

Evaluation of Extraction Methods and Cleaning Agents for Chlorpyrifos Residue Reduction in Grapes Using Gas Chromatography–Mass Spectrometry (GC-MS)

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Structured Abstract

Background: Chlorpyrifos is an organophosphorus pesticide commonly used in grape cultivation. Its persistence on fruit surfaces poses significant health and environmental risks, especially since grapes are often consumed raw. Grapes are listed among the top pesticide-contaminated fruits by the Environmental Working Group's "Dirty Dozen." This study aimed to evaluate the effectiveness of various household washing agents in reducing chlorpyrifos residues on grapes and to assess the performance of Solid Phase Extraction (SPE) combined with Gas Chromatography–Mass Spectrometry (GC-MS) for residue analysis.

Methods: Grape samples were artificially spiked with 200 ppm chlorpyrifos and subjected to five washing treatments: 10% baking soda, 5% acetic acid, a commercial cleaning agent, tap water, and 5% salt solution. The extraction efficiency of Solid Phase Extraction (SPE) was compared with Solid Phase Microextraction (SPME). Due to its superior performance, SPE was selected for subsequent analysis. Chlorpyrifos quantification was conducted using GC-MS equipped with an HP-5MS capillary column in splitless mode at an injection temperature of 280°C. The temperature program began at 70°C (held for 2 minutes), ramped at 15°C/min to 280°C, and was held for 5 minutes. Helium was used as the carrier gas at 1.0 mL/min.

Results: The highest chlorpyrifos removal (72%) was achieved using 10% baking soda, followed by 5% acetic acid (60%), commercial cleaning agent (54%), tap water (48%), and salt solution (44%). The GC-MS method demonstrated excellent linearity and reliability for chlorpyrifos detection. SPE produced cleaner extracts and outperformed SPME in extraction efficiency.

Conclusion: The findings confirm that 10% baking soda is the most effective household washing agent for reducing chlorpyrifos residues in grapes. The combination of SPE and GC-MS offers a reliable analytical approach for monitoring pesticide residues in food, supporting safer consumption and improved food safety practices.

Keywords: Chlorpyrifos, pesticide residue, grapes, Gas Chromatography-Mass Spectrometry (GC-MS), Solid Phase Extraction (SPE).

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