residue Limit.

3.2 Discussion

Figures 2 and 3 display, respectively, the average concentrations of pesticide residues that are organochlorine and organophosphorous that were found in soil samples taken from three irrigation locations in southern Bauchi State, Nigeria: Gubi Dam, Waya Dam, and Gudum Fulani.

The average amounts of organochlorine pesticides detected in the soil samples are shown in Figure 2. The findings showed that 1,1-dichloropentane and heptachlor were present. Samples of soil from Waya Dam and Gudum Fulani had 1,1-dichloropentane, whereas samples from Gubi Dam contained heptachlor. According to [13], the maximum residual limit (MRL) of 0.05 mg/kg was severely exceeded by the mean concentration of eptachlor, which was 12.278±0.023 mg/kg. The mean 1,1-dichloropentane concentrations were 0.5296±0.0012 mg/kg and 0.797±0.014 mg/kg, respectively, above the 0.04 mg/kg MRL [11]. It's possible that uncontrolled fumigation and pesticide application at Waya Dam and Gudum Fulani sites contributed to the higher 1,1-dichloropentane levels in the soil samples.

Because 1,1-dichloropentane can be harmful if breathed in or absorbed through the skin, the high concentration presents health risks. Skin and eye irritation or burns may result from exposure to contaminated material. Many farmers don't know about the adverse effects of these pesticides and instead blame any health problems on chance rather than realizing the cause-and-effect connection. Therefore, it is essential to routinely run campaigns raising awareness of the dangers pesticide use poses to one's health.

Farmers frequently don't know how to apply pesticides correctly or how important it is to wear personal protective equipment (PPE), like masks, gowns, gloves, socks, and eye protection. It is imperative to properly wash hands after using a spray before consuming food. Eating liquid milk right away can assist in offsetting the effects of p esticides.

In comparison to water samples, soil samples had greater concentrations of pesticide residues that were organochlorine and organophosphorous [13]. This disparity is explained by the fact that soil is the main source of contamination, with very little pesticides seeping into bodies of water. In addition, the higher persistence and concentration of these pesticides in soil samples as opposed to water samples can be attributed to variables like temperature, pressure, soil texture, ionic strength, and the presence of metal ions.

Conclusion

The data unequivocally shows that traces of the organic pesticides dichlorvos (an organic phosphorous pesticide), 1,1-dichloropentane (an organochlorine pesticide), and hepthachlor (an organochlorine pesticide) were found in the three irrigation locations (Gubi Dam, Waya Dam, and Gudum Fulani). In certain parts of southern Bauchi State, Nigeria, the quantities of these pesticides were higher above their maximum residue limits, indicating that they are important sources of soil pollution.

The main causes of this contamination are farmers' prolonged or uncontrolled use of pesticides, their disregard for precautionary measures, and their overall lack of understanding of safety

precautions. Furthermore, the persistence of toxic chemicals in the environment has been facilitated by the neglect of stakeholders and the government, endangering the health of the local population.

References

1. Aiyesanmi, A. F., & Idowu, G. A. (2018). Organochlorine pesticide residues in soil of cocoa farms in Ondo State Central District, Nigeria. *Environment and Natural Resources Research,* **2**(2).
2. Chawla, P., Kaushik, R., Swaraj, V. J. S., & Kumar, N. (2018). Organophosphorous pesticide in food and their colorimetric detection. *Environmental Nanotechnology Monitoring and Management,* 10, 292- 307.
3. Chikezie, O. I., Abdullahi, M., Jonathan, Y., & Abdullahi, B. (2017). Assessment of organochlorine pesticide residues in soil and water from Fadama farming communities in Minna, North Central, Nigeria. *Journal of Environment,* **2**(5), 48-55.
4. FAO/WHO. (2004). Codex Alimentarius Commission, additives and contaminants, joint FAO/WHO Food Standards Program, ALINORM 01/12A, PP, 1-289. Pesticides residues monitoring.
5. Fosu-Mensah, B. Y., Okoffo, E. D., Darko, G., & Gordon, C. (2016). Assessment of organochlorine pesticide residue in soil and drinking water sources from cocoa farms in Ghana. *SpringerPlus*, 5, 869.
6. Food Standards Program. (2004). Pesticides program residues monitoring. ALINORM 01/12A, 1-289. Codex Alimentarius Commission, Joint FAO/WHO Food Standards Program.
7. Ibigbami, O. A., Aiyesanmi, A. F., Adeyeye, E. I., Adebayo, A. O., & Aladesanwa, R. O. (2015). Assessment of organochlorine and organophosphorous pesticide residues in water and sediment from Ero River in South Western Nigeria. *Journal of Chemical Biological and Physical Sciences*, **5**(4), 4679-4690.
8. Ize-Iyamu, O. K., Asia, I. O., & Egwakhida, P. A. (2007). Concentration of residues from organochlorine pesticides in water and fish from rivers in Edo State, *Nigeria. International Journal of Physical Sciences*, **2**(9), 237-241.
9. Knedel, W., Chiquin, J. C., Perez, J., & Rosales, S. (2017). Determination of pesticides in surface and ground water used for human consumption in Guatemala.
10. Mazlan, N., Ahmad, M., Muharam, F. M., & Alam, M. D. A. (2017). Status of persistent organic pesticide residues in water and food and their effects on environment and a farmer: A comprehensive review in Nigeria. Network of Scientific Journal from Latin America, **38**(4), 2221-2236.
11. Njogu, P. M. (2011). Assessment of pollution and production of environmental risks of organochlorine pesticide residues on aquatic communities in Lake Naivasha, Kenya. A thesis submitted in fulfillment for the degree of Doctor of Philosophy in Environmental Technology at Jomo Kenyatta University of Agriculture and Technology, 45-57.
12. Shahat, M., Ammar, M. S., Ramzy, S. M., & Baz, A. M. (2017). Health risks assessment of toxic heavy metals and pesticide residues in some raw animal foods collected from Egyptian market. *Current Science International*, **6**(2), 421-430.
13. Sallau, S., Umar, S., Abubakar, A., Zango, A.M., Idris, S.Y., & Abubakar H. (2022). 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. *Journal of Chemistry Letters*.**3** (2022): 119-123.