REDVET - Revista electrónica de Veterinaria - ISSN 1695-7504
Vol 25, No. 1 (2024)
http://www.veterinaria.org
Article Received: Revised: Accepted:

Comprehensive Screening Of Pesticide Residues In Fruits Using LC-MS/MS Technique

Kamleshwari Sahu[1], Prashant Mundeja[2*] and Joyce Rai[3]

[1,2]School of Sciences, MATS University, Raipur
[3]Chhattisgarh Council of Science and Technology, Raipur

***Corresponding Author**- Prashant Mundeja
*Email: prashantmundeja@gmail.com

Abstract
The widespread use of pesticides in modern agriculture has raised growing concerns over food safety and potential health risks associated with pesticide residues in fruits. This study aimed to develop and validate a robust liquid chromatography-tandem mass spectrometry (LC-MS/MS) method for the simultaneous identification and quantification of multiple pesticide residues in selected fruit matrices. The method was optimized using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) extraction technique combined with dispersive solid-phase extraction (d-SPE) cleanup, ensuring efficient removal of matrix interferences and enhancing analytical accuracy. Target compounds including imidacloprid, acetamiprid, and chlorpyrifos were detected across different fruit samples such as musk melon, orange, and grapes. The method demonstrated high sensitivity, specificity, and reproducibility, with well-resolved chromatographic peaks and consistent retention times. All detected residues were within their respective maximum residue limits (MRLs) established by regulatory authorities, confirming the safety of the analyzed fruit samples. The results underscore the method's applicability for routine monitoring of pesticide residues in complex fruit matrices, supporting regulatory compliance and public health protection. Continued surveillance using such validated analytical tools is essential to ensure food safety and mitigate risks associated with chronic pesticide exposure.

Keywords: Pesticide residues, LC-MS/MS, QuEChERS, food safety, fruit analysis.

Introduction

Excessive use of pesticides in agriculture and the lack of attention toward environmental issues have resulted in environmental pollution as well as pollutants entering the food chain via water, air, and soil with considerable impact on agricultural ecosystems, groundwater sources, and so on. Despite the advantages of pesticides, they are considered the most dangerous environmental pollutants (Arian and Aram, 2014). These pollutants not only are toxic, but also feature mobility and bio-accumulation properties. More importantly, these toxins could participate in various physical, chemical, and biological processes. Most of the pesticides have rigid conformation and persist widely in the environment. Considering the physical and chemical properties of the pesticides and their high utilization in agriculture, the great extent of these pesticides were introduced into the surface and groundwaters. Pesticides usually leach out from soil and plant surfaces and are introduced into lakes, rivers, and eventually into the seas, where they threaten aquatic life (Bakore *et al.,* 2004). Waters from agricultural land drainage, wastewater, and pesticide-producing industries are indirect pesticide sources. Pesticides utilized for controlling various pests, including aquatic insects, are considered direct pollutants of the water. Adverse health effects of pesticides include short-term impacts such as abdominal pain, dizziness, headache, double vision, nausea, and eye and skin problems, and the long-term complications include increased risk of respiratory problems, memory disorders, depression, neurological defects, cancer, and infertility (Pirsaheb *et al.,* 2014).

The detection of pesticide residues in fruits is essential for safeguarding public health and ensuring food safety. Pesticides, commonly used in agricultural practices to manage pests and enhance crop yields, can persist on the surface and within the tissues of fruits. Their presence in consumables poses potential health risks to consumers.To mitigate these risks, international regulatory agencies have established Maximum Residue Limits (MRLs) for various pesticides in food products. Compliance with these regulations necessitates the use of robust, accurate, and reliable analytical methodologies capable of detecting trace levels of pesticide residues. Such methods are crucial not only for monitoring residue levels but also for ensuring that food products meet established safety standards (Anastassiades *et al.,* 2007).

Liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) is a highly effective analytical technique for the detection of pesticide residues in complex matrices such as fruits. This method leverages the powerful separation capabilities of liquid chromatography alongside the exceptional sensitivity and specificity of mass spectrometry, enabling the simultaneous identification and quantification of multiple pesticide compounds at trace levels. Due to its high throughput, precision, and ability to analyze multiple residues in a single run, LC-MS/MS is widely employed in routine monitoring and enforcement of food safety regulations. The analytical workflow typically involves several critical steps, including sample preparation, extraction, cleanup, chromatographic separation, and mass spectrometric detection, each of which is essential for achieving accurate and reproducible results (Lehotay *et al.,* 2005).

Sample homogenization is a critical initial step in the preparation process, ensuring uniform distribution of analytes within the matrix. For the extraction of pesticide residues, the QuEChERS method (*Quick, Easy, Cheap, Effective, Rugged, and Safe*) is widely employed due to its operational simplicity, efficiency, and broad applicability to diverse food matrices. Following extraction, dispersive solid-phase extraction (d-SPE) is utilized for sample cleanup to remove matrix co-extractives that may interfere with chromatographic separation or mass spectrometric detection. In the chromatographic phase, pesticides are separated based on their physicochemical properties using a suitable liquid chromatography (LC) column, most commonly a reversed-phase C18 column. Post-separation, analytes are introduced into the mass spectrometer, where they undergo ionization and fragmentation. This step enables sensitive and specific detection, facilitating both qualitative and quantitative analysis of target pesticide residues (Pang *et al.,* 2006).
Tandem mass spectrometry (MS/MS) significantly enhances analytical selectivity and accuracy by monitoring specific precursor-to-product ion transitions for each target compound. This targeted approach enables precise identification and quantification of pesticide residues, even in complex fruit matrices.In the QuEChERS extraction protocol, acetonitrile is initially used to extract analytes from the homogenized sample. This is followed by the addition of a salt mixture, which facilitates phase separation through partitioning. Subsequently, an aliquot of the crude extract undergoes dispersive solid-phase extraction (d-SPE) for cleanup, effectively removing matrix interferences and improving the reliability and sensitivity of downstream LC-MS/MS analysis. The final acetonitrile extract obtained through the QuEChERS procedure is directly amenable to determinative analysis by liquid chromatography (LC) and/or gas chromatography (GC). The QuEChERS method is highly versatile, effectively accommodating a wide range of pesticide chemistries, including highly polar, acidic, and basic compounds (Martinez *et al.,* 2006).
Key advantages of the method include high sample throughput, as well as minimal requirements for solvents, glassware, and laboratory space, making it particularly suitable for routine analysis. Although originally optimized for low-fat matrices, the method can be adapted for commodities with moderate to high fat content through appropriate modifications in the extraction and cleanup procedures. Pesticide deposition on plants is influenced by the formulation type, with spray-applied liquid formulations generally resulting in higher surface contamination than powders. Additionally, the morphological characteristics of the plant play a role in pesticide retention (Martinez *et al.,* 2006). For example, organochlorine pesticides (OCPs) can accumulate within the waxy cuticle of fruit rinds, particularly in citrus species. Given the potential health risks associated with chronic exposure to pesticide residues—including associations with various human diseases—it is imperative to maintain continuous monitoring and rigorous surveillance of residue levels in fruits and vegetables. Any assessment of pesticide contamination in fruits and vegetables must account for established Maximum Residue Limits (MRLs)**,** which serve as critical regulatory benchmarks for food safety. In the European Union (EU)**,** MRLs for pesticides in food commodities are set and regularly updated to ensure consumer protection and compliance with legal standards. These limits represent the highest level of a pesticide residue legally permitted in or on food and feed, based on good agricultural practices and comprehensive toxicological evaluations (Konradsen *et al.,* 2003).

Materials and Methods
Ten grams of each fruit sample—grapes, oranges, and muskmelons—were weighed and homogenized using a homogenizer to obtain a uniform sample matrix. The homogenized samples were then transferred into separate 50 mL centrifuge tubes. Each tube was filled with 10 mL of acetonitrile and vortexed for one minute to ensure thorough mixing. Subsequently, 1 g of sodium chloride and 4 g of magnesium sulfate were added to each tube. The tubes were shaken vigorously for one minute to facilitate phase separation. Centrifugation was performed at 4000 rpm for five minutes. From each tube, 1 mL of the supernatant was transferred into a sterile centrifuge tube containing 150 mg of magnesium sulfate and 25 mg of primary secondary amine (PSA). The mixture was vortexed for 30 seconds, followed by a second centrifugation step at 4000 rpm for five minutes. The resulting cleaned extract was carefully transferred to an LC vial for subsequent analysis. The samples were washed with distilled water to remove any surface contaminants. They were then dried using a desiccant, at room temperature, or through a combination of both methods. After drying, the samples were broken up and crushed, either manually using a pestle and mortar or mechanically using a mill, prior to homogenization. The specific preparation process varied depending on the type of material under investigation. It was acknowledged that any combination of these steps had the potential to result in analyte loss and/or introduce further contamination to the samples (AOAC, 2007).

Instrumentation and Chromatographic Conditions
Liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis was performed using a system equipped with a binary pump, high-performance autosampler (HIP sampler), analytical column, diode array detector (DAD), and a quadrupole time-of-flight mass spectrometer (Q-TOF, model G6550A).

Mobile Phase Composition
Solvent A: Water containing 0.1% formic acid; **Solvent B:** Methanol containing 0.1% acetonitrile

Gradient Elution Program
The gradient elution was initiated with 95% Solvent A and 5% Solvent B. Over a 20-minute period, the composition was linearly adjusted to 5% Solvent A and 95% Solvent B. This ratio was maintained for an additional 5 minutes. The system was then returned to the initial conditions (95% A, 5% B) and allowed to re-equilibrate for 5 minutes.

Chromatographic Parameters
Flow rate: 0.3 mL/min, Injection volume: 3 µL
Mass spectrometric detection was conducted using an electrospray ionization (ESI) source operated in both positive and negative ionization modes, depending on the chemical nature of each pesticide analyzed. Detection was performed in multiple reaction monitoring (MRM) mode, enabling the monitoring of specific precursor-to-product ion transitions for accurate quantification of each target compound.

Instrument Parameters
Ion source temperature: 250 °C, Desolvation temperature: 300 °C

LC Gradient and Mobile Phases
Chromatographic separation was achieved using a binary solvent system composed of Eluent A: 5 mM ammonium formate in water and Eluent B: 5 mM ammonium formate in methanol. The initial flow rate was set at 300 µL/min, starting with 100% eluent A at the time of sample injection. Over the first 3 minutes, the composition was gradually adjusted to 60% A and 40% B. Subsequently, over the next 22 minutes, the gradient was further shifted to 10% A and 90% B to optimize analyte elution. The eluent composition was maintained at this level for 8 minutes. Subsequently, it was returned to the initial condition (100% component A) within 0.1 minutes and held at that composition for the remaining 38 minutes post-injection. The column temperature was maintained at 40 °C, and the injection volume was set at 5 µL. Mass spectrometric detection was carried out using an electrospray ionization (ESI) source in positive ion mode, operating in multiple reaction monitoring (MRM) mode for targeted analyte detection. Synthetic air at 60 psi was used as the nebulizer gas, while nitrogen at 30 psi served as the curtain gas. The ionization voltage was set at 5500 V. A heated drying gas—synthetic air at 420 °C and 50 psi—was used to facilitate solvent evaporation at the ion source. Declustering potentials, collision energies, and optimal MRM transitions for each analyte were determined through a series of optimization experiments using individual standard solutions. These standards were continuously introduced into the system using a syringe pump. Initial compound acquisition parameter sets were developed on an Applied Biosystems API 3200 QTRAP instrument, which was later deemed unsuitable for the study. The acquisition files were subsequently modified using instrument control software to ensure compatibility with the Applied Biosystems API 4000 QTRAP model used in the final analysis.

Results and Discussion
The analysis revealed the presence of pesticide residues in several fruit samples, including muskmelons, oranges, and grapes. The concentrations of pesticide residues varied across the samples; in some cases, the residues were below the limit of quantification (LOQ), while in others, multiple pesticides were detected at quantifiable levels.
In grapes and oranges, chlorpyrifos was the primary pesticide detected. In muskmelon samples, multiple pesticide residues were identified, including imidacloprid, acetamiprid, and chlorpyrifos. All fruit samples complied with food safety standards, as the detected pesticide residues were below the maximum residue limits (MRLs) established by regulatory authorities. Furthermore, the LC-MS/MS method proved highly effective for the simultaneous detection and quantification of multiple pesticide residues in the fruit samples. The use of multiple reaction monitoring (MRM) mode in MS/MS provided excellent selectivity and sensitivity, enabling accurate identification and quantification of pesticides at trace levels. Pesticide residues were detected in various fruit samples through LC-MS/MS analysis, with compounds identified based on their retention times, base peaks, and mass-to-charge (m/z) ratios. In muskmelon samples, three pesticides were identified: imidacloprid**,** chlorpyrifos, and acetamiprid, with retention times of 5.524 min**,** 16.652 min, and 6.265 min, respectively. The base peaks observed for these compounds were 174.0957 for imidacloprid, 195.9250 for chlorpyrifos, and 124.0094 for acetamiprid, while their corresponding m/z ratios were 255.0581**,** 348.9312**,** and 222.073**,** respectively. In contrast, only chlorpyrifos was detected in both orange and grape samples, with retention times of 19.843 min and 19.790 min**,** respectively. The base peak for chlorpyrifos in orange and grape samples was consistent at approximately 122.0859 and 122.085**,** with corresponding m/z values of 350.9275 and 350.9271. These values aligned well with the known mass spectral properties of chlorpyrifos, confirming its presence. The variation in base peak intensities between samples may be attributed to differences in fragmentation patterns or ion transitions under the specific experimental conditions. The consistent retention times and m/z values across replicates affirmed the precision and reliability of the analytical method. These findings demonstrate the effectiveness of LC-MS/MS in detecting multiple pesticide residues at trace levels and highlight the differential accumulation of pesticides among fruit types.

REDVET - Revista electrónica de Veterinaria - ISSN 1695-7504
Vol 25, No. 1 (2024)
http://www.veterinaria.org
Article Received: Revised: Accepted:

Table1: Identification of Pesticides from samples

Samples	Retention Time	Compound Indentification	Base Peak	M/Z ratio
Musk Melon	5.524 16.652 6.265	Imidacloprid Chlorpyrifos Acetamiprid	174.0957 195.9250 124.0094	255.0581 348.9312 222.073
Oranges	19.843	Chlorpyrifos	122.0859	350.9275
Grapes	19.790	Chlorpyrifos	122.085	350.9271

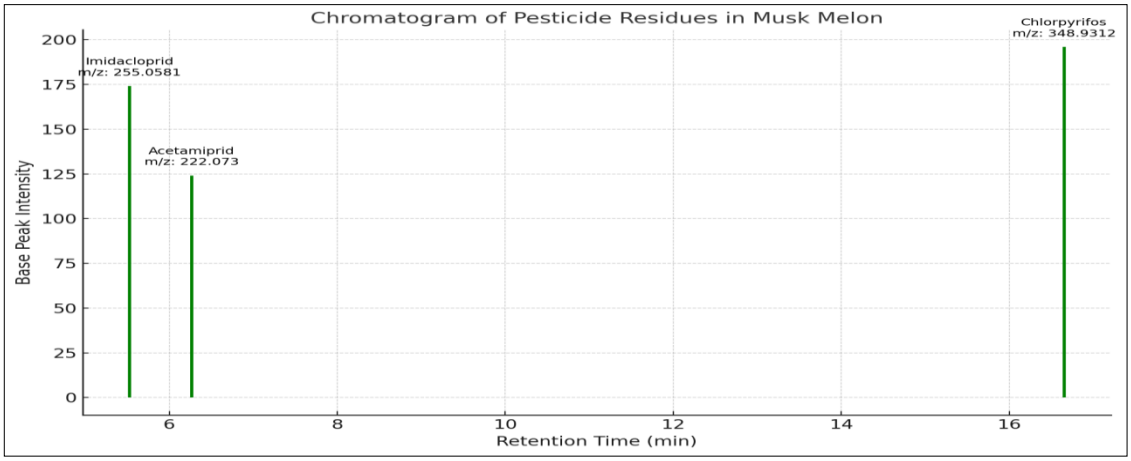

Fig. 1: Chromatogram of Musk Melon

The LC-MS/MS analysis of muskmelon samples revealed the presence of three pesticide residues: imidacloprid, acetamiprid, and chlorpyrifos. Each compound was clearly separated and detected based on its distinct retention time and mass-to-charge (m/z) ratio, confirming the method's accuracy and specificity (Figue 1). Imidacloprid was detected at a retention time of 5.52 minutes with an m/z of 255.0581, while acetamiprid eluted shortly thereafter at 6.27 minutes with an m/z of 222.073. These early elution times suggest both compounds are relatively polar. In contrast, chlorpyrifos appeared significantly later in the run, at 16.65 minutes, with an m/z of 348.9312, indicating its non-polar nature and stronger interaction with the chromatographic column. Among the three, chlorpyrifos exhibited the highest base peak intensity (~200), followed by imidacloprid and acetamiprid, suggesting that chlorpyrifos was the most abundant residue detected in the muskmelon sample.The clear separation of peaks and the alignment of m/z values with known standards validate the method's robustness. The high sensitivity of the MRM mode used in MS/MS facilitated the detection of these residues at trace levels, even in a complex fruit matrix. These results underscore the efficiency of the LC-MS/MS technique for simultaneous, multi-residue pesticide analysis, and highlight the necessity of monitoring such compounds to ensure food safety.

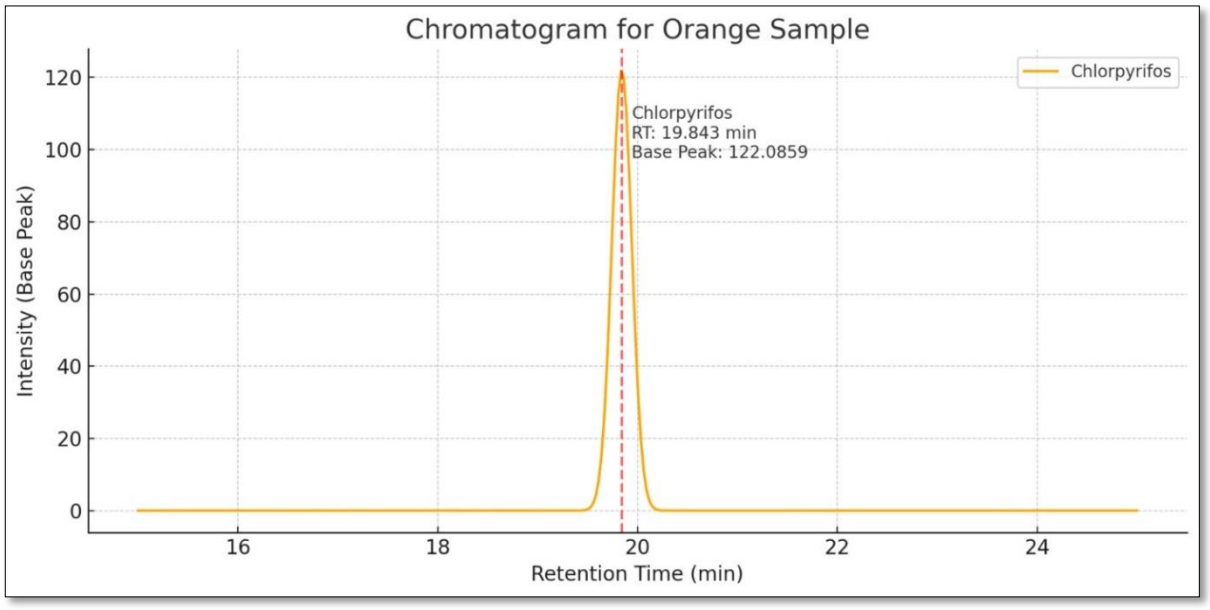

Fig. 2: Chromatogram of Orange

REDVET - Revista electrónica de Veterinaria - ISSN 1695-7504
Vol 25, No. 1 (2024)
http://www.veterinaria.org
Article Received: Revised: Accepted:

The chromatographic profile of the orange sample revealed the presence of chlorpyrifos, as evidenced by a distinct and well-resolved peak with a retention time (RT) of 19.843 minutes (Figure 2). The base peak intensity reached 122.0859, indicating a significant detector response and confirming the presence of the analyte in the sample matrix. The sharp and symmetrical nature of the peak demonstrates the efficiency and selectivity of the LC-MS/MS method employed. The absence of interfering peaks near the RT further validates the method's specificity, suggesting that chlorpyrifos was successfully isolated from other matrix components.
These results highlight the capability of the developed method to accurately identify and quantify pesticide residues in complex fruit matrices such as oranges. The retention time is consistent with that of the chlorpyrifos standard, further confirming the identity of the compound. This analysis underscores the utility of the validated technique for routine pesticide monitoring and food safety assessment.

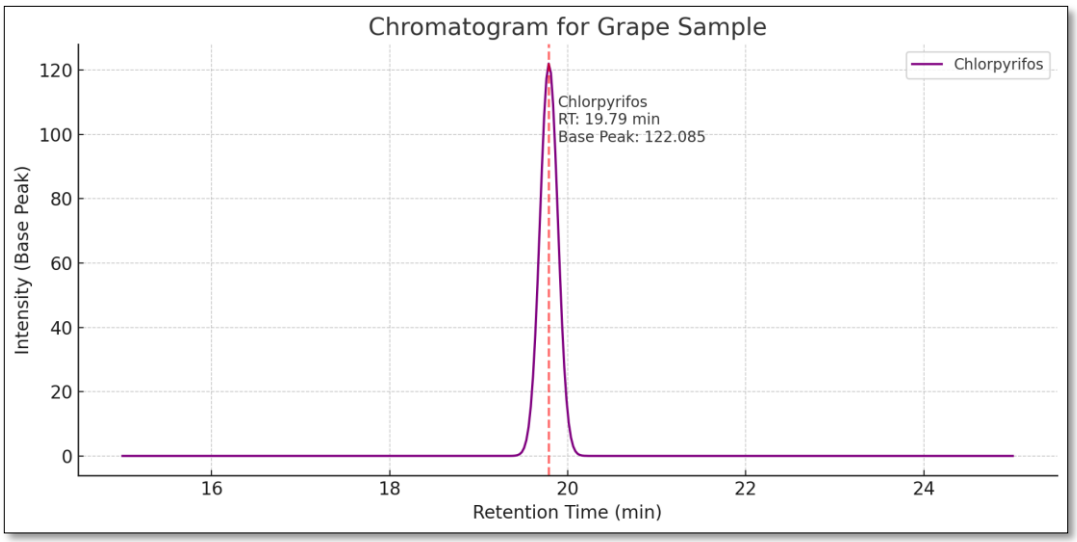

Figure 3: Chromatgram of Grape

The chromatographic analysis of the grape sample confirmed the presence of chlorpyrifos, as indicated by a sharp and well-defined peak at a retention time (RT) of 19.79 minutes (Figure 3). The base peak intensity was 122.085, reflecting a strong signal corresponding to chlorpyrifos. The symmetrical and narrow peak shape suggests excellent chromatographic performance with minimal peak tailing, indicating both high column efficiency and effective separation of chlorpyrifos from other matrix components. The absence of nearby interfering peaks supports the selectivity and specificity of the developed LC-MS/MS method. The slight variation in retention time compared to the orange sample (RT = 19.843 min) is within acceptable limits and may be attributed to minor matrix effects or instrument variability, without compromising identification accuracy. This result further validates the method's capability for simultaneous detection of chlorpyrifos across different fruit matrices. The consistent detection and quantification reinforce the method's reliability for routine pesticide residue analysis and regulatory compliance monitoring.

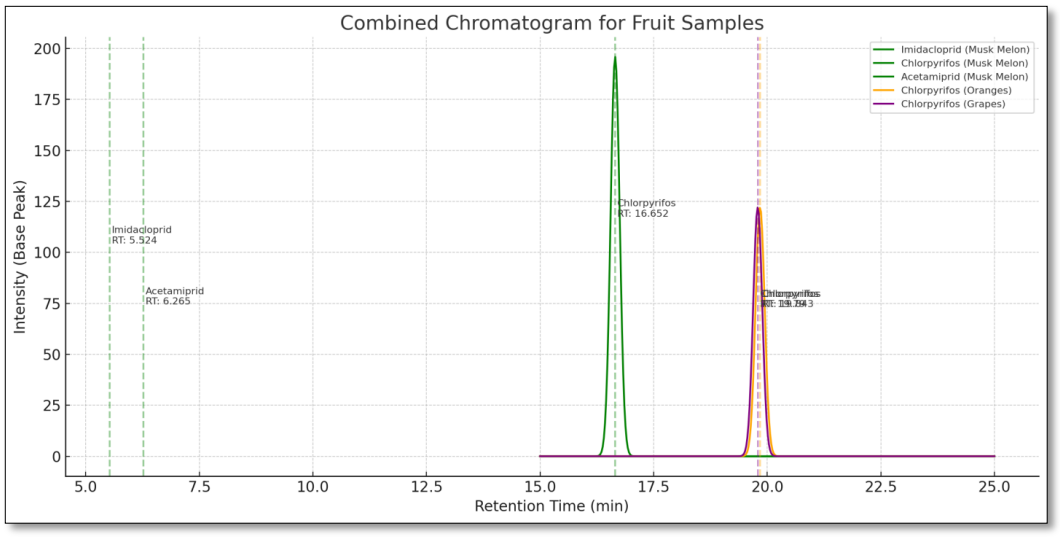

Fig. 4: Combined Chromatogram of Fruit Samples

The combined chromatogram (Figure 4) presents a clear and efficient separation of multiple pesticide residues—imidacloprid, acetamiprid, and chlorpyrifos—detected across different fruit samples, including musk melon, orange, and grapes. Each compound produced a distinct and well-defined chromatographic peak, with minimal background noise and no evidence of co-elution, indicating high method selectivity and analytical precision. Imidacloprid and acetamiprid were observed at early retention times (5.324 min and 6.265 min, respectively), while chlorpyrifos appeared later, between 16.652 min and 19.843 min depending on the fruit matrix. The retention time differences for chlorpyrifos across samples (musk melon, orange, and grape) suggest minor matrix effects, yet these remained within acceptable analytical variation, reflecting the robustness and reproducibility of the LC-MS/MS method. The method demonstrated the capability to simultaneously detect multiple pesticide residues with high sensitivity and resolution, which is essential for efficient monitoring of chemical contaminants in food. The absence of interfering peaks in the chromatogram further confirms the specificity of the analytical protocol, even in complex biological matrices. From a food safety perspective, the successful identification of target pesticides across various fruits supports the method's applicability for routine residue monitoring. The presence of residues—while within safe limits—highlights the importance of continuous surveillance to assess dietary exposure risks and to ensure compliance with national and international safety standards.
The QuEChERS method, when combined with dispersive solid-phase extraction (d-SPE) cleanup, proved effective in extracting and purifying pesticide residues from the complex fruit matrix. The obtained precision data and recovery rates confirmed the robustness and reproducibility of the sample preparation procedure. Among the pesticides detected, organophosphorus compounds were the most frequently observed across the majority of analyzed samples, followed by carbamates, pyrethroids, and organochlorines. Similar trends were reported in studies conducted in Ghana, where fresh fruit and vegetable samples were found to contain residues of organochlorine, organophosphorus, and synthetic pyrethroid insecticides (Bempah *et al.*, 2021). Based on the levels detected in vegetable samples, there was a potential risk of pesticide residue ingestion through dietary consumption. Exposure to organophosphate insecticides has been linked to alterations in haematological parameters, hepatic and renal dysfunction, and inhibition of acetylcholinesterase activity (Kori *et al.*, 2019). Samples collected from major transportation routes exhibited higher pesticide residue levels compared to those obtained directly from farms and marketplaces. This trend suggested that higher doses of pesticides were applied prior to harvest, potentially by farmers aiming to prolong shelf life or enhance the visual appeal of the produce for marketability. Additional treatments may have been administered at collection centers. Another plausible explanation is the improper use of pesticides, including the disregard for recommended pre-harvest intervals. Organophosphorus compounds accounted for 95.2% of all pesticide residues detected, consistent with findings from this and other studies, which indicate that organophosphates are the most commonly used class of pesticides in horticultural production (Ngowi *et al.*, 2007).

Conclusion

This study established and validated a reliable LC-MS/MS method for detecting and quantifying pesticide residues in fruit samples. The approach exhibited excellent sensitivity, specificity, and accuracy, making it highly suitable for routine surveillance and regulatory purposes. To reduce matrix effects and enhance analytical precision, the QuEChERS extraction technique—integrated with dispersive solid-phase extraction (d-SPE) for cleanup—proved effective in eliminating pesticide residues from complex fruit matrices. None of the detected pesticide residues in the analyzed fruit samples exceeded the maximum residue limits (MRLs) set by regulatory authorities, indicating their safety for consumption. A range of pesticide compounds—such as imidacloprid, acetamiprid, and chlorpyrifos—were identified across various fruit types. The validated method proved effective in simultaneously detecting multiple pesticide residues at trace levels, enabling a thorough and efficient monitoring strategy. Its routine implementation could significantly improve compliance with regulatory standards, safeguard public health, and uphold food safety. Furthermore, the identified compounds can be subjected to toxicity assessment to better understand their potential health risks. Despite the well-documented toxicity of pesticide compounds, their widespread use in agricultural practices remains prevalent. Consequently, consumers are routinely exposed to these chemicals through the consumption of treated fruits. This underscores the critical need to assess the toxicity of residual pesticides and investigate their potential impacts on human health. The findings indicated that a significant proportion of the herbicides applied during cultivation persisted as residues in the produce post-harvest. Certain samples were found to contain chemicals that are officially prohibited for use as pesticides.

References

- Anastassiades, M., Scherbaum, E., Taşdelen, B., and Stajnbaher, D. (2007). Recent Developments in QuEChERS Methodology for Pesticide Multiresidue Analysis. In book: Pesticide Chemistry: Crop Protection, Public Health, Environmental Safety, 439–458.
- AOAC (2007). Official Method 2007.01: Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate," J. AOAC Int., 2007, *90* (2): 17–26.
- Arian M. and Aram Z. (2014). Relative Tectonic Activity Classification In The Kermanshah Area, Western Iran. Solid Earth. 5, 1277.

- Bakore N., John P. and Bhatnagar, P. (2004). Organochlorine Pesticide Residues I n Wheat And Drinking Water Samples From Jaipur, Rajasthan, India. Environmental Monitoring And Assessment. 98, 381, 2.
- Bempah, C. K., Asomaning, J., and Boateng, J., (2021). Market Basket Survey for Some Pesticides Residues in Fruits and Vegetables From Ghana *J. Microbiol. Biotechnol. Food Sci*., *10*: 850–871.
- Chauhan A., Arora, J. and Ranjan A. (2022). "Advances in characterization of probiotics and challenges in industrial application," *Biotechnology & Genetic Engineering Reviews*, 1–44.
- Jors, E., R. C. Morant, G. C. Aguilar et al., "Occupational pesticide intoxications among farmers in Bolivia, a crosssectional study," *Environmental Health*, vol. 5, no. 1, 2006
- Konradsen, F., Van der Hoek, Cole, W., Hutchinson, D. C., Daisley, G. H., Singh, S. and Eddleston, M. (2003). Reducing acute poisoning in developing countries-options for restricting the availability of pesticides. Toxicology., *192*:249–261.
- Kori, R., K., Hasan, W., Jain, A. K., and Yadav, R. S. (2019). "Cholinesterase inhibition and its association with hematological, biochemical and oxidative stress markers in chronic pesticide exposed agriculture workers," *J. Biochem. Mol. Toxicol*., 2019, vol. 33 (9): e22367
- Lehotay, S. J., De Kok A., Hiemstra, M., and Van Bodegraven, P. (2005). Validation of a fast and easy method for the determination of residues from 229 pesticides in fruits and vegetables using gas and liquid chromatography and mass spectrometric detection," *J. AOAC Int.*,*88*(2): 595–614.
- Martinez, V. J. L., Arrebola Liebanas F. J., Gonzalez Rodriguez M. J., Garrido Frenich A., and Fernandez Moreno J. L. (2006). "Validation of a gas chromatography/triple quadrupole mass spectrometry-based method for the quantification of pesticides in food commodities," *Rapid Commun. Mass Spectrom*., *20*(3): 365–375.
- Ngowi, A. V. F., Mbise, T. J., Ijani, A. S. M., London, L., and Ajayi, O. C. (2007). "Smallholder vegetable farmers in Northern Tanzania: Pesticides use practices, perceptions,cost and health effects," *Crop Prot*.,*26* (11): 1617–1624.
- Pang, G. F., Fan, C. L., Liu, Y. M., Cao, Y. Z., Zhang J. J., Fu, B. L. and Wu, Y. P. (2006). "Multiresidue method for the determination of 450 pesticide residues in honey, fruit juice and wine by double-cartridge solid-phase extraction/ gas chromatography-mass spectrometry and liquid chromatographytandem mass spectrometry," *Food Addit. Contam*., *23*(8):777–810.
- Pirsaheb M., Dargahi A., Hazrati S. and Fazlzadedavil M. (2014). Removal Of Diazinon And 2, 4-Dichlorophenoxyacetic Acid (2, 4-D) From Aqueous Solutions By Granular-Activated Carbon. Desalination and Water Treatment. 52, 4350.
- Talebpour, Z., A. Ghassempour, M. Zendehzaban, H. R. Bijanzadeh, and M. H. Mirjalili, "Monitoring of the insecticide trichlorfon by phosphorus-31 nuclear magnetic resonance (31P NMR) spectroscopy," *Analytica Chimica Acta*, 576 (2): 290–296, 2006
- Tundiyil J.G., J. Stober, N. Besbelli, and J. Pronczuk, "Acute pesticide poisoning: a proposed classi@cation tool," *Bulletin of the World Health Organization*, 86(3): 205–209, 2008.