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Research Article

Extent and Nature of Pesticide Residues in Selected Fruits and Vegetables in Lahore, Pakistan

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ABSTRACT

Food safety is a global challenge with chemical residues in foods causing harm to consumers. In particular, pesticide residues in food value chains is one of the leading causes of toxicities and a major cause of human diseases globally. The increasing demand for fruits and vegetables due to rapid population growth has driven growers to enhance pesticide use to boost crop production. This study was conducted to assess the extent of pesticide residues in selected fruits and vegetables commonly sold at wholesale markets in the Lahore region, the second largest city in Pakistan. The concentrations of six commonly used pesticide residues were determined using High Performance Liquid Chromatography (HPLC) with a mass Photodiode Array Detector (PDA), in selected fruits (Citrus, Guava, and Apple) and vegetables (Okra, Brinjal, Spinach, Cabbage, and Cauliflower). During survey, a total of 120 samples were collected and analyzed for three commonly used pesticides (Bifenthrin, Imidacloprid, and Chlorpyrifos) residues. The results of this study revealed that nearly all samples were contaminated with pesticide residues. Among the fruits, 67% of samples and 40% of vegetable samples contained pesticide residues exceeding the Maximum Residue Limits (MRLs), while 33% of fruit samples and 60% of vegetable samples had pesticide residues below the MRLs. This study highlights the significant risk and threat to health posed by the accumulation of these poisonous substances in our daily food.

Keywords: Pesticide, analysis, Residues, HPLC, fruits, vegetables, Pakistan.

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INTRODUCTION

Rapid population growth in countries like Pakistan demands enhanced agricultural production to meet food requirements. To achieve the maximum crop production potential, extensive measures, including the indiscriminate use of pesticides. According to Munir *et al.* (2024), pesticide use increased by 36% during the period of 2000- 2019 worldwide. Globally, the trend in pesticide use has continued to upsurge gradually to 4.1 million tons per year in 2017, an increase of nearly 81% since 1990. However, extensive and increasing trend in use of synthetic pesticides pose risks to human health, the environment, and the livelihoods of smallholder farmers who are the majority of actors in agriculture (Radwan and Salama, 2006). In developed countries, the risks associated with pesticide residues have been acknowledged, leading to the establishment of critical limits and legislative measures aimed at minimizing these risks.

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Persistent compounds have been banned, and the development of safer pesticide formulations has been promoted to ensure human safety (Jones and De Voogt, 1999). Globally, approximately 2.5 million tons of pesticides are used annually, a figure that continues to rise (FAO/WHO, 2004). In Pakistan, pesticide use is particularly concentrated on certain crops, especially fruits and vegetables, due to poor legislation and farmers' lack of awareness (Parveen *et al.*, 2004; Parveen *et al.*, 2005; Saqib *et al.*, 2005), similar to other underdeveloped countries.
Control programs for pesticide residues in developing countries are often constrained by limited resources and inadequate legislation (Chen *et al.*, 2011). Balancing the effectiveness of pesticides with their potential risks remains a significant challenge for scientists striving to achieve optimal yields through safer agricultural practices (Osman *et al.*, 2010). Pesticide use also poses environmental contamination risks. Therefore, monitoring these toxic compounds is crucial to regulate their rational use, especially since farmers often apply these chemicals even during marketing, disregarding basic principles and ethics. Given the potential toxicity of pesticides sprayed heavily after cotton in Pakistan, and the lack of information on pesticide residue levels in fruits and vegetables sold in Lahore, the second-largest city in Pakistan, this study aimed to gather data for farmer awareness and policymaker information. Lahore encompasses both urban and rural areas, with much of the population engaged in agriculture but lacking proper knowledge of pesticide application practices and the associated risks due to communication gaps and inadequate information from government authorities (Latif *et al.*, 2011).
This study specifically assessed pesticide residues in Lahore, a pivotal market for fruit and vegetable distribution in the province. The pesticides studied included chlorpyrifos, bifenthrin, and imidacloprid. Chlorpyrifos, moderately toxic, is suspected to affect the endocrine system, with chronic exposure linked to neurological, developmental, and autoimmune disorders. Overdoses of bifenthrin and imidacloprid can also have detrimental effects on human health.

MATERIALS AND METHODS

Sampling

A total of 120 samples comprising fruits (Guava, Apple, and Citrus) and vegetables (Okra, Brinjal, Spinach, Cabbage, and Cauliflower) were collected from two major Fruit and Vegetable Markets: Muridke and Lahore City. Each sample, weighing approximately 1-2 kg, was collected, packed in polyethylene bags, and transported to the Pesticide Residue Laboratory in Kala Shah Kaku. The samples were stored at -20°C to prevent degradation prior to analysis.

Chemicals and Solvents

Certified standards of Chlorpyrifos, Bifenthrin, and Imidacloprid with purities ranging from 97% to 99%, sourced from Dr. Erhenstorfer, were used for the analysis. Anhydrous sodium sulfate and sodium chloride from Merck (Germany), and methanol, acetonitrile, acetone, and ethyl acetate (HPLC grades) from BDH (UK) were employed as solvents and reagents. Ultrapure deionized water was obtained from a water purification system (UK). For the clean-up of the extracts, solid phase extraction columns (C18, Waters, USA) were utilized.

Sample Extraction and Cleanup

Pesticide residues were extracted from the samples using a modified method based on Kadenczki *et al.* (1992). Fruit and vegetable samples were cut into small pieces, and 50 g of each sample was placed in a Waring Blender jug. To this, 50 ml of ethyl acetate, 20 g of anhydrous sodium sulfate, and 10 g of sodium chloride were added, and the mixture was blended at high speed for 3 minutes. The resulting mixture was filtered and passed through a florisil column, followed by an activated charcoal column. The collected contents were concentrated using a rotary evaporator at 35°C until dryness. The concentrated material was then filtered through a 0.45 micron membrane. The dried contents were dissolved in 1 ml of acetone and analyzed using HPLC-PDA. Pesticide residue analyses were conducted following the procedures outlined by Dekok and De Kok and Hiemstra (1992) and Ohlin (1986). Methanol-water and acetonitrile-water mobile phases were prepared, and the solutions were filtered through a 0.45 micron filter paper using a filtration assembly and sonicated for 10 minutes to remove air bubbles. The flow rate of the HPLC-PDA system was set at 1 ml per minute.
For Bifenthrin, gas chromatograph-mass spectrometer (Agilent model 6890N), coupled with a quadrupole mass spectrometer (model 5975B) with a GC column (HP-5MS 5% phenyl—95% methyl siloxane, 30 m × 0.25 mm id × 0.25 μm film thickness) was used in the analysis. The samples were injected in splitless mode. The GS operation conditions used in this analysis were splitless injections at an injector temperature of 250 °C. The carrier gas used was helium (99.9 purity) at a flow rate of 0.9 mL/min with column head pressure of 7.4 psi, oven temperature at 70 °C (for 2 min), then raised to 130 °C at a rate of 25 °C/min and then increased to 220 °C at a rate of 2 °C/min, and then again raised to 280 °C at a rate of 10 °C/min and held for 4.6 min. The selective ion monitoring (SIM) mode was routinely set in the MS system used. Based on the peak area, one target and one or two qualifier ions based on standard procedures were used to quantify each compound (Pihlström *et al.*, 2007).

Data Analysis

MS-Excel 2013 and Statistics Software were used to compile and analyze the data.

RESULTS

The primary objective of this assessment was to investigate the occurrence and extent of pesticide residues in the two main markets of Lahore region, Pakistan, which is surrounded by extensive agricultural lands and has seen increased adoption of tunnel farming over the past decade. Farmers have increasingly utilized pesticides to protect their crops and meet the growing demand for off-season fruits and vegetables, thereby enhancing the risk of pesticide residues in these perishable commodities. Despite the potential health issues associated with consuming contaminated food, public awareness regarding pesticide residues remains low, necessitating monitoring to inform both the public and policymakers.

The results presented in Tables 1 & 2 highlight alarming circumstances. A total of 25 samples of selected vegetables from major retailers were analyzed for three commonly used pesticides: Imidacloprid, Chlorpyrifos, and Bifenthrin. All vegetable samples contained detectable levels of pesticide residues. Bifenthrin showed the highest residue concentration, with 2.140 ppm in Okra and 0.558 ppm in Spinach. Imidacloprid followed as the second most concerning pesticide, with maximum residues of 0.210 ppm in Okra and 0.103 ppm in Cabbage. Chlorpyrifos residues were found at the lowest concentrations among the pesticides analyzed. Okra was identified as the most contaminated vegetable, followed by Cabbage.

Table 1: Pesticide residues in vegetable samples

Commodities	No	Imidacloprid (ppm)	Chlorpyrifos (ppm)	Bifenthrin (ppm)	Mean values
Okra	05	0.210 c	0.001 d	2.410 a	0.870^{A}
Brinjal	05	0.020 d	0.006 d	0.017 d	0.050^{CD}
Spinach	05	0.024 d	0.001 d	0.558 b	0.190^{B}
Cabbage	05	0.103 d	0.012 d	0.055 d	0.560^{C}
Cauliflower	05	0.018 d	0.002 d	0.002 d	0.007^{D}
Mean	25	0.075^{B}	0.005^{C}	0.607^{A}	

Table 2: Classification of vegetables on the basis pesticide residues

Commodities	No of Samples analyzed for pesticide residues				
	Total	Not Detected	< MRLs	> MRLs	> MRLs %
Okra	15	02	09	04	26.7
Brinjal	15	01	14	0	0.0
Spinach	15	04	09	02	13.3
Cabbage	15	02	13	0	0.0
Cauliflower	15	06	09	0	0.0
Total	75	15	54	6	40.0

Among the 15 Okra samples analyzed, only 2 were free from contamination, while 27% exceeded permissible residue limits (Figure. 1). Spinach ranked second in terms of pesticide residues, with 13% of samples exceeding determined Maximum Residue Limits (MRLs). Residues were also detected in Brinjal, Cabbage, and Cauliflower samples, but their concentrations were within safe limits. Overall, only 20% of the selected vegetable samples were free from pesticide residues.

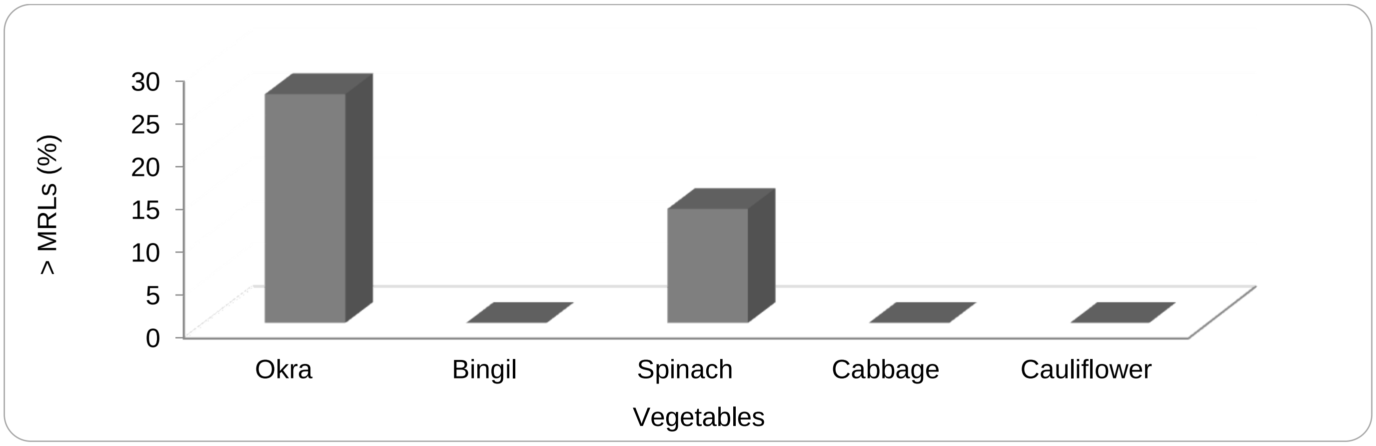

Figure 1: Vegetable samples (> MRLs)

Data regarding pesticide residues in fruits are detailed in Tables 3 & 4. Apple, Guava, and Citrus fruits were assessed for contamination with Imidacloprid, Bifenthrin, and Chlorpyrifos, with five samples of each fruit type

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analyzed. Imidacloprid was found to be the most contaminated pesticide, with concentrations reaching 1.213 ppm in Apple and 0.793 ppm in Guava. Bifenthrin followed as the second most contaminated pesticide, with levels of 0.362 ppm in Guava and 0.051 ppm in Apple. Chlorpyrifos, similar to vegetables, showed minimal residues, likely due to its relatively limited use in the area despite its high toxicity. Fruit samples showed lower contamination compared to vegetables. Out of 45 fruit samples analyzed, 62% were found to contain residues, with 22% exceeding permissible limits (Figure. 2). Apples were identified as the most contaminated fruit, followed by Citrus and then Guava.

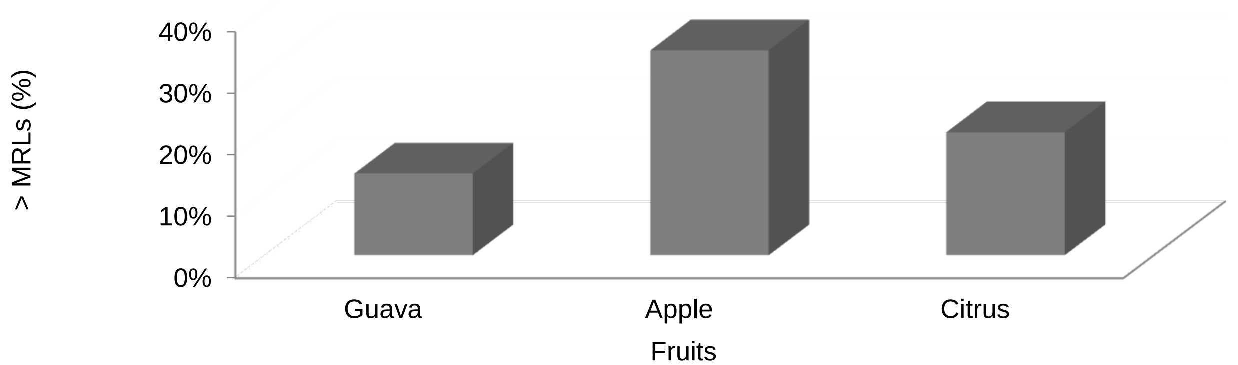

Figure 2: Fruits samples (> MRLs)

Table 3: Pesticide residues in fruit samples

Commodities	No	Imidacloprid (ppm)	Chlorpyrifos (ppm)	Bifenthrin (ppm)	Mean values
Guava	05	0.022	0.004	0.362	0.130^{C}
Apple	05	1.213	0.000	0.051	0.423^{A}
Citrus	05	0.793	0.071	0.006	0.290^{B}
Mean		0.68^{A}	0.025^{C}	0.140^{B}	

Table-4: Classification of fruits on the basis of pesticide residues

Commodities	No of Samples analyzed for pesticide residues				
	Total	Not Detected	< MRLs	> MRLs	> MRLs %
Guava	15	04	09	02	13.3
Apple	15	08	02	05	33.3
Citrus	15	05	07	03	20.0
Total	45	17	18	10	66.7

DISCUSSION

Taking into consideration that pesticides play a major role in increasing the production of agricultural products with high quality when moderately and safely applied in the control of crop pests, diseases, and weeds, their misuse may cause severe health problems. Pesticide residues' determination in food is an important action for monitoring contamination and ensuring food safety. This might help farmers and stakeholders in the proper handling of pesticides in terms of the applied dose, times of application, as well as the permissible level locally in each type of food for the health and safety of consumers (El-Sheikh *et al.*, 2022). During this survey, 120 fruit and vegetable samples were collected from sale points across Lahore for residue analysis. Information gathered from the local farming community included data on prevalent insects and pests, as well as the most commonly used pesticides in the region, which guided the selection of three pesticides for analysis. Analytical findings (Tables 1, 2, 3, 4) revealed that out of the 120 samples collected, 32 samples (26.66%) were free from any pesticide residues, while 88 samples (73.33%) showed contamination. Among these contaminated samples, 16 (18.18%) exceeded the Maximum Residue Limits (MRLs) set by the Codex Alimentarius Commission (FAO/WHO, 2004). However, 72 samples (81.81%) were found to have residues within safer limits. The increasing use of pesticides raises concerns that contamination levels may surpass safe thresholds, consistent with previous studies (Zahida Parveen *et al.*, 2011).
The MRLs were most frequently exceeded in apples (5 times) among fruits, followed by citrus (3 times), and in vegetables, okra (4 times) was most affected, followed by spinach (2 times). Okra is primarily attacked by borers,

while spinach faces issues with flies and sucking pests. Citrus crops commonly encounter citrus psylla and sucking pests, which are typically controlled through pesticide sprays. Data provided by growers helped identify the specific pesticides used for analysis, excluding those with minimal usage. Variations in pesticide concentrations within and across commodities may result from factors such as cropping periods, temperature and climate variations at harvest, timing of pesticide application, and the size of the produce (Chen *et al.*, 2011).

Chlorpyrifos, known for its high toxicity, was found within safe limits, indicating its limited use in the fruits and vegetables sampled in the study area. Interviews with farmers and analytical data revealed multiple pesticide residues, as farmers often apply pesticides as needed, even up to the time of marketing, to maintain product quality, often disregarding potential detrimental effects (de Pinho *et al.*, 2010). These findings align with results from other studies (Sheikh *et al.*, 2013; Latif *et al.*, 2011)

CONCLUSION AND RECOMMENDATIONS

The presence of pesticide residues in food that exceed the permissible MRLs leads to significant environmental and health damages. To preserve the health of consumers, it is necessary to monitor pesticide residues in food on an ongoing basis to determine the dynamics of pesticide presence in food, especially vegetables and fruits that are freshly consumed. The present study underscores the significant threat posed by pesticide residues in fruits and vegetables sold in the Lahore region. Analysis of 120 samples revealed that 16 samples (13.33%) exceeded Maximum Residue Limits (MRLs), potentially posing health risks. Pesticide residues were detected in 77.33% of the samples, while only 26.66% were found to be free from pesticide contamination. These findings highlight a critical lack of awareness among the farming community regarding the severity of pesticide residues, with their primary focus being on maximizing crop yields through extensive pesticide application.

The widespread and increasing use of pesticides on fruits and vegetables has escalated health risks for consumers. Addressing this issue requires urgent public education and awareness initiatives. Additionally, the establishment of standards and legislative measures is imperative to ensure safer practices in agricultural production aimed at higher yields

AUTHOR CONTRIBUTIONS

All authors contributed equally to this research.

COMPETING OF INTEREST

The authors declare no competing interests.

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