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Assessment of Pesticide Residue Levels in Vegetables, Soil, and Water from Selected Subdivisions in the Northwest and West Regions of Cameroon

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Abstract

The assessment of pesticide residues in agricultural commodities is a critical component of global food safety and public health. Even though pesticides are essential for maintaining high crop yields and meeting the demands of a growing global population, their persistence in the environment and presence in the human diet pose significant toxicological risks. This study evaluated consumer safety by analyzing pesticide residue levels in vegetables, soil, and water collected from matched agricultural sites. It assessed risks by comparing detected concentrations against established Maximum Residue Limits (MRLs). Twenty-four vegetable samples, six soil samples, and six water samples were analyzed. The Multi-Residue QuECHERS Buffered AOAC 2007.01 method was used for sample analysis. The results showed the presence of pesticide residues in 16.7% of the vegetables, with chlorpyrifos-methyl and chlorothalonil being the detected compounds. After quantification, the concentrations of chlorpyrifos-methyl and chlorothalonil in celery from Foumbot (2.5 mg/kg) and Babadjou (62.1 mg/kg) exceeded their respective Codex Maximum Residue Limits (MRLs) of 0.05 mg/kg and 20 mg/kg respectively. Furthermore, phenol-2,4-bis (1,1-dimethylethyl), was detected in all soil samples. No residue was detected in the water samples. The study revealed a critical issue of pesticide residue exceedance in vegetables, posing a direct food safety risk. The soil contamination with phenol-2,4-bis (1,1-dimethylethyl) points to a separate environmental concern, suggesting diverse sources of pollution in the regions.

Keywords

Vegetable, Contamination, Pesticide Residues, Analysis, Risk

1. Introduction

The Western Highlands of Cameroon, characterized by fertile volcanic soils (andosols and ferrosols) and favorable microclimates, serve as the "breadbasket" for much of Central Africa, producing high volumes of Irish potatoes, tomatoes, celery, and carrots [1]-[3]. The intensification of agricultural production in this region has led to a significant reliance on synthetic pesticides to meet the food demands of an expanding population [1] [2] [4] [5]. Farmers frequently mixed different classes of chemicals, sometimes exceeding recommended dosages [6] [7]. The indiscriminate application of pesticides by farmers has raised critical concerns regarding toxic residues in the food chain and the environment [4] [5]. Limited data on specific pesticide residue concentrations in local vegetables creates a safety gap, despite WHO estimates indicating significant annual poisoning cases [8]. This study aims to provide a comprehensive assessment of the current pesticide burden in six strategic locations within the Western Highlands of Cameroon. It quantifies residue levels in specific crops (*Daucus carota*, *Solanium lycopersicum*, *Solanum tuberosum*, and *Apium graveolens*) to provide vital data for regulatory bodies to enforce Maximum Residue Limits (MRLs).

2. Materials and Methods

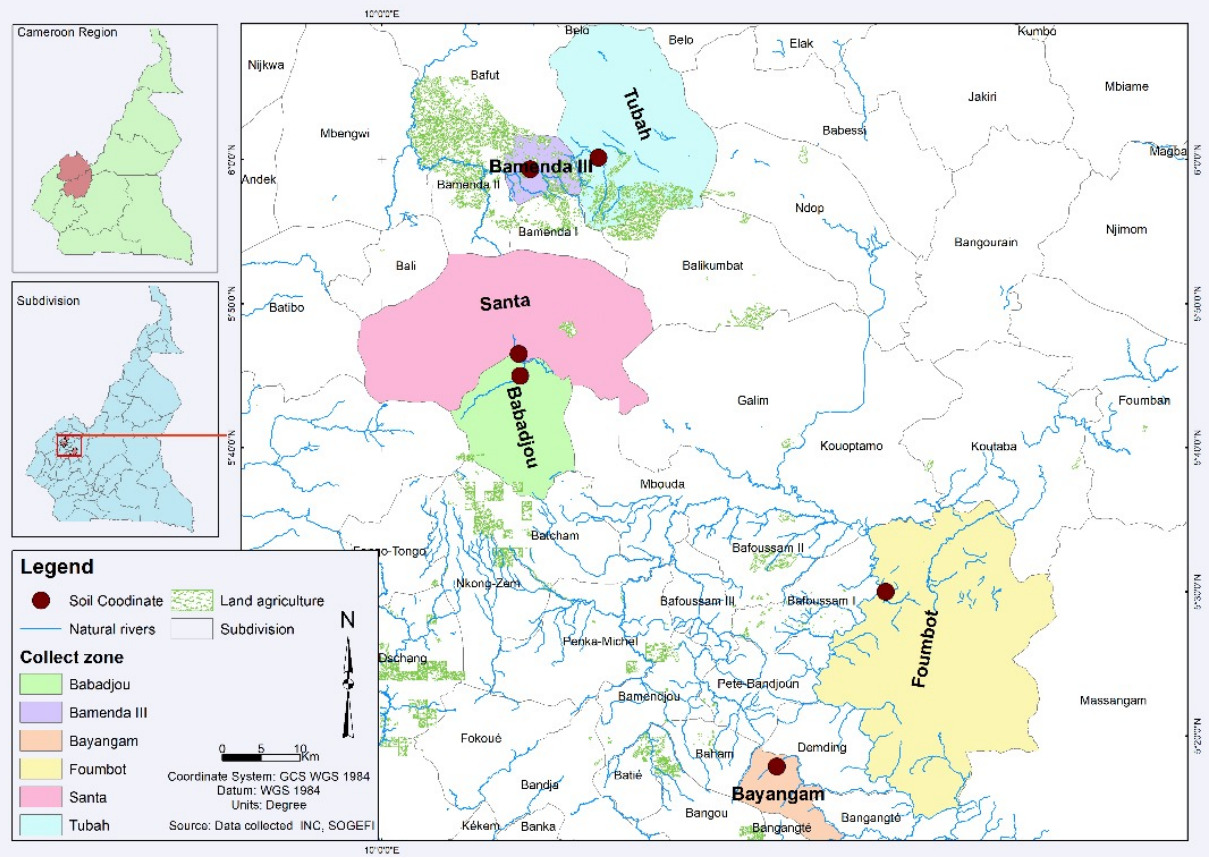
2.1. Description of the Study Area

The Northwest and West Regions of Cameroon fall under the Western Highlands' Ecological Zone, also known as the Grass Field Zone. These locations are situated within the Western High Plateau, an area defined by altitudes ranging from 1,000 to over 2500 meters above sea level [3] [9]. This zone is characterized by high relief, cool temperatures, heavy rainfall and Savanna vegetation [10]. It consists of mountain ranges and volcanoes made of crystalline and igneous rocks. This zone is boarded by the South Cameroon Plateau to the Southeast, the Adamawa Plateau to the Northeast and the Cameroon Coastal plain to the South. The map of the western Highlands is shown in **Figure 1**.

2.2. Sample Collection and Preservation

Vegetables: A total of twenty-four vegetable samples, comprising *Daucus carota* (6), *Solanium lycopersicum* (6), *Solanum tuberosum* (6), and *Apium graveolens* (6), were procured from six distinct agricultural sites: Tubah, Bamenda III, Santa, Babadjou, Foumbot, and Banyangam. At each location, one kilogram of each vegetable species was harvested from two independent farmers and subsequently pooled to create a representative composite sample. From these composites, 500

g aliquots were sealed in Ziploc polyethylene bags, labeled, and transported under refrigerated conditions (4˚C) to the Directorate of Regulations and Quality Control (DRCQ) laboratory in Yaoundé, Cameroon. Upon receipt, all samples were inspected, coded, and stored at 4˚C to maintain analytical stability.



Source: Adapted from NIC (2023). Map of the Western Highlands of Cameroon. Yaoundé: National Institute of Cartography.

Figure 1. The Map of the Western Highlands.

Soil: To ensure a representative assessment of soil characteristics, six composite samples were collected across the study area. At each site, topsoil (0 - 15 cm depth) was extracted from eight distinct locations using a clean soil auger. These eight increments were thoroughly homogenized to form a single composite. From each composite, a 500 g subsample was weighed, transferred to a sterile container, and immediately stored in a portable cooler at approximately 4˚C to minimize microbial activity and chemical degradation during transport.

Water: Six water samples were collected using 1.5 L high-density polythene containers. To prevent cross-contamination, each vessel was rinsed three times with the source water prior to final collection. Samples were obtained by gently immersing the container below the surface, ensuring sufficient headspace remained for subsequent laboratory homogenization. Immediately following collection, samples were stored in a portable cooler maintained at 4˚C to inhibit micro-

bial activity and chemical degradation. This protocol was replicated across all designated sampling sites, and samples were transported to the laboratory for immediate processing and preservation.

2.3. Analytical Technique

The samples were analyzed at the Directorate of Regulations and Quality Control (DRCQ) of the Ministry of Agriculture and Rural Development (MINADER) in Yaoundé, Cameroon using the Multi-Residue QuEChERS AOAC official 2007.01 method. Extraction was performed by adding acetonitrile to each homogenized sample. The extract was purified using a dispersive solid-phase extraction (d-SPE) cleanup, employing anhydrous magnesium sulphate ($MgSO_4$) to induce phase separation by removing water, sodium acetate (CH_3COONa) to maintain an optimal pH buffer, and Primary/Secondary Amine (PSA) to remove polar matrix interferences, such as organic acids and sugars. Octadecylsilane (C_{18}) was also added to remove lipids and non-polar interferences.

The pure extract was analyzed using an Agilent 7890A Gas Chromatograph coupled with an Agilent 5975C VL Mass Selective Detector (MSD) (Agilent Technologies, Palo Alto, CA, USA). Separation was achieved using a HP-5MS capillary column (30 m × 0.250 mm inner diameter, 0.25 μm film thickness) (Agilent Technologies). The GC-MS system was operated in electronic impact (EI) ionization mode (70 eV) with Single Ion Monitoring (SIM) acquisition.

Helium was employed as the carrier gas at a flow rate of 1.0 - 1.2 mL/min. A 1.5 μl sample volume was injected in split less mode, with the inlet temperature maintained at 280˚C. The GC oven temperature program was initiated at 150˚C (held for 1 min), increased to 260˚C at a rate of 45˚C/min, and finally ramped to 280˚C at a rate of 20˚C/min, holding for 9 min, resulting in a total run time of 13.44 min. The MS heat transfer line temperature was set to 250˚C. Chromatograms displaying pesticide retention times and abundances were produced, allowing for identification by matching observed retention times with the instrument's database.

2.4. Method Validation

The analytical method was validated according to EU SANTE/11312/2021 guideline for specificity, linearity, accuracy, precision and sensitivity. The method demonstrated robust performance, with recoveries consistently ranging from 70% - 120% and relative standard deviations (RSDs) within 20% across the validated range. Limits of detection (LOD) and quantification (LOQ) were established to ensure compliance with regulatory Maximum Residue Limits (MRLs), with the LOQ set at 0.01 mg/kg.

2.5. Quantification of Pesticide Residues

The concentrations of the detected pesticides in the extracts were determined using standard calibration solutions prepared from analytical-grade pesticide standards. The calibration curves demonstrated high linearity with a correlation coef-

ficient greater than 0.9950 ($R^2 > 0.9950$). The calibration graphs are shown in Figure 2 and Figure 3.

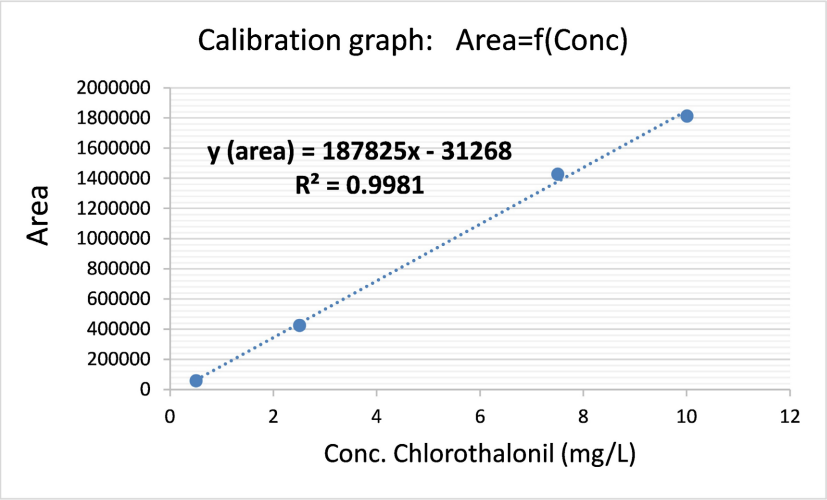


Figure 2. Calibration graph of chlorothalonil.

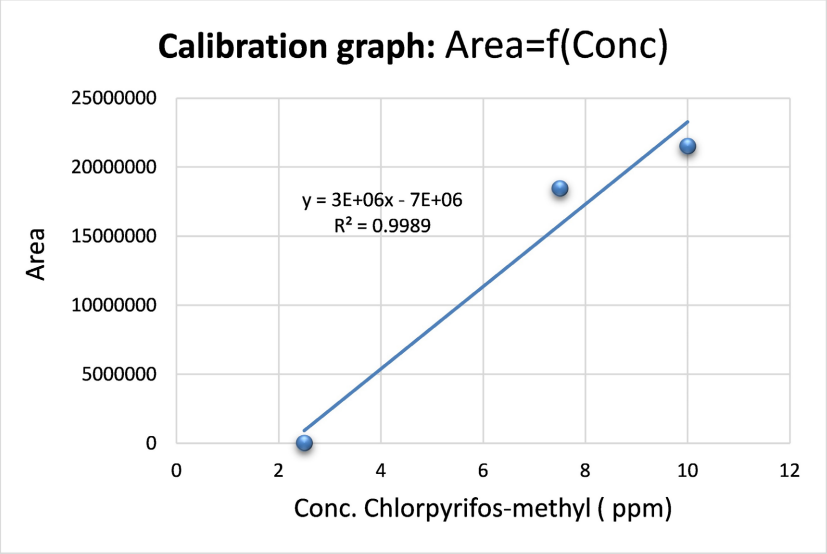


Figure 3. Calibration graph of chlorpyrifos-methyl.

Concentrations obtained from the calibration graphs were substituted into Equation (1) to determine the corresponding pesticide residue concentrations in the vegetables. These results were then compared with MRLs to determine the level of contamination

$$\text{Concentration of pesticide residue}(\text{mg/kg}) = \frac{\text{Conc. of sample}(\text{mg/L}) \text{ in extract} \times \text{Volume of extract}(\text{L})}{\text{Weight of sample}(\text{kg})} \quad (1)$$

The concentrations were compared with Maximum Residue limits (MRLs) to determine the level of contamination.

3. Results

The result of the analysis of the samples and the quantification of pesticide residues is presented in chromatograms and tables.

3.1. Chromatograms of Detected Pesticides

Sample chromatograms showing the detected pesticides residues in the samples are presented in Figures 4-7.

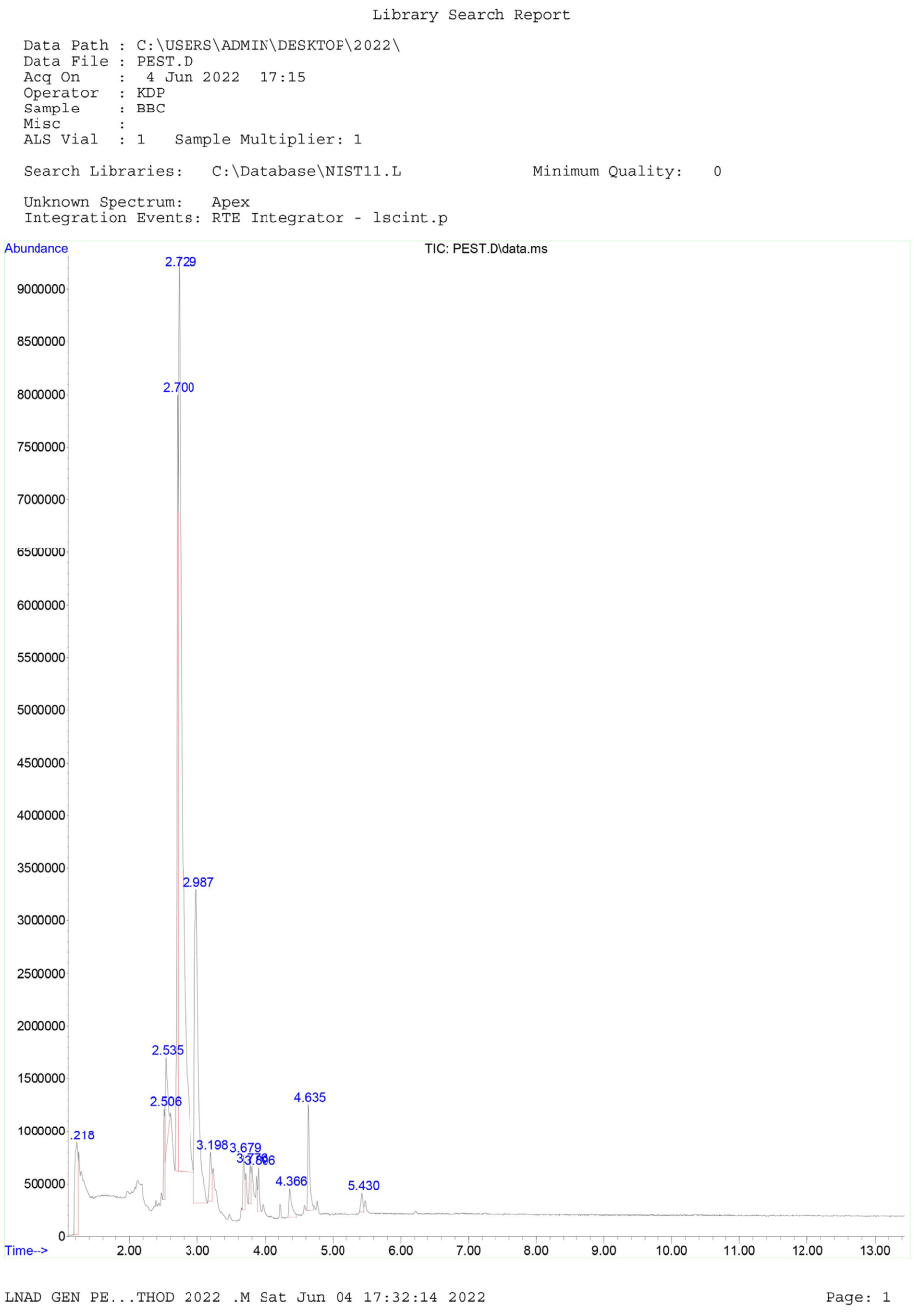


Figure 4. Chromatogram of celery sample from Babadjou, containing chlorothalonil with a retention time of 2.987 minutes.

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                                    Library Search Report

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Data File : PEST.D
Acq On    :  6 Jun 2022  13:08
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Sample    : NC
Misc      :
ALS Vial  : 1   Sample Multiplier: 1

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Unknown Spectrum:   Apex
Integration Events: RTE Integrator - lscint.p
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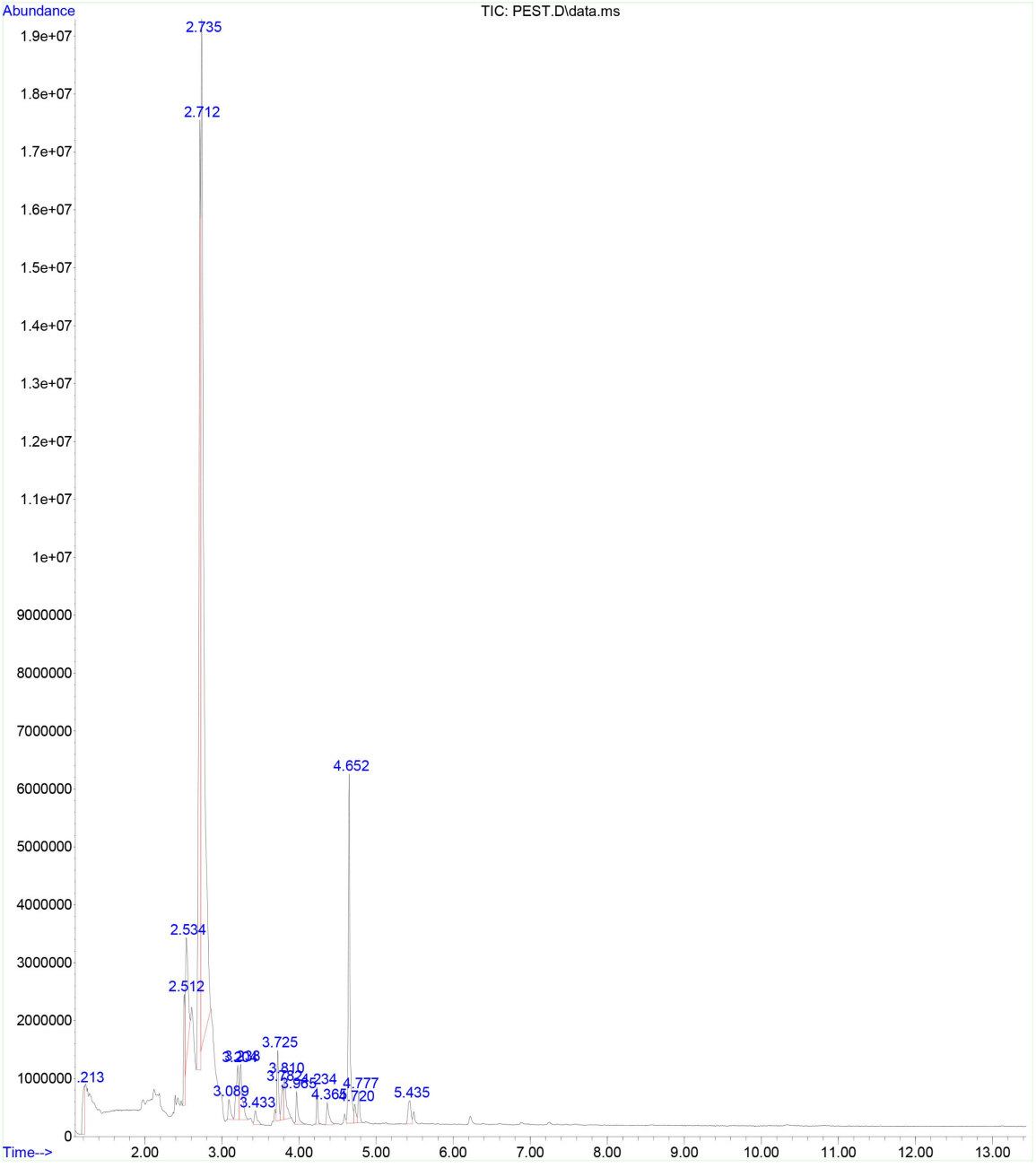


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Figure 5. Chromatogram of celery sample from Foumbot, containing chlorpyrifos-methyl with a retention time of 3.433 minute.

Library Search Report

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Data File : PEST.D
Acq On : 4 Jun 2022 17:55
Operator : KDP
Sample : B3C
Misc :
ALS Vial : 1 Sample Multiplier: 1

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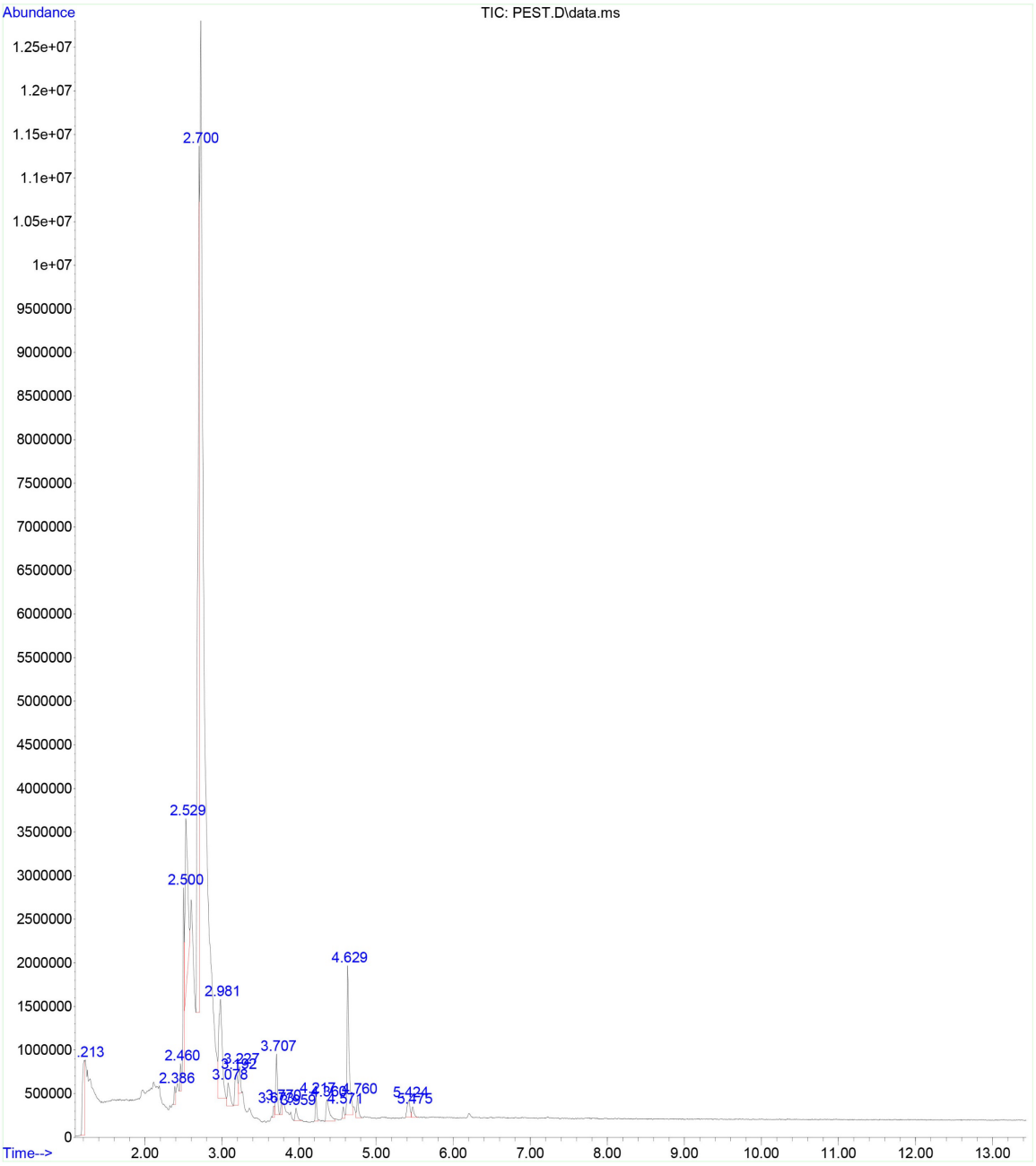

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Figure 6. Chromatogram of celery sample from Bamenda III, containing chlorothalonil with a retention time of 2.981 minutes.

Library Search Report

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Data File : SANTA SOIL.D
Acq On : 17 Mar 2025 21:52
Operator : LNAD SEF
Sample : SANTA SOIL
Misc :
ALS Vial : 2 Sample Multiplier: 1

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Integration Events: RTE Integrator - lscint.p

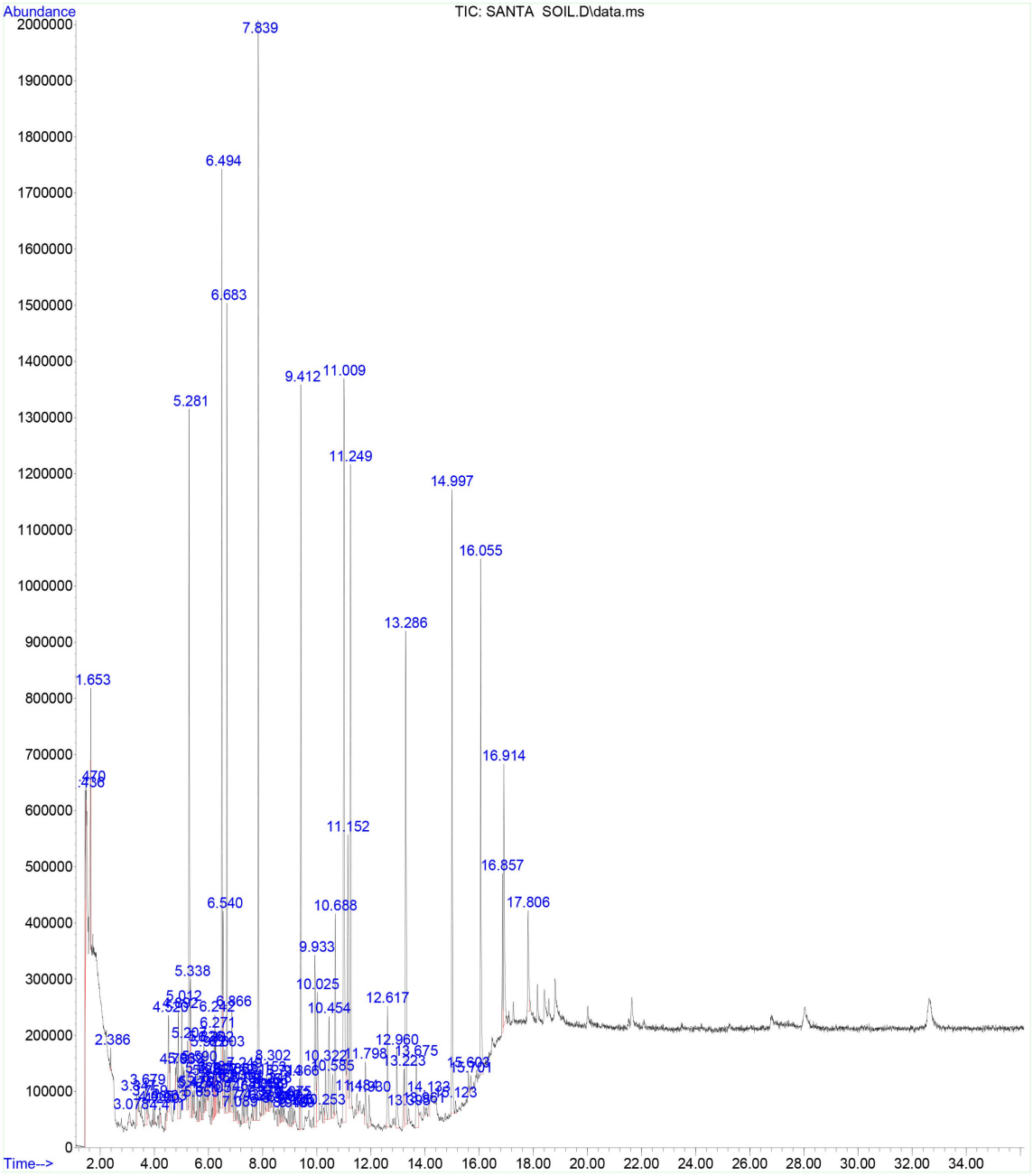

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Figure 7. Chromatogram of soil sample from Santa, containing phenol-2,4-bis (1,1-dimethyl) with a retention time of 6.683 minutes.

As seen on the chromatograms, chlorothalonil was detected in celery samples from Bayangam, Babadjou, and Bamenda III, while chlorpyrifos-methyl was found only in the celery sample from Foumbot. The summary of vegetable samples containing pesticide residues is presented in Table 1.

Table 1. Summary of vegetable samples containing pesticide residues.

Vegetable type	Collection site	Pesticide detected	Area	Retention time (min)
Celery	Bayangam	Chlorothalonil	1,203,282	2.969
Celery	Babadjou	Chlorothalonil	11,640,158	2.987
Celery	Bamenda III	Chlorothalonil	3,412,824	2.981
Celery	Foumbot	Chlorpyrifos-methyl	577,911	3.433

3.2. Result of the Quantification of Pesticide Residues

The results of the quantified pesticide detections, including concentrations and corresponding maximum residue limits (MRLs), are presented in Table 2.

Table 2. Concentration of pesticide residues and maximum residue limits (MRLs).

			Maximum Residue Limits			
Sample	Detected pesticide	Concentration (mg/kg)	Codex (mg/kg)	European Union (mg/kg)	Japan (mg/kg)	Remarks
Foumbot	Chlorpyrifos-methyl	2.5	0.05	0.01	0.03	Exceeded MRLs
Bamenda III	Chlorothalonil	18.3	20.0	0.05	20.0	Below MRLs
Bayangam	Chlorothalonil	6.6	20.0	0.05	20.0	Below MRLs
Babadjou	Chlorothalonil	62.1	20.0	0.05	20.0	Exceeded MRLs

The concentration of chlorpyrifos-methyl in Foumbot celery and chlorothalonil in Babadjou celery exceeded established MRLs, whereas the rest complied with regulatory standards. The frequency of sample contamination by pesticide residues is presented in Table 3.

Table 3. Frequency of sample contamination by pesticide residues.

Vegetable samples	Number	Percentage
Analysed	**24**	**100%**
Containing chlorothalonil	03	12.5%
Containing chlorpyrifos-methyl	01	4.2%
Total Containing pesticide residues	04	16.7%
Total without pesticide residues	20	83.3%

4. Discussion

Results of this study indicate that 16.7% of the 24 vegetable samples collected from

the Western Highlands of Cameroon contained detectable pesticide residues, while 83.3% were free of target residues or contained levels below the limit of detection (LOD). Chlorpyrifos-methyl and chlorothalonil were the only compounds detected. Notably, 50% of the positive samples exceeded the Maximum Residue Limits (MRLs) established by Japan, the EU, and the Codex Alimentarius Commission. While no major residues were detected in corresponding water or soil samples, the analysis of soil samples revealed the presence of 2,4-di-tert-butylphenol (Phenol-2,4-bis (1,1-dimethylethyl)), a prominent metabolite in all samples, which suggests the breakdown of environmental pollutants in the farming environment.

4.1. Reasons for Low Residue Detection

The high frequency of negative samples (83.3%) suggests that the majority of crops were either produced without the application of synthetic pesticides, were harvested in strict compliance with established Pre-Harvest Intervals (PHIs), or contained residues below the analytical Limit of Detection (LOD). Adherence to PHIs allows for the natural degradation of active ingredients via hydrolysis, photolysis, oxidation, and microbial metabolism, often reducing concentrations below the 0.01 mg/kg threshold. While Gas Chromatography-Mass Spectrometry (GC-MS) is highly effective for volatile, non-polar compounds such as organochlorines and organophosphates, it possesses inherent limitations regarding polar or systemic residues. These substances are more accurately quantified using Liquid Chromatography-Mass Spectrometry (LC-MS). Therefore, samples categorized as negative may represent instances where residues were present but fell below the specific sensitivity range of the GC-MS instrumentation.

4.2. Exceedance of Maximum Residue Limits (MRLs)

Quantification results indicate that celery samples from Babadjou exhibited the highest concentration of chlorothalonil (62.1 mg/kg), which significantly exceeds the 20 mg/kg Maximum Residue Limit (MRL) established, by the Codex Alimentarius and Japanese regulatory standards. Samples from Bamenda III (18.3 mg/kg) remained within the established MRL, while samples from Bayangam (6.6 mg/kg) were also compliant. Conversely, the concentration of chlorpyrifos-methyl in celery from Foumbot (2.5 mg/kg) represents a substantial exceedance of the MRLs established by the Codex Alimentarius (0.05 mg/kg), and the European Union (0.01 mg/kg). The detection of these residues at levels exceeding regulatory thresholds suggests non-compliance with Good Agricultural Practices (GAP), specifically regarding dosage, application timing, or failure to observe the mandatory Pre-Harvest Interval (PHI).

4.3. Comparison with Previous Studies on Pesticide Contamination in Cameroon

The findings of this study regarding pesticide exceedance align with previous in-

vestigations conducted across Cameroon. Research in the Western Highlands (Bamenda) by Sonchieu *et al.* [11] identified high levels of organophosphate residues, specifically chlorpyrifos that exceeded Codex Maximum Residue Limits (MRLs) in market garden crops. Furthermore, a 2017 pilot study by Tarla *et al.* [12] documented the application of banned substances and the persistent presence of chlorothalonil residues in vegetables within the Northwest and West regions. Complementing these findings, Galani *et al.* [13] reported that 100% of analyzed samples contained at least one pesticide residue, with 38% surpassing European Union MRLs. Collectively, these investigations underscore that non-compliant residue levels of pesticides, including chlorpyrifos-methyl and chlorothalonil, represent a critical food safety challenge for agricultural products in Cameroon.

Limitations

This study was limited by a small sample size and reliance on a single analytical method (GC-MS), which restricts the ability to detect polar or thermally labile pesticides. Additionally, the results do not account for the substantial reduction in pesticide residue levels that generally occurs through household processing like washing or cooking. Finally, the study was unable to definitively determine the source of phenol, 2,4-bis (1,1-dimethylethyl) in the soil samples.

5. Conclusion and Perspectives

The study reveals a critical issue of pesticide residue exceedance in celery from Foumbot and Babadjou, posing a direct food safety risk. This necessitates immediate action to address agricultural practices and ensure compliance with MRLs. The soil contamination with phenol-2,4-bis (1,1-dimethylethyl) points to a separate environmental concern, suggesting diverse sources of pollution in the regions. The absence of any detected contaminants in the water samples is a positive finding. However, this does not rule out the presence of other contaminants not analyzed or at concentrations below the detection limit.

Further research is needed to identify the exact source and potential environmental and health impacts of phenol-2,4-bis (1,1-dimethylethyl) in the soil. This could involve analyzing industrial waste, landfill leachate, or plastic degradation products, expanding the scope of environmental sampling or investigating whether the detected soil contaminants and other pesticides are accumulating in other parts of the food chain or local ecosystems. Furthermore, the methodology can be enhanced in future research by expanding the analyte list to include a broader spectrum of commonly used pesticides and environmental contaminants to provide a more comprehensive assessment, or by utilizing analytical methods with lower detection limits to identify trace levels of contaminants that might still have long term environmental or health implications.

Data Availability

Should further details be required, the data are available from the corresponding author upon reasonable request.

Acknowledgements

We extend our sincere gratitude to the farmers who graciously facilitated access to their lands for sample collection. Furthermore, we express our profound appreciation to the staff at the National Laboratory for the Analysis and Diagnosis of Agricultural Products and Inputs (DRCQ), Yaoundé, for their technical expertise and diligent execution of the analytical procedures.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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