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# Determination ofSynthetic PyrethroidsPesticide Commonly used by farmers in Upper Yedzaram Basin Adamawa State, Nigeria.

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**ABSTRACT:** In this study, Gas Chromatography/Mass Spectroscopy (GC/MS), equipped with an electron capture detector (ECD) was used to determine the levels ofsynthetic pyrethroid pesticide residues in fruits, crops, and vegetables along the Upper Yedzaram Basin of Adamawa State, Nigeria.The result shows cypermethrin as the most frequently detected pyrethroid pesticide residue in cabbage, tomato, garden egg, guava, and sweet potato samples. Detected in 62% of the samples followed by permethrin, detected in 38% of the samples.the result further show thatcypermethrin as the most frequently detected isomers of cypermethrin in the samples (24%) with zeta- cypermethrin, alpha-cypermethrin, and beta- cypermethrin detected in 19% of the samples respectively. furthermore, an isomer of cypermethrin detected in all the samples constitutes about 40% in cabbage samples, 24% in the tomato samples, and 22% in Guava samples respectively. The Estimated Daily Average Intake (EDAI) of cypermethrinandtheHazard quotient (HQ) estimated suggest the consumption of the vegetables/crops cultivated from the study location is unlikely to exert any adverse health hazard. Showing values below theAverage Daily Intake (ADI) of cypermethrin (0.02 mg/kg/day) and an HQ<1.Further assessment of the health indices (HI) for the mixture of endosulfan and cypermethrinsignifies that the adult population exposed to the mixture are unlikely to experience any adverse health hazard following the consumption of the plants. However, a level of concern exists, following the consumption of tomato and sweet potato by the children, showing an HI>1.Suggesting that the children's populace are more likely at risk for non-carcinogenic related complications on exposure to the frequently detected endosulfan and cypermethrin.

**KEYWORDS:**Pyrethroid,Vegetable, Fruit, Crop,Pesticide Residue, Yedzaram basin.

## I. INTRODUCTION

It is an undeniable fact that consumers' right to quality and safe food is under threat due to unwholesome agricultural practices involving indiscriminate use and applications of pesticides.Besides compromising food security, the threat is also manifested through ingestion, inhalation, and dermal exposureespecially to the farmers handling the applications (Bwatanglang, *et al*., 2019; Gerage, *et al*., 2017). This massive use of agrochemical by farmers to boosttheir enterprise contradicts the much-desired efforts to ensure food security due to the ubiquitous nature of pesticides such as its bioaccumulation potential, lipophilic properties and persistence in the environment (Gerage, *et al*., 2017; Li, *et al*., 2015; Olisah, *et al*., 2020). The persistence of pesticides in the soil medium interferes with the life cycle of non-targeted species and transcends easily through the trophic levels (Racke, 1992); as studies shows more than 90% of contaminants come through the food chain (Fürst, *et al*., 1990).Though enough information on the risk is in the public domain, pesticides remain a major ingredient in the entire agricultural enterprises; boosting production and increasing revenue generation at the risk of food safety and security. Showing pesticides usage in Africa accounting to about 2.1% of the world average from 1990 to 2016 (https://www.fao.org/faostat/en/#data/RP/visualize), which are reported to be largely influenced by biological, economic, and climatic factors (Olisah, *et al*., 2010). In Nigeria, about 125,000-130,000 metric tons of pesticides are expended annually (AsogwaandDongo, 2009). From 1983-1990, about 135 locally made pesticide product and nearly 200 different pesticide formulation were imputed into the country (Asogwa, and Dongo, 2009; Ikemefuna, 1998). Putting farmers and the public at risk due to a possible influx of substandard and fake pesticide products.This was supported in a study were about 72% of farmers in Nigeria were reported to procure pesticides in the open markets, which according to the report increases the likelihood of adulteration and imputation of fake and substandard pesticides products (WAAPP, 2013)

Against this backdrop, several studies were conducted in Africa and Nigeria, indicating the widespread use of organochlorine pesticides (OCPs) in the agricultural enterprises and its related health and environmental implications. Details were reported in the two-decade review conducted by Olisah, *et al*., (2020) and in the review presented by Mazlan, *et al*., (2016). Other studies reported traces of OCPs still in use in Nigeria (Akan *et al*., 2014; Bwatanglang *et al*., 2019; Ogah, *et al*., 2018; Tongo, and Ezemonye, 2015; WHO, 1990, WHO, 2010). However, this review and the studies were limited to the OCPs and with little or no emphasis on synthetic pyrethroid also used in agriculture.Apyrethroidis synthetic analogs of pyrethrin derived from pyrethrum plant (Chrysanthemum cinerariaefolium)and designed to mimic the insecticidal activity of the natural pyrethrum (Özkara*et al*.,2016).Although, they are often preferred because there are biodegradable and relatively less toxic to a mammal, (Sorgob and Villanova, 2002),however, found to exert toxicity to aquatic species such as mollusks, fish, and arthropods (Kourease*t al*., 2012; Roberts and Routt, 2013). A recent study by Ingenbleek*et al*. (2019) revealed the presence of 39 pesticides with 294 total occurrences, including 47.3% organophosphate pesticides and 35.7% pyrethroids in foodstuffs, commonly consumed in Benin, Cameroon, Mali, and Nigeria. Pertinent environmental health issues related to the use of synthetic pyrethroids in agriculture and general household in Nigeria are further discussed in a review by Ojo (2016).

Since it was established that farmers used mixtures of OCPs, organophosphate (OPs), and synthetic pyrethroids, it will not be out of place to assert that the application of more than one pesticide product with different metabolite, increases the chance of consumers ingesting a mixture of pesticides residues. Thus raising the level of concern about their potential synergistic interactions (Renwick, 2002). This observation was made in a study conducted by Arnold *et al.* (1996) reported a 1000-fold greater oestrogenic activity with combinations of pesticides than with single pesticides. Several studies have shown that endosulfan in combination with other pesticides, enhances the binding affinity to estrogen and perturb the endocrine system (Vega *et al*., 2007). We have already established and reported in our previous study that that farmers in the Upper Yedzaram basin frequently used endosulfan pesticides which according to the farmers interviewed are used either as a single insecticide or in a mixture with other pyrethroids or OPs(Zira*et al*., 2020). Based on these findings, this present work aimed toward establishing the most frequently usedpyrethroids pesticide commonly applied by farmers in the same study locations. And to further assess the health risk it will pose to consumers in association to the most frequently used OCPs (endosulfan) established from the previous studies.

## II. MATERIAL AND METHODS:

**Study Area:**Upper Yedzaram Basin with a total length of about 330 kilometers drive its source from Hudu Hills, in the south-east of Mubi, and extend northward toward the Lake Chad in Borno state (Martins and Gadiga, 2015). The study areas are situated along the river channels covering Mubi North, Mubi South and Maiha Local Government Area in the Northeastern part of Adamawa State, Nigeria. Adamawa state is located between latitudes 10°11 '30" and 10° 22' 30"N and between longitudes 13°13' 00''and 13° 30' 00" 'E (Adebayo and Dayya, 2004). The community along the basin are farmers thriving in vegetables, fruit, and cultivation of crops such as beans, soya bean, maize, groundnut, etc.

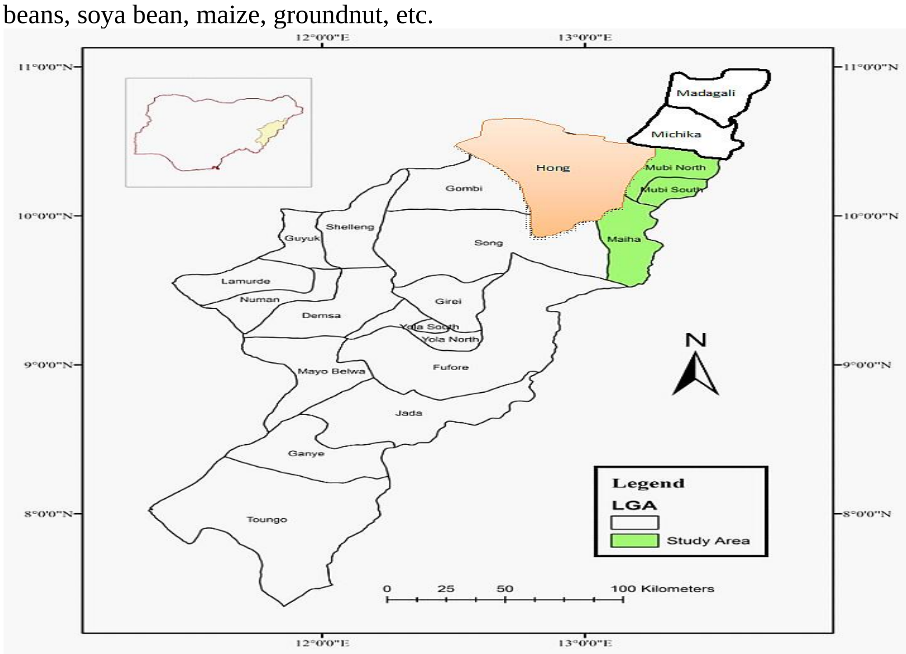



**Fig. 1 Map of Adamawa state showing upper Yedzaram covering Mubi North, Mubi South, and Maiha local government area of the state.**

**Sample Preparation and Analysis:**The samples were collected from three different locations, Shuware, WuroGude, and SabonGari along the Upper Yedzaram Basin covering Mubi North, Mubi South, and Maiha. Composite samples of the vegetables, (cabbage, tomato, garden egg), fruit (guava), and sweet potato were collected in each location from six farmlands. The sampling was carried out and treated using the same procedure described in our previous study (Zira *et al*, 2020) following the steps described by USEPA, (2007).

**Determination of Pesticide Residues in the Samples:**Following the extraction, the samples were collected and analyzed using the SHIMADZU GC/MS (GC-17A), equipped with a fluorescence detector for chromatographic separation achieved using a 35% diphenyl, 65% polysiloxane column. The retention time, peak area, and peak height of the sample were compared with those of the standards for quantization as described in detail by Zira *et al*. (2020). The data collected were analyzed and presented as Mean ±SD andthe level of significance was set at $p<0.005$. The risk level of the concentration of the pesticides was assessed by comparing the obtained data for each sample with the maximum residual limits (MRLs), average daily intake (ADI) rate, and the health risk indices (USEPA, 1996). For the risk characterization,the EADI (mg/kg/day) was estimated by multiplying the pesticides residue concentration in the samples (mg/kg) by the average daily food consumption rate (Cri), the conversion factor (0.085), divide by the bodyweight (Bw) for an adult (60kg) and children (15kg) respectively (Bwatanglang *et al*., 2019).The Hazard Quotient (HQ), an index used in assessing the risk associated with non-carcinogen is obtained by dividing the EDAI values with an established acceptable daily intake (ADI) values set by USEPA (1996). The sum of the hazard quotients (HQ) was used to estimate the health index (HI) that could arise from the consumption of more than one pesticide residues USEPA (1996).

## III. RESULTS AND DISCUSSION:

According to the results presented in figure 2, the only pyrethroid residues detected in the cabbage samples were isomers of cypermethrin (alpha-cypermethrin, theta-cypermethrin, and zeta-cypermethrin). The isomer with the highest mean concentration is zeta-cypermethrin (4.513± 0.84mg/kg) found in Shuwareandthe least is alpha-cypermethrin(0.156±0.01) in a sample from W/Gude. Even though isomers of cypermethrin are classified under non-persistent pesticide, the concentrations found in this study was observed to exceeds the maximum ADI valuesof 0.02 mg /kg set by FAO/WHO in edibles(FAO/WHO, 2013), and significantly above the MRLs of 1.0mg/kg.This implies that the failure of the farmers to observe the withholding periods in the study location puts the consumer at risk from cypermethrin exposure.

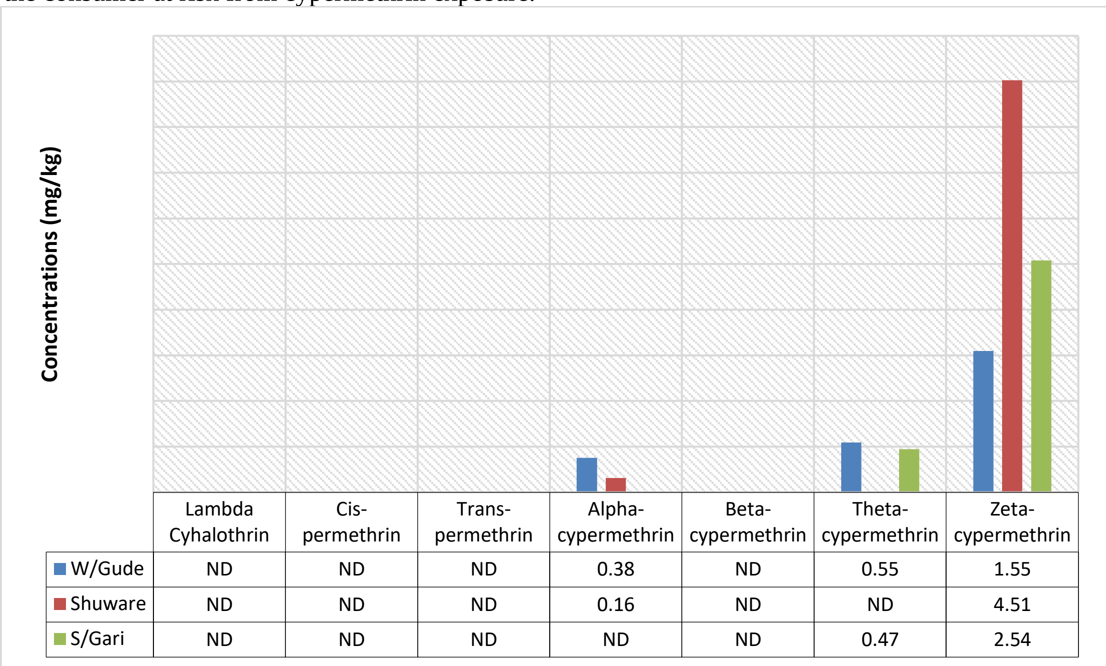
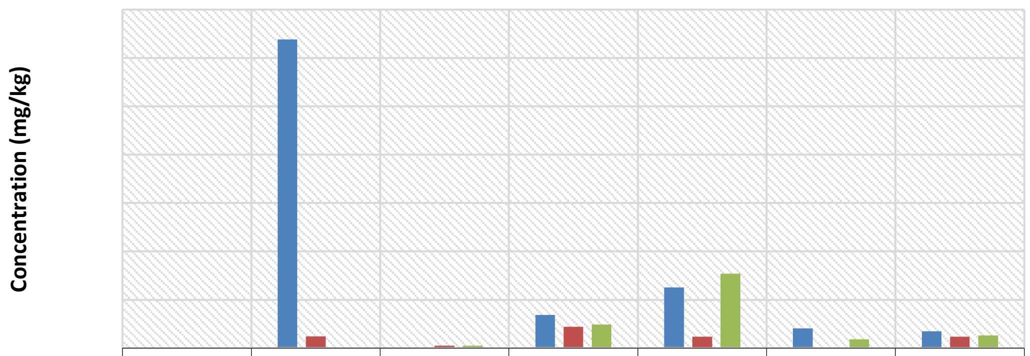



**Figure2. Mean Concentrations of Pyrethroid Pesticide Residues in Cabbage Samples. The results are presented as Mean ±SD of replicate values. ND signified Not-detected**

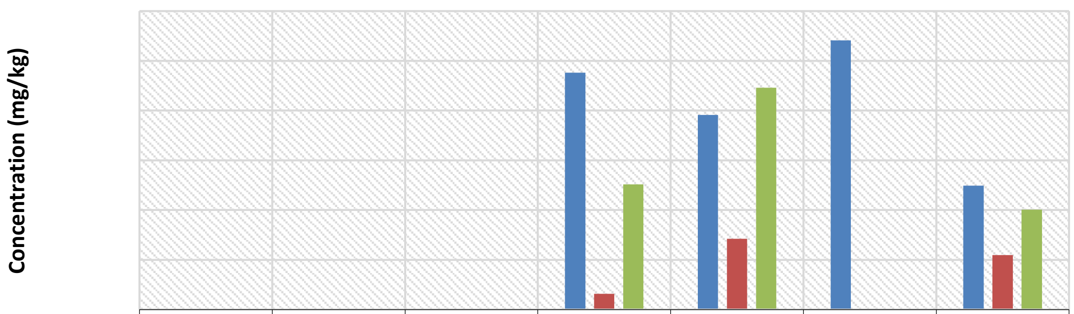
As shown in figure 3, the highest concentration of cis-permethrin in tomato was found in farmlands from Shuware (6.385±0.2 mg/kg) followed by samples from W/Gude(0.240±0.01 mg/kg). Cis-permethrin was detected with a maximum mean concentration of 6.385±0.2mg/kg which was above the MRLs by 6.8times. The concentrations of beta-cypermethrin in the two samples were also above the MRLs. The concentrations of zeta and alpha-cypermethrin were both below their MRLs. In general most of the pesticide residues detected in tomato samples were having concentration above their MRLs. These findings suggest a possible health threat to consumers. The level of contamination found could be linked to improper agricultural practices by farmers or lack of knowledge on the judicious use of pesticides (Barkat*et al*, 2012).




| | Lambda Cyhalothrin | Cis-permethrin | Trans-permethrin | Alpha-cypermethrin | Beta-cypermethrin | Theta-cypermethrin | Zeta-cypermethrin |
|---|---|---|---|---|---|---|---|
| Shuware | ND | 6.39 | ND | 0.69 | 1.26 | 0.41 | 0.35 |
| W/Gude | ND | 0.24 | 0.05 | 0.44 | 0.24 | ND | 0.24 |
| Jairi | ND | ND | 0.05 | 0.49 | 1.54 | 0.18 | 0.26 |

**Figure3. Mean Concentrations of Pesticide Residues in Tomato Samples. The results are presented as Mean ±SD of replicate values. ND signified Not-detected**

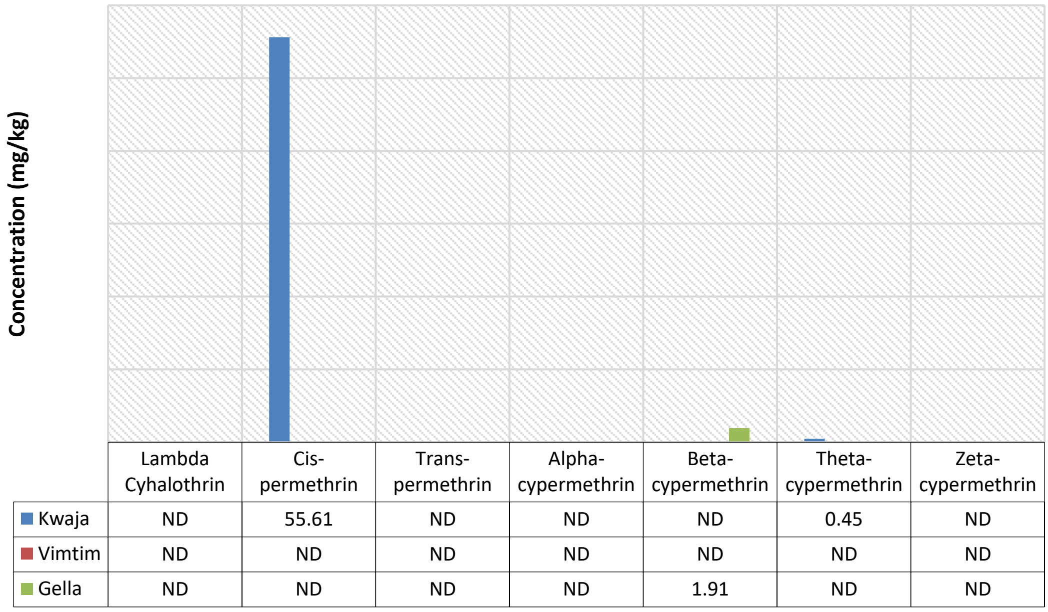
As shown in figure 4, Lambda Cyhalothrin, Cis-permethrin, and Trans-permethrin were all below the detection limits in Garden egg samples from all the sample locations. Among the Pyrethroid pesticide residues analyzed only isomers of cypermethrin (alpha-cypermethrin, beta-cypermethrin, and zeta-cypermethrin) were detected in the samples from the three different farmlands under study.The total mean concentration of Pyrethroid in the garden egg was below that obtained in cabbage and tomato in this study. The detection of numerous pesticide residues in garden egg samples from these farmlands suggests persistent use of these pesticides. The concentrations of all the isomers of cypermethrin in the various samples of the garden egg from the three farmlands were above their MRL except the residue of alpha-cypermethrin detected from SabonGari farmland, which has concentration below the MRL. Generally, the findings revealed that the majority of the pesticide residue was above the MRL. This by implication means they can be potential health hazards to the consumers (Barkat*et al*, 2012).




| | Lambda Cyhalothrin | Cis-permethrin | Trans-permethrin | Alpha-cypermethrin | Beta-cypermethrin | Theta-cypermethrin | Zeta-cypermethrin |
|---|---|---|---|---|---|---|---|
| W/Kondon | ND | ND | ND | 0.95 | 0.78 | 1.08 | 0.50 |
| S/Gari | ND | ND | ND | 0.06 | 0.29 | ND | 0.22 |
| W/Gude | ND | ND | ND | 0.50 | 0.89 | ND | 0.40 |

**Figure 4: Mean Concentrations of Pesticide Residues in Garden egg Samples. The results are presented as Mean ±SD of replicate values. ND signified Not-detected**

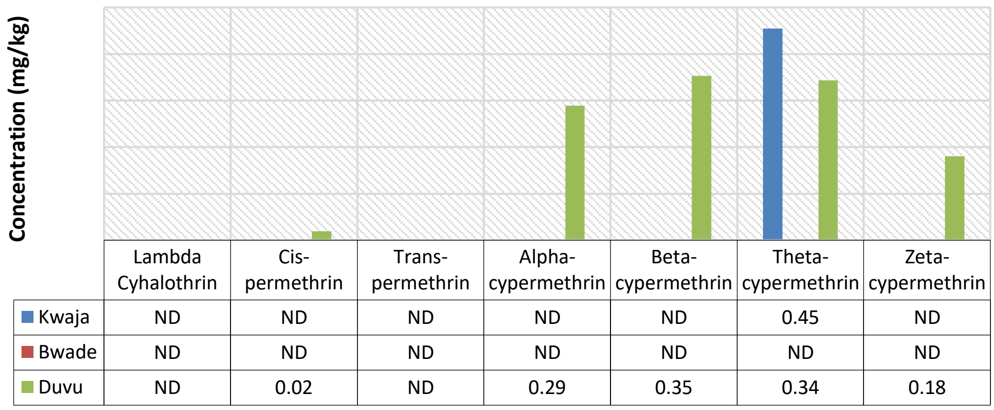
Figure5 shows the levels ofpyrethroidpesticidein Guava samples. From the result, *cis-permethrin*(55.605±5.34 mg/kg), and theta-cypermethrin (0.454±0.05 mg/kg) were detected only in a sample fromKwaja. Similarly,beta-cypermethrinwas detected only in samples from Gella(1.909±0.04 mg/kg). The concentrations of Lambda Cyhalothrin, trans-permethrin, alpha-cypermethrin, and zeta-cypermethrinwereall below the level of detection.The concentration of cis-permethrin detected in guava samples fromKwaja farmland was significantly high compared to its MRL by 5560 times. Both beta and theta cypermethrin have their concentration above their MRL by 45.4 and 190.9 times respectively. The high concentration of the pesticide residues detected in this study more especially cis-permethrin indicates incessant or overuse of the pesticide for killing insects that attacked crops planted around the area.




| | Lambda Cyhalothrin | Cis-permethrin | Trans-permethrin | Alpha-cypermethrin | Beta-cypermethrin | Theta-cypermethrin | Zeta-cypermethrin |
|---|---|---|---|---|---|---|---|
| Kwaja | ND | 55.61 | ND | ND | ND | 0.45 | ND |
| Vimtim | ND | ND | ND | ND | ND | ND | ND |
| Gella | ND | ND | ND | ND | 1.91 | ND | ND |

**Figure5. Mean Concentrations of Pesticide Residues in Guava Samples. The results are presented as Mean ±SD of replicate values. ND signified Not-detected**

In sweet potato, as shown in figure6 only the samples from Duvu shows the presence of most of the pyrethroid pesticides analyzed. Pyrethroid pesticides were not detected in samples in farmlands from Bwade. The concentration of cis-permethrin concentration in the sweet potato samples was below its MRLs. Isomers of cypermethrin including alpha-cypermethrin, beta-cypermethrin, theta-cypermethrin, and zeta-cypermethrin have all their mean concentration above their MRLs. In general, only cis-permethrin pesticide residue did not exceed its MRL in sweet potato samples under study. The traces of pesticides detected may also be linked to the mechanism of transportation of the pesticides in water soil and air (ATSDR, 2013; Watts, 2008).




| | Lambda Cyhalothrin | Cis-permethrin | Trans-permethrin | Alpha-cypermethrin | Beta-cypermethrin | Theta-cypermethrin | Zeta-cypermethrin |
|---|---|---|---|---|---|---|---|
| Kwaja | ND | ND | ND | ND | ND | 0.45 | ND |
| Bwade | ND | ND | ND | ND | ND | ND | ND |
| Duvu | ND | 0.02 | ND | 0.29 | 0.35 | 0.34 | 0.18 |

**Figure6: Mean Concentrations of Pesticide Residues in Sweet Potato Samples. The results are presented as Mean ±SD of replicate values. ND signified Not-detected**

Figure 7 shows the percentage concentrations of pyrethroid pesticideresiduesamples. The results in figure 7a show cypermethrin as the most frequently detected pyrethroid pesticide residue in the cabbage, tomato, garden egg, guava, and sweet potato samples. Detectedin 62% of the samples followed by permethrin, detected in 38% of the samples. In the class of cypermethrin as shown in figure 7b, the isomers that are widely detected in the samples are theta-cypermethrin (24%) with zeta-cypermethrin, alpha-cypermethrin, and beta-cypermethrindetected in 19% of the samples respectively. Cumulatively, the result shows the isomer of cypermethrinwidely detected in all the samples (figure 7c).Constituting about 40% in cabbage samples, 24% in the tomato samples, and 22% in Guava samples respectively. Cis-permethrin was detected only in tomato, guava, and sweet potato samples, while trans-permethrin detected in tomato samples only.Cypermethrin is often used by farmers to control a wide range of insects in field crops, fruits, and vegetables. Ingenbleek*et al.*, (2019) in a separate study investigated the presence of 39 pesticides in Sub-Saharan Africa foodstuff. In the study, the total occurrences ofcypermethrin were detected in 34% of the foodstuff. Similar results documented by Chen *et al*., (2011), detected cypermethrin in 18.7% of fruits and vegetables from Xiamen, China. The survey conducted in Nigeria on the pesticides commonly used by the farmers along the River Ngadda of Maiduguri to include Cypermethrin (73.4%) (Abubakar*et al.*, 2015).And about 60.8%, and 56.2%, of the farmers in Kaduna and Ondo state Nigeria, indicated using majorly chlorpyrifos and cypermethrinin farming (Issa, 2016). The level of cypermethrin detected in this study could be due to the indiscriminate and frequent application of these pyrethroid pesticides by farmers in the study location or the failure by the formers to observethe withholding time between each application or before harvesting (McEwen *et al*.,1980). More so, it could be due to the pesticide retention capacity of the plants analyzed (Li *et al*.,2014; Gad Alla*et al*.,2015).

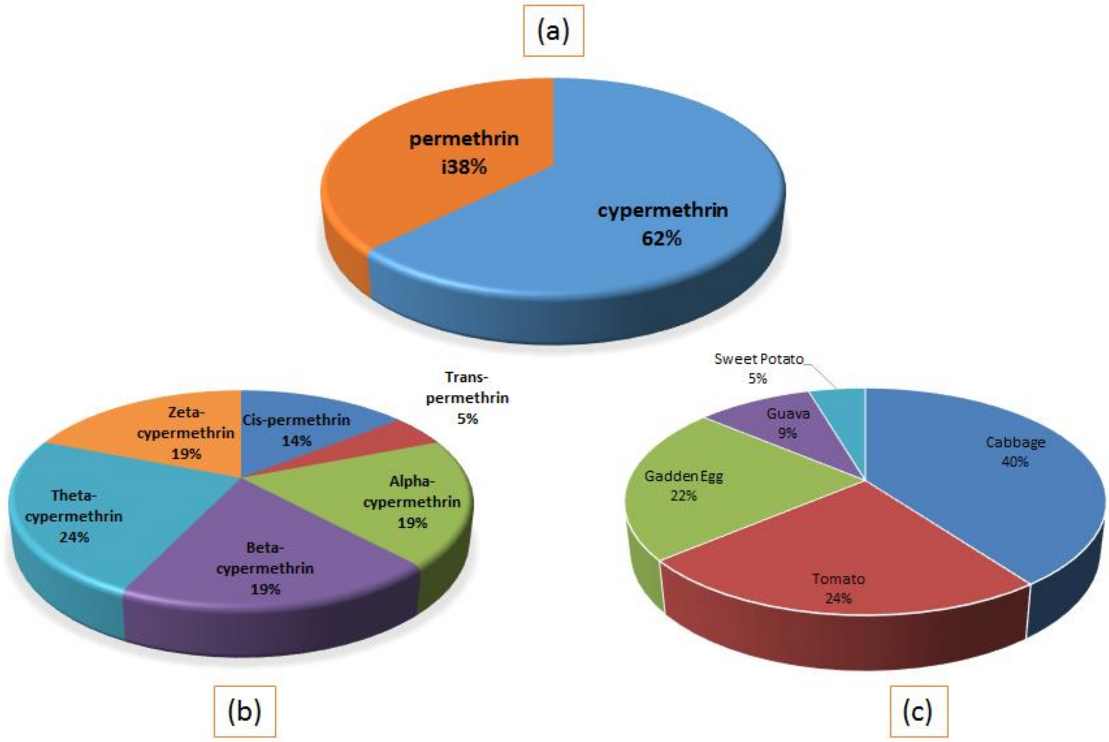



**Figure 7.Showing (a) the percentageconcentration (mg/kg) of pyrethroids (cypermethrin and permethrin)Pesticide Residues detected in the samples, (b) the percentage concentration of the cypermethrin isomers detected, and (c) the percentage concentration of the cypermethrin in the respective samples.**

Based on the verbal communication with the farmers, it was observed that the farmers purchase the pesticide mostly in the open markets. This finding agrees with the survey by Issa, (2016), showing 34.6%, and 32.3% of the farmer's patronizing agro-service centers and open market respectively with just 5.0% patronizing licensed vendors. Similar results were also reported by Mokwunye*et al*., (2012). This of course further put the farmers and the entire food chain risk from adulterated or substandard pesticide products.Besides the indiscriminate application of pesticides by Nigerian farmers without recourse to standard and quality, the survey reported by Abubakar*et al*., (2015) shows out of the 93.8% of farmers that use pesticides in farming, about 85.2% do not apply safety measures during the application, thus increasing the exposure risk (Abubakar*et al*., 2015). Consequently, the frequent and indiscriminate application of the pesticides as mentioned earlier infringe on the right to quality and safe food. Bioaccumulating in the plants and transcends across the trophic level to the food chain. Therefore, understanding the health risk of the pyrethroids will undeniably provide a safety guide to the farmers in the study location. Pyrethroids exert their potency by binding to the protein molecules that regulate the voltage-gated sodium channel (Malhat*et al*.,2016). Thus impairing the normal function of the nerve fibers (Burr and Ray,2004). They are differentiated structurally from each other by the presence of cyano groups. Pyrethroids such as permethrin exert their neurotoxicity by interfering with the sodium channel, while cypermethrin on the other hand due to the cyano group on its structure disrupts voltage-gated sodium or chloride-calcium channels (Burr and Ray,2004). Such interferences alter motor activity and impair cognitive development (Gomez-Gimeriz*et al*., 2017; Imanishi*et al*., 2013). The study by Maurya*et al.,* (2016) supports these findings. In the study, cypermethrinwas observed to induceneuroinflammation and cognitive impairment in young rats. And further observed in another study to increase the level of cytokines (IL-1β) in Xenopus (Martins *et al*., 2010).

The risk assessment conducted in this study is however narrowed to ascertain if the farmers or consumers of food cultivated in the study location are at risk of the health issues discussed above. Based on the results presented in table 1, the EDAI and HQ established for the commonly used Pyrethroids (cypermethrin) were observed to be below the ADI (0.02 mg/kg/day) and HQ<1 estimated for the adults and children respectively. This, however, implies that the consumer on the consumption of the vegetables/crops are unlikely to experience any adverse health hazard (Sun and Chen, 2018).The results were observed to be lower than the findings reported by Elgueta*et al*., (2017). In the study, the maximum EADI obtained for cypermethrin 0.05 mg kg-1, about 0.3-fold, higher than the corresponding ADI values. Similarly, the HQs for cypermethrin obtained in the study is 30.4. Showing a potential health risk associated mainly with methamidophos and cypermethrin. Farming activities often involved the use and application of different pesticides or in some situations mixture of OCPs and the pyrethroidsin other to enhance the efficiency of treatment. Such cocktails of pesticides besides enhancing broad-spectrum insecticidal activities also carry associated health risks of a significant proposition to the public (Ram *et al*., 2011). In our previous study endosulfan was determined as the most frequently used OCPs in the study location (Zira *et al*., 2020) and having established cypermethrin as the commonly used pyrethroids by formers in the same study location, it is, however, obvious to assume that the farmers in the study area use cocktails of a pesticide containing OCPs and pyrethroids. In this case, based on their frequency of use, a mixture ofendosulfan and cypermethrin could suffice. Thus conducting a risk assessment on cocktails of pesticide on the commonly used endosulfan and cypermethrin will help toward establishing the potential health risk from such practices to the public.

For this reason, the HQ values obtained in the previous study for endosulfan (Zira*et al*., 2020) are used to establish the cumulative health risk (HI) associated with it in combination with cypermethrin. The HI<1 established for the mixture of endosulfan and cypermethrin as shown in table 1 signifies that the adult population exposed to the mixture are unlikely to experience any adverse health hazardfollowing the consumption of the plants. However, a level of concern exists, following the consumption of tomato and sweet potato by the children, showing an HI>1, suggesting that the children's populace are more likely at risk for non-carcinogenic related complications due to exposure to endosulfan and cypermethrinin tomato and sweet potato. The unintentional oral ingestion common among children, their slow detoxification processes, and their higher intake rates per unit body weight are factors favoring the observed non-carcinogenic risks in children compared to the adult's populace (Sun and Chen, 2018). The non-carcinogenic risks are expected to be enhanced due to the exposure to the cocktail. The mechanism of action of Cypermethrin and endosulfandiffer in a certain respect.Cypermethrin disrupts voltage-gated sodium or chloride channels while endosulfan inhibits gamma-aminobutyricacidreceptors(Casida, 2009; Costa, 2015). And as such, a mixture of these pesticides will bring about a synergistic effect of both cypermethrin and endosulfan exposure simultaneously (Gomez-Gimenez*et al*., 2018).Raj *et al.* (2013) observed that cocktails of pesticide may have an additive, synergistic, or antagonistic effect. In the study, a combination of endosulfan and cypermethrinwas observed totrigger a sequence of neurotoxicity responses in an animal model.

**Table 1: Hazard characterization of endosulfan and cypermethrinresidues on consumption.**

| | EDAI (mg/kg) | | HQ | | | | HI | |
|---|---|---|---|---|---|---|---|---|
| Plants | Adults | Children | Adults (Cypermethrin) | Children (Cypermethrin) | Adults* (Endosulfan) | Children* (Endosulfan) | Adults | Children |
| Cabbage | 0.005 | 0.013 | 0.2 | 0.7 | 0.1 | 0.3 | 0.4 | 0.9 |
| Tomato | 0.003 | 0.008 | 0.1 | 0.4 | 0.6 | 1.5 | 0.7 | 1.9 |
| Garden egg | 0.003 | 0.007 | 0.1 | 0.4 | 0.2 | 0.5 | 0.3 | 0.9 |
| Guava | 0.001 | 0.003 | 0.1 | 0.2 | 0.3 | 0.8 | 0.4 | 1.0 |
| Sweet potato | 0.001 | 0.002 | 0.0 | 0.1 | 0.4 | 1.1 | 0.4 | 1.2 |

The asterisk '*' Signified data from Zira et al, (2020).

## IV. CONCLUSION:

The findings of this research revealed that the people of Upper Yedzaram Basin were applying a high amount of the pyrethroid pesticides in their farmlands which are mostly above their MRLs concentrations. This may be attributed to the farmers not judiciously using the pesticide. Based on the results of this study, the Government should enforce the routine monitoring of pesticide residues in these study areas to prevent, control, and reduces environmental pollution. The farmers and the inhabitants of the study areas should be educated on the dangers of pesticides for pest control.

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