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High performance liquid chromatographic determination of pesticide residue in zobo leaves available at local markets in Bayelsa State, Nigeria

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Abstract

The ingestion of food contaminated with residue pesticides is dangerous to human health. In order to avoid health risks to consumers, residue pesticides must be kept within the WHO/FAO-recommended limits. The aim of this study was to determine residues of Methomyl, carbendazim, acetamiprid, thiacloprid, and thiophanate methyl in zobo leaves. Zobo leaf is a major constituent of zobo drink; a local beverage in Nigeria, Ghana, Caribbean, etc. Seven zobo leaf samples were purchased from three local markets of Bayelsa State, Nigeria. Extraction of the pesticides was carried out on each zobo leaf samples using a mixture of ethyl acetate, hexane, and sodium chloride in the ratio of 3:1:1. The extracts were cleaned up on a glass column (containing activated charcoal and anhydrous sodium sulphate) and pre-concentrated in a water bath maintained at 45 °C. Residue pesticides were detected in all samples. The concentrations of residue pesticides ranged from 0.07–0.1 mg per kg of zobo sample while the % contamination in respect of 50 g zobo sample ranged from 0.0001–0.0004. It was found that the concentrations of all residue pesticides were within the Maximum Residue Limits (MRL) of Codex Alimentarius Commission standard of 0.1 mg kg⁻¹ in zobo leaves. The results indicate that zobo leaves sold at the local markets have sufficient quality for making zobo drinks with respect to residue pesticides.

Keywords: Determination, residue, pesticides, zobo leaves.

1. Introduction

Pesticides are used to improve the quantities and qualities of crops and foods either during pre- or post-harvest periods^[1]. However, pesticides are persistent and non-biodegradable and sometimes get bioaccumulated via the biological chains: soil-plant-food and seawater-marine organism food^[2]. And because of their persistence and non-biodegradability, they pose great danger to human health and the general environment^[3].

In order to protect consumers from eating contaminated fruits, vegetables, etc., many researchers have determined pesticide residues^[4-10].

The serious economic importance of residue pesticides to man has informed the aim of this work which was to determine some residue pesticides in zobo leaves (Zobo leaves is a major constituent of zobo drink; a local beverage in Nigeria, Ghana, Caribbean etc.) sold in the markets of Bayelsa State. Pesticides such as Methomyl, carbendazim, acetamiprid, thiacloprid, thiophanate methyl were determined in Zobo leaves.

2. Materials and Methods

2.1 Chemicals and Reagents: The Methomyl (98.5%), carbendazim (99.5%), acetamiprid (99.0%), thiacloprid (97.5%), thiophanate methyl were the analytes and were supplied by BDH, England.

Acetonitrile (HPLC grade), anhydrous sodium sulphate, activated charcoal, ethyl acetate, sodium chloride were bought from Merck, Germany. Agilent Chemstation software for controlling LC and data analysis. Agilent 1200 series HPLC (Agilent Technologies, Santa Clara, CA, USA).

2.2 Collection and Preservation of Samples

Zobo leaf samples ($n=7$) were bought from three markets in Yenagoa metropolis, Bayelsa State, Nigeria. The samples were fresh, free from blemishes or rot. Following collection, the samples were refrigerated at 4 ± 1 °C overnight and analyzed the next day.

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2.3 Sample Extraction

300 g of zobo leaves was chopped and a 50 g portion was macerated with ceramic mortar and pestle with a total volume of 50 mL of ethyl acetate, hexane, and sodium chloride in the ratio of 3:1:1. 20 g of anhydrous sodium sulphate was added to dry the sample. The mixture was further macerated for 5 min. The samples were then centrifuged for 5 min at 3000 rpm, and the supernatant collected. The organic extract was concentrated to 5 mL using a vacuum rotary evaporator with a water bath at 45 °C. The sample was then cleaned up with a glass column having 5 mL layer of anhydrous sodium sulphate and 10 g activated charcoal in order to remove any residual components that may interfere with the high performance liquid chromatographic analysis. The clean sample was further re-dissolved in 5 mL of acetonitrile for the HPLC analysis.

2.4 Analysis with HPLC

Following the cleaning of extract, the HPLC analyses were carried out on a Nucleosil 100, C₈ analytical column. A combination of 70% ACN and 30% water was used as the mobile phase, running at a flow rate of 1.0 mL/min and on isocratic mode. The column oven was maintained at 25 °C. All detections were done at 208 nm.

3. Results and Discussion

The chromatograms of the HPLC analyses of the zobo samples were presented in figure 1.

The HPLC analyses of the zobo samples showed that samples were contaminated in the range of 0.07–0.1 mg per kg of sample. The extent of contamination was expressed in weight per weight basis and as percentages as presented in Tables 1 and 2 respectively.

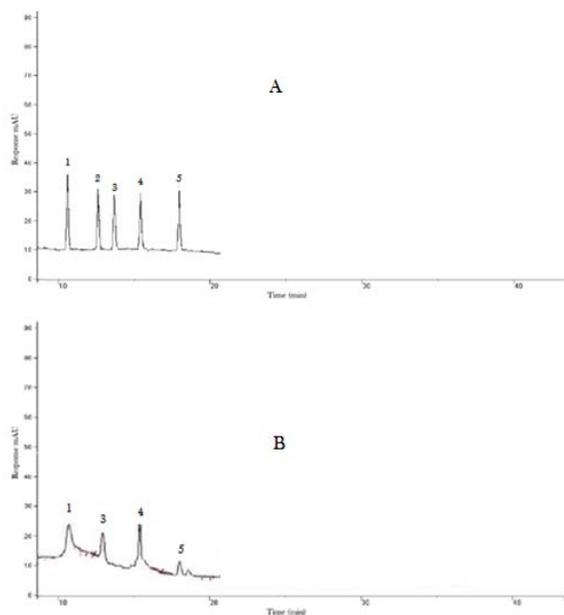


Fig 1: Chromatogram of pesticides; A, of pesticide standards, B, of Zobo sample. 1 = Methomyl, 2 = carbendazim, 3 = acetamiprid, 4 = thiacloprid, 5 = thiophanate methyl

Table 1

Pesticide	Retention time (minutes)
Methomyl	11.077
Carbendazim	12.877
Acetamiprid	13.720
Thiacloprid	15.664
Thiophanate methyl	18.366

Table 2: Contamination of pesticides in zobo leaves (weight per weight)

Pesticide	Concentration (mg/kg)
Methomyl	0.10±0.015
Carbendazim	0.07±0.021
Acetamiprid	0.09±0.022
Thiacloprid	0.10±0.015
Thiophanate methyl	0.09±0.054

Table 3

Pesticide	% contamination with respect to the weight (50 g) of zobo leaves
Methomyl	0.0004
Carbendazim	0.0001
Acetamiprid	0.0002
Thiacloprid	0.0003
Thiophanate methyl	0.0001

From Tables 2 and 3, it was found that the concentrations of residue pesticides ranged from 0.07–0.1 mg per kg of zobo sample while the % contamination in respect of 50 g zobo sample ranged from 0.0001–0.0004. This shows that the concentration of pesticide in zobo leaves was highest with Methomyl and Thiacloprid (0.10mg/Kg) while the lowest was with carbendazim (0.07mg/Kg). Thus, the contamination levels in zobo leaves for all the pesticides under investigation were found to fall within the acceptable limits. The different concentration levels of pesticide in the zobo leaves may be due to their different degrees of persistence in the environment, since their application or usage were relatively the same. The residue-pesticide contamination levels in all the zobo leaf samples were found to fall within The Codex Alimentarius Commission of the Food and Agricultural Organization (FAO) of the United Nation and World Health Organization (WHO); recommended an acceptable MRL in zobo leaves and vegetables to be 0.1 mg kg⁻¹

4. Conclusion

The contamination levels in zobo leaves for all the pesticides under investigation were found to fall within the acceptable limits. Therefore, the zobo leaves used in preparing the zobo drinks were safe for such preparations.

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