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# Analysis of Pesticide Residues In Some Vegetables Grown in Gashu'a Fadama Area, Yobe State, Nigeria using High Perfomance Liquid Chromatography (HPLC)

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**ABSTRACT**
The indiscriminate use of pesticides in vegetables farming may leave residues in food which may cause poisoning to human. A method was developed for the determination of some pesticide residues in some selected vegetables; lettuce (Locatuca sativa) and spinach (Spinacia oleracea) samples by high performance liquid chromarography (HPLC). In this study, few pesticide residues were detected in some of the vegetable samples. The detected pesticide residues are Dichlovors, Cypermethrin, Imdacloprid, Firpronil, Dimethoate, and Cyhalothrin. Though the analysis did not determine the levels of these pesticide residue but, the findings from this study is expected to go a long way in providing a baseline data for the assessment of the levels of pesticides residues in vegetables grown in Gashua, Damaturu Fadama farms and farms across Yobe State, in other to safeguard the lives of the consumers.

**Key words:** Pesticides, HPLC, Vegetables, Gashua

## I. INTRODUCTION

Vegetables are essential in human diet component since they provide vital nutrients that are needed for most of the reactions taking place in the body. High intake of vegetables and fruits up to five or more serving in a day has been encourage not only to hinder consequences of deficiency in vitamins but also to lessen the prevalence of major diseases such as cardiovascular diseases, cancer, and obesity (Marina, Helle, & Philippe, 2008). Fruits and vegetables like most crops are confronted by both disease and pest through production and storage leading to damage that reduces their quality and the yield. To reduce the loss and maintain the value of fruits and vegetables harvest, pesticides are used with other techniques of pest management during cropping to destroy pest and prevent disease pollutants produced, by food infecting organisms, however the use of pesticides leaves residues in fruit and vegetables after harvesting (Gelden *et al.,* 2010). These kinds of measure are less labour intensive than other pest control method.

The presence of pesticides residue is of great health concern to human beings because pesticides of potential harmful effects to other non-targeted organisms (consumers) than pest and disease, (Lubomir *et al.,* 2014). The main concerns on human health are their toxic effects include, feotal development, intrude with the reproductive system, and their capacity to cause cancer and asthma, some of the pesticides are persistent and therefore remain in the body causing long term exposure (Gelden *et al.,* 2010). This concern has led to setting up of monitoring systems by national and international regulators to assess safety situations and make informed decisions when passing legislation. These pesticides can be absorbed by the plant surface (waxy cuticle and root surfaces) and enter the plant transport system (systemic) or stay on the surface of the plant (contact), (OECD, 2003).

Crop pesticides can undergo volatilization, photolysis chemical, and microbial degradation

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while still on the surface, all these activities can decrease original pesticides concentration but can also activate some metabolites in the crops, (Celik *et al.*, 1995). Volatilization of the pesticides usually ensues instantly when applied in the field. The volatilization process mainly depends on the velocity of vapour density of the pesticides, pesticides with high vapour pressure usually volatilize rapidly into the atmosphere while those with low vapour pressure stay longer on the surface, (Celik *et al.*, 1995). Volatilization rate also depends on conditions such as location temperature and wind speed. The pesticides will evaporate further as the faster the wind speed and the higher the temperature intensifies. (www.ncbi.nlm.nih.gov/pubmed/19226822).

Photolysis takes place when molecules absorb sunlight energy resulting in pesticide degradation. The indirect reaction can also be triggered by some other chemicals being broken by the sunlight and their product reacting with pesticide, and use the pesticides as nutrients thereby breaking them into carbon dioxide and other component (Holland and Sinclair2004). Although degradation of pesticides is influence by different environmental field condition, volatilization remains process that affects pesticide.

Lettuce (Lactuca sativa) is the Danish family annual plant of Asteracae L.Scriola var.integrate (Gren. and Godr). It is not only grown as a leaf vegetable but in some cases for its stems and seeds. Lettuce was said to be cultivated first by the ancient people of Egypt who turned from a Weed 420BC, its seeds were used oil in production; later as food plant grown for its succulent leaves, in addition to its oil-rich seeds. Lettuce extends to the Greeks and Romans, the latter of whom gave it the name ‘’Lactuca’’, from which the English name “Lettuce” is eventually derived. By 50AD, multiple types were described, and lettuce appeared often in medieval writings, including several herbals. The 16th through 18th centuries saw the development of many lettuce varieties in Europe, and by the mid-18th century cultivars were described that can still found in gardens. Europe and North America originally dominated the market for lettuce, but by the late 1900s, the consumption of lettuce has spread through the world.

Spinach (Spinachia Oleracea) is one of the edible flowering plants in the Amaranthalege family. It originated from southwestern and central Asia. It is an annual plant (rarely biennial) that grows up to 3cm height. Spinach can survive over winter temperature areas. The leaves are alternate, simple ovate to triangular, very variable in size from about 2-30cm long and 1-15cm broad, with larger leaves at the base of the plant and small leaves higher on the flowering stem. The flowers are inconspicuous, yellow, green, 3-4cm diametre, maturing into small, hard, dry, humpy fruit cluster 5-10cm across, containing several seeds.

Vegetables play a significant role in human nutrition intake. They are mostly low in fat and calories but are bulky and filling. They supply dietary fibre and are vital for essential vitamins, minerals, and trace elements. Particularly important are the antioxidant vitamins A, C and E. When vegetables are incorporated in the diet, is found to be a reduction in the incidence of stroke, cancer, cardiovascular diseases, and other related chronic diseases (Terry Leon, 2011). Vegetables are consumed with various combinations; as part of main meals and as snacks.

The use of pesticides have increased in recent years due to their ability to protect against infecting organism and are less labour intensive relatively to other pest control techniques. However, the application of pesticides during production brings about the presence of pest residues in the harvested crops is of health concern to consumers because, pesticides are identified to have possible harmful effects on both animal and human health. This study tends to determines the presence of pesticide residue in Spinach (Amaranthus) and Lettuce (Lactuca Sativa) which are vegetables grown in Gashu’a.

**Study Area**

Gashua, the headquarters of Bade Local Government is among the most developed towns in Yobe State, Nigeria. The town is also among the major economically and ecologically important towns in the state (Neiland and. Ladu 2010). Most vegetables, rice and other food crops produces in Yobe State comes from Gashua; and mostly through Irrigation farming. According to International Centre for Investigative Reporting (ICIR) report (ICIR 2022), and other hospital reports within Yobe State, Nigeria, no less than 150 residents of Gashua, Yobe State, die of kidney disease yearly. According to the report, many residents die of chronic kidney failures because they preserve the foods they eat with chemicals, while many farmers in the town use banned herbicides on their crops”. (Marcus, 2022)

This study focused on the analysis of pesticide residues in some selected vegetables samples in order to determine their presence or otherwise using HPLC.

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## II. MATERIALS AND METHODS

**Sample collection, preparation, treatment and pesticide analysis**

Vegetable samples were collected from three different Fadama Sites in Gashu'a namely: X, Y, Z and Damaturu Market. The vegetables collected include; Lactuca sativa (lettuce), and spinach (Spinachia Oleracea).

The Samples were washed with distilled water to eliminate the pollutants; the leaves were separated from the stem and dried using drying cabinet. The dried samples were smashed into fine powder using porcelain pestle and mortar. 5g of each sample was weighed in different vessels before analysis. 10 ml of acetone was added and ultrasonically extracted for 20 mins. The sample was centrifuge for 20 mins at 8,000rpm and allowed to stand overnight and then the supernatant was collected in different vessels. A mixture of 10ml of petroleum ether and 10ml of dichloromethane was added for and votex mixed for 1 mins. The sample mixture was filtered with Whatman No 1 filter paper through funnel containing Anhydrous Sodium Sulphate and Activated Charcoal. The filtrate was kept in an incubator for 24 hours to evaporate; finally, the extract was collected in a vials and 1ml of methanol was added for analysis. This method was adopted by (Chang et al 2005).

Pesticides residues content of the sample were determined by the use of HPLC Machine

Table 3.1 Chemicals/Reagents

| S/No. | Chemicals | Company |
|---|---|---|
| 1 | Acetone 1 | Lob Chemie Pvt, India |
| 2 | Petroleum ether | Guangding Guanhau Sci-Tech Co. Ltd, China |
| 3 | Dichloromethane | Guangdong Guanhua Sci-Tech Co. Ltd, China |
| 4 | Anhydrous Sodium Sulfate | LobaChemie Pvt Ltd, India V |
| 5 | Activated Charcoal | BDH Laboratory Supplies Poole, BHIS, Ltd, England |

Table 3.2 Operating Parameters

| S/No. | Operating Parameters |
|---|---|
| 1 | HPLC Agilent 1200 Infinity |
| 2 | Column Agilent ODS C18, 2.1 X 100 mm, 1.8 pm |

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| | |
|---|---|
| 3 | Guard Column C18, 4 X 3.0 mm |
| 4 | Column Temperature 30°C |
| 5 | Mobile Phase A: 0.1% formic acid in water |
| 6 | Mobile Phase B: 0.1% formic acid in methanol |
| 7 | Flow rate: 1 mL/min, injection volume - 100 µL |
| 8 | Gradient run: initial 5% B, then 10 min 95% B, then 5 min 100% B |
| 9 | Run time: 15 min |

**Results and Discussion**

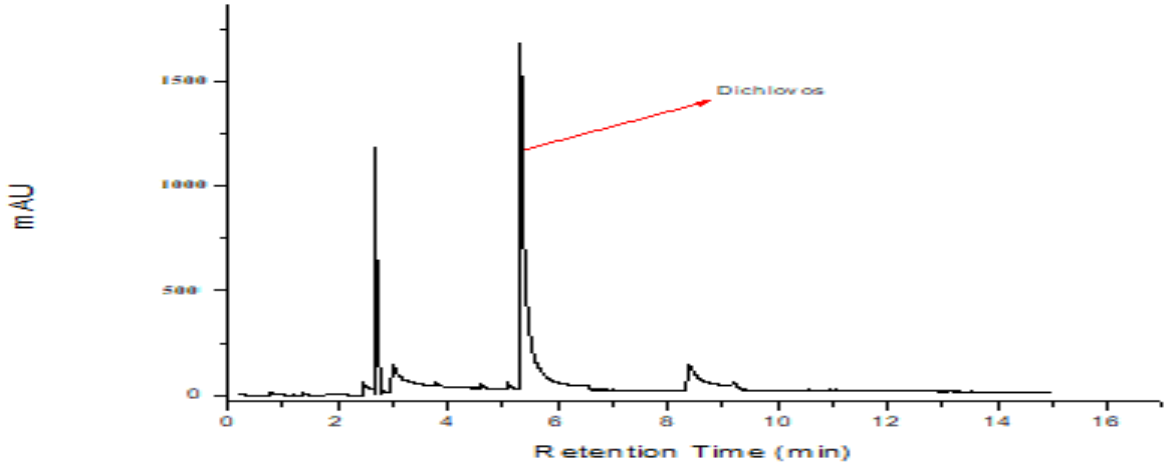


Figure 4.1: Shows the peak of Dichlovors in spinach sample of Mashangwari

ZI1D= (Dried Spinach sample (spinacia oleracea)), Zl2F= (Fresh Spinach sample inacia ~oleracea)), Yl1D= (Dried Spinach sample (spinacia oleracea) from ifferent farm), Xl2F=_(Fresh Spinach sample (spinacia oleracea) of another farm), LGF1= (Fresh Lettuce sample (Lactuca sativa)), LGD;= Qfiried Lettuce sample), Lactuca sativa.

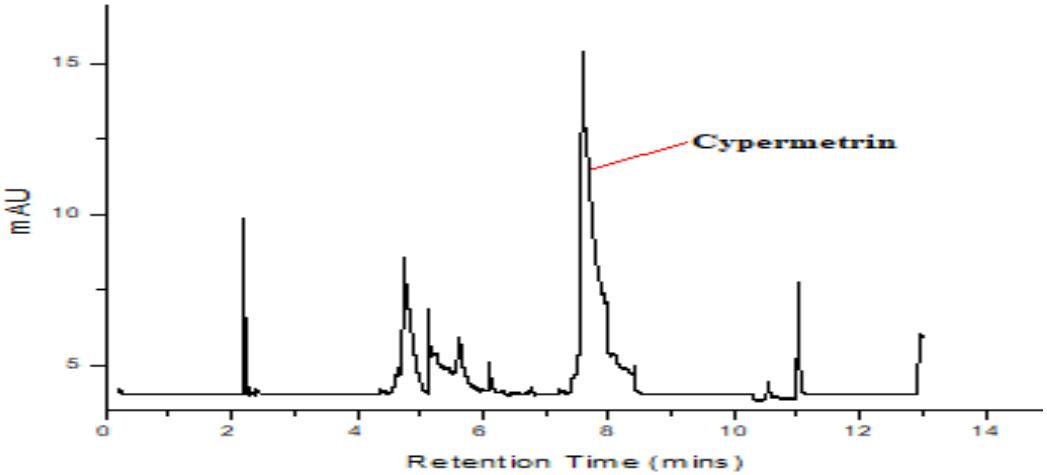


Figure 4.2 shows the peak of Cypermethrin in dried Spinacia oleracea (Spinach) sample of Gonan Babadan Atta at a retention time (7.39)

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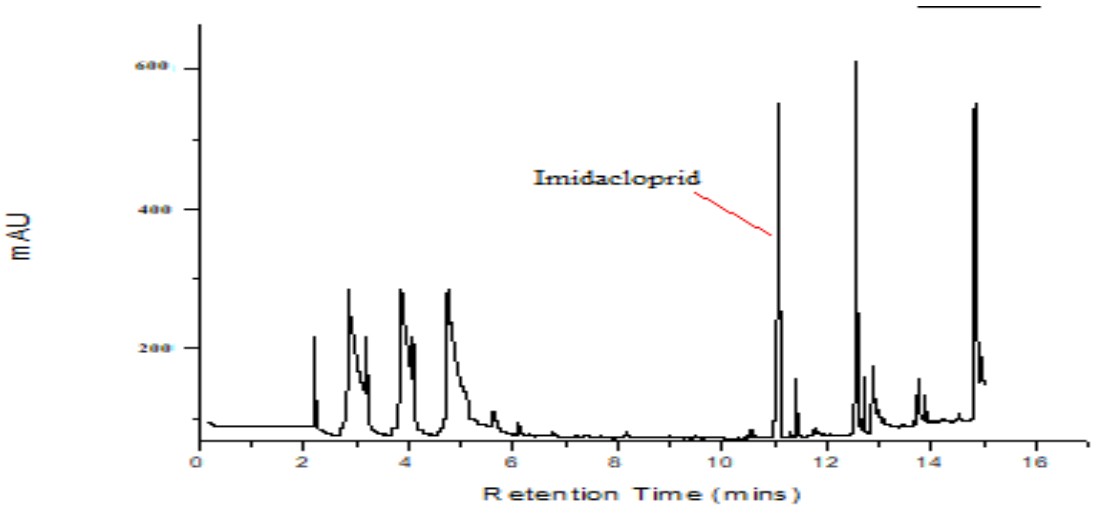


Figures 4.3 shows the peak of Imdacloprid in fresh Lactuca sativa (Lettuce) of Tsamiya Uku at a retention time (l1.0min).

Some pesticide residues and other unknown compounds were detected in some of the vegetable samples; while in some samples, no pesticide residues were discovered. A reversed-phase column was applied for simultaneous determination, reducing the solvent volume and the measurement period and achieving high seperativity.

Dichlovors, Cypermethrin, Imidacloprid were detected in spinach and lettuce samples using HPLC after extraction with acetone. The identification was based on retention period matching of the analytes in the sample with that of pesticides standards. The chromatograms of the detected pesticides are shown in figure 4.1-4.3.

Figure 4.1 shows the peak of Dichlovors in fresh Spinaeia oleracea (Spinach) sample from Mashingwari at a retention time (5.32min). Figure 4.2 shows the peak of Cypermethrin in dried Spinacia oleracea (Spinach) sample of Gonan Babadan Atta at a retention time (7.53min), while Figures 4.3 shows the peak of Imdacloprid in fresh Lactuca sativa (Lettuce) of Tsamiya Uku at a retention time (l1.0min).

Nothing was detected in most of the samples and this might be due to the fact that the samples were collected during the raining season. It is expected that most of the residues were washed away by rain. More so, some pesticide residues were not detected due to lack of standards.

Table 4.1: Showed some pesticide residues detected in spinach and lettuce samples from Gashua.

| S/No. | Retention Time (mins) | Pesticides | |
|---|---|---|---|
| 1 | 5.32 | Dichlovos | |
| 2 | 7.53 | Cypermetrin | |
| 3 | 11.00 | Imidacloprid | |

The peak of Dichlorvos in spinach sample at a retention time (5.32 min) and peak of Imidachloprid with retention time (11.0 min). The results obtained in this study were in agreement the ones reported by (Christos A. Damalas et al 2011).

CYPERMETHRIN

The peak of Cypermethrin identified in dried Spinacia oleracea (Spinach) sample of Gonan Baba dan Atta at a retention time (7.53min), is similar to What was reported by (Cesnik *et al.*, 2001-2004).

IMDACLOPRID

The peak of Imidacloprid shows in Figure 4.5 in fresh Lactuca sativa (Lettuce) at a retention times (11.0), 10mins time. Some of the results obtained in this study were also reported by (Aisha Sale Makinta, 2015).

## III. CONCLUSION

The results of this study discovered the presence of some pesticide residues in fresh and dried sample of Spinacia oleracea (Spinach) and that of fresh Lactuca sativa (Lettuce). These vegetables are high in demand in Gashua areas and

Damaturu as it is serving as portion of daily staple food. Frequent consumption of these vegetables will certainly result in health risks. The result obtained from this study is expected to go a long way and contribute in providing a baseline data for the assessment of the levels of pesticide residues in vegetables grown in Gashua, Damaturu Fadama farms and other farms across Yobe State, in order to safeguard the lives of the consumers. Due to health problems associated with these pesticides, there is the need to continually examine the levels of pesticide in foodstuffs in and around Yobe State in order to maintain and/or improve measures to reduce their levels in foodstuffs and ultimately prevent these avoidable health problems. Further studies can be done to ascertain what types of chemical products are the most befitting tools for environmentally based pest management. To discover the most promising opportunities to expand benefits and condense health and environmentally associated risks of pesticide treatment. A proper role for the public sector in research, product development, product testing and registration, implementation of pesticide application strategies, and public awareness about effects pesticide residues is also recommended.

## REFERENCE

[1]. A.E. Neiland, B.M.B. Ladu. "Enhancement of inland fisheries in Nigeria: the institutional context provided by traditional and modern systems of fisheries management". Food and Agriculture Organization.

[2]. Celik, S., Kunc, S. And Asan T. (1995). Degradation of some pesticide in the field and effect of processing. Analyst, 120,1739-1743.

[3]. Christos A, Damalas, and Illias G. Eleftherohorinos. (2011). "Pesticides Explosure, Safety Issues, and Risk Assessment Indicators". International Journal of Enviromnental Research and Public Health. 8(5): 1402 19. Doi: 10.3390/ijerph8051402. PMC 3108117. PMID 21655127. Line feed character in journal=at position 25.

[4]. Finotti, Enrico; Bertone, Aldon; Vivanti, Vittorio (2006). "Balance between nutrients and anti-nutrients in nine Italian potato cultivars". Food chemistry 99 (4): 698. Doi: 10.1016/j.foodchem.2005.08.046.

[5]. Garg & Prakash, Garg H.P., (2000). Solar Energy: Fundamentals and Applications.Tata McGraw-Hill Education. P.191 .ISBN 978-0-07-463631-2.

[6]. Gilden, R.C., Huffling, K., Sattler, B. (January 2010). "Pesticides and health risk". J Obstet Gynecol Neonatal Nurs 39(1): 103-10.

[7]. Gruda, N. (2015)."Impact of environmental factors on product quality of greenhouse vegetables for fresh consumption." Critical Reviews in plant Sciences 24 (3):227-247. Doi:10.1080/O7352680591008628.

[8]. Holgate MW (1976): A prespective of environmental pollution, Cambridge University Press, Cambridge. Pp.55-65.

[9]. Holland, J. & Sinclair, P.(2004). Environmental fate of pesticides and the consequences for residues in food and drinking water, in: "Pesticide Residues in Food and Drinking Water: Human Exposure and Risks, Hamilton D. & Crossley S. (Ed.), 27-62, John Willey &Sons LTD, 0-471-48991-3, West Sussex, England.

[10]. IUPAC, Compendium of Chemical Terminology, 2nd ed. (the "Gold book") (1997). Online corrected version: (2006) "pesticide residue".

[11]. Jane Thamson. 'Ayton, Charles John,' from the Dictionary of New Zealand Biography. Te Ara-the Encyclopedia of New Zealand, update 4-Jun-2013 URL:http//www.TeAra.govt.nz/en/biographies/2a19/ayton-charles-john.

[12]. Lobe, J. (Sept 16, 2006), "WHO urges DDT for malaria control Strategies," lnter Press Service, cited from Commondreams.org Retrieved on September 15, 2007.

[13]. Marcus, F. (2022). Kidney Failure Kills 150 Yearly in Yobe Town. Leadership June 2022

[14]. Marina Bjorling-Poulsen, Helle Raun Andersen and Philippe Grandjean. (2008). "Potential developmental neurotoxicity of pesticides used in Europe". Environmental Health. 7:50:50.doi:10.1186/1476-069X-7-50. PMC 2577708. PMID 18945337

[15]. OECD, (2003), Description of selected key generic terms used chemical hazard/risk assessment. OECD Series on Testing Assessment, No.44

[16]. OECD, (2003), Turning Science into Business: Patenting and Licensing at Public Research Organizations, Paris: OECD.

[17]. Porcher, Michael H. (2005). "Sorting Lactuca Names". Multilingual Multiscript Plant Name Database. University of Melbourne. Retrieved 2 April 2012

[18]. Rickman Joy C., Bruhn Christine M., Barrett Diane M.,(2007). "Nutritional comparison of fresh, frozen, and canned fruits and vegetables II. Vitamin A and carotenoids, vitamin E, minerals and fiber". Journal of the Science of Food and Agriculture 87 (7): 1185-1196.doi:10.1002/jsfa.2

[19]. Stephen W.C Chung, Benedict L.S Chen. (2011). "Determination of organochlorine pesticide residues in fatty foods: A critical review on the analytical methods and their testing capabilities". Journal of Chromatography A. 1218(33):5555-5567.DOI:10.1016/J.chroma.2011.06.066. PMID 21742333.

[20]. Swedenborg, Emanuel (2003). Swendenborg Concordance 1888. Kessinger Publishing. P. 502. ISBN 0-7661-3728-7.

[21]. Terry, Leon (2011). Health-Promoting Properties of Fruits and Vegetables. CABI. Pp.2-4. ISBN 978-1-84593-529-0.

[22]. US Enviromental (July 24, 2007), What is a pesticide? Epa.gov.Retrieve on October 24, 2012.

[23]. Victor R. Boswell, "Garden Peas and Spinach from the Middle East". Reparint of "Our Vegetable Travelers" National Geographic Magazine, Vol 96:2 (Aug 1949) (www.Aggie Horticulture. Accessed 03/07/2010). Aggie Horticulture.

[24]. Walter J Crinnion. (2009). "Chlorinated Pesticides: Threats to health and Importance of Detection". Environmental Medicine. 14(4):347-555555559 PMID 20030461.

[25]. Waxman, Michael F., (1998) Application Equipment. In: Agrochemical and Pesticide Safety Handbook Ed. M. Wilson. CRC Press, Boca Raton (ISBN 978-1-56670-296-6) pp. 326.

[26]. Winter, C.K. and J .M. Katz. (2011). Dietary exposure to pesticide residues from commodities alleged to contain the highest contamination levels. Journal of Toxicology. doi:10.1155/2011/589674. http://www.ncbi.nlm.nih.gov/pmc/articles/PMC3135239/.

[27]. Y.J. Chang, Tseng, S.H., Chous, S.S (2005). Detection of dintramine residual in dry beans by high speed gas chromatography. Annual Scientific Report of BFDA, Department of Health, Executive Yuan. 22:129-128.