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Environmental and Food Safety Assessment of Emamectin Benzoate Residues on Apple Fruits: Field Efficacy of Emamectin Benzoate and Lufenuron against Codling Moth (*Cydia pomonella* L.)

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Abstract

This study assessed the environmental and food safety implications of using Emamectin benzoate and the insect growth regulator Lufenuron for the management of codling moth, Cydia pomonella L., on Ibrahimi apple trees during the 2023-2024 growing season in Al-Husseiniya district, Karbala, Iraq. The work combined field efficacy evaluation with residue monitoring of Emamectin benzoate on apple fruits using high-performance liquid chromatography (HPLC), because pesticide performance should be interpreted together with residue persistence and consumer-safety considerations. Treatments differed significantly according to pesticide type, spray number, and post-treatment interval. Emamectin benzoate showed the highest rapid efficacy, reaching 62.83% after 7 days when means across sprays were considered and 67.66% after the second spray, whereas Lufenuron showed a delayed but sustained effect, with the highest efficacy of 58.33% after 28 days following the second spray. Residue analysis indicated that Emamectin benzoate deposits declined from 412.6 ug kg-1 at 1 h after spraying to 42.0 ug kg-1 by day 5, and residues were not detected by HPLC at day 7 and later intervals. The estimated half-life was 38.298 h. These findings indicate that Emamectin benzoate can provide rapid suppression of C. pomonella, while Lufenuron may serve as a delayed-action component in integrated pest management. From an environmental and food safety perspective, the rapid residue decline supports a suggested 7-day pre-harvest interval under the conditions of this experiment, provided that official maximum residue limits, method recovery, LOD, and LOQ are confirmed before final publication.

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1. Introduction

Apple, Malus domestica Borkh. (Rosaceae), is one of the most economically and nutritionally important fruit crops worldwide. Apple fruits contain carbohydrates, proteins, lipids, vitamins, minerals, and bioactive compounds such as flavonoids, catechins, quercetin, and carotenoids. In Iraq, summer apple production was estimated at 78,917 tonnes in 2021, representing a decrease of 0.62% compared with 79,413 tonnes in 2020, and the average yield was estimated at 29.98 kg tree-1 (Central Statistical Organization, 2021; Al-Hijami, 2020). Apple production is affected by several environmental and biological factors, among which insect pests are among the most important limitations. The codling moth, Cydia pomonella L. (Lepidoptera: Tortricidae), is a major pest of apple and other pome fruits. Larvae penetrate the fruits and feed internally, causing direct economic loss, premature fruit drop, reduced marketability, poor storage quality, and increased susceptibility to secondary infection. If management is neglected, damage can reach economically unacceptable levels (Al-Jamali, 1995; Depalo et al., 2022).

Chemical control remains widely used in orchard pest management because of its rapid action, availability, and compatibility with scheduled crop-protection programs. However, repeated and intensive insecticide use may increase residue risks, affect human and environmental health, and accelerate the development of resistance in pest populations (Abbas, 2023; Bosch et al., 2018; Abu-Duka and Mohammadali, 2021; Abdul-Jaleel et al., 2023; Al-Dahwi et al., 2005; Al-Mashhadani, 2010). Therefore, the evaluation of insecticide efficacy should be accompanied by residue monitoring, especially on fruits consumed fresh.

Emamectin benzoate is a semi-synthetic derivative of abamectin, produced from avermectin compounds of Streptomyces avermitilis. It belongs to the avermectin group and acts mainly on glutamate-gated chloride channels, leading to paralysis and death of susceptible insects after ingestion or contact. Lufenuron is a benzoylurea insect growth regulator that inhibits chitin synthesis. It acts mainly on eggs and immature stages and commonly produces delayed mortality because its effect appears during molting and development (Salam and Mohammadali, 2021; Mansour and Al-Douri, 2020).

This study aimed to evaluate the field efficacy of Emamectin benzoate and Lufenuron against C. pomonella on Ibrahimi apple trees and to assess the environmental and food safety significance of Emamectin benzoate residue dissipation on apple fruits using HPLC.

2. Materials and Methods

2.1. Study site and experimental design

The experiment was conducted during the 2023-2024 growing season in an apple orchard located in Al-Husseiniya district, Karbala Governorate, Iraq. Ibrahimi apple trees of similar size, age, and fruiting condition were selected from a 2-dunam orchard naturally infested with C. pomonella. The experiment was arranged according to a randomized complete block design (RCBD) with three replicates per treatment. Each replicate consisted of three trees, and the control treatment was sprayed with water only.

2.2. Insecticides used

The pesticides used in the field experiment are shown in Table 1. Both pesticides were applied at the recommended field concentration according to the manufacturer instructions and the approved local registration information.

Table 1. Trade names, active ingredients, and chemical groups of the pesticides used.

Trade name and formulation	Active ingredient and concentration	Chemical group	Manufacturer	Registration information
Proclaim 0.5 SG	Emamectin benzoate 5%	Avermectins	Syngenta, Switzerland	Re-registered in 2021
Match 50 EC	Lufenuron	Benzoylurea insect growth regulator	Syngenta, Switzerland	Registered by the Ministry of Agriculture, No. 609

2.3. Field application and efficacy assessment

Spraying was carried out on 15 May 2024 using a calibrated 16-L backpack sprayer. The spray solution was applied uniformly from the lower to the upper canopy to ensure adequate coverage of leaves and fruits. Two spray applications were evaluated. The relative efficacy of each pesticide was assessed at 1, 3, 7, 14, 21, and 28 days after spraying. At each sampling date, five medium-sized fruits were randomly collected from different positions and heights within each replicate. Samples were placed in labeled polyethylene bags and transported to the laboratory for assessment. Relative efficacy was calculated using the Henderson and Tilton correction formula (Henderson and Tilton, 1955):

Efficacy (%) = 100 x [1 - ((T_after x C_before) / (T_before x C_after))]

where T_before and T_after are the pest counts in the treated plots before and after spraying, respectively, and C_before and C_after are the corresponding counts in the untreated control.

2.4. Sampling for Emamectin benzoate residue analysis

To determine the dissipation of Emamectin benzoate residues, apple fruits were sampled at 1 h, 5 h, 24 h, 3 days, 5 days, 7 days, 14 days, 21 days, and 28 days after application at the recommended concentration of 0.5 g L-1. Medium-sized fruits were collected from the four cardinal directions of each treated tree. The samples were placed in black airtight plastic bags, labeled, and stored at -20 °C until HPLC analysis (Sivaperumal et al., 2015).

Table 2. Sampling intervals for Emamectin benzoate residue analysis on Ibrahimi apple fruits.

No.	Sampling date	Time after treatment
1	15 May	1 h
2	15 May	5 h
3	16 May	24 h
4	18 May	3 days
5	20 May	5 days
6	22 May	7 days
7	29 May	14 days
8	5 June	21 days
9	12 June	28 days

2.5. Extraction and clean-up of pesticide residues

Apple samples were homogenized, and 5 g of the homogenate was transferred to a centrifuge tube. Ten milliliters of 1% acetic acid in acetonitrile were added, and the mixture was vortexed thoroughly. An aliquot of the extract was transferred using a micropipette and subjected to rapid clean-up. The cleaned extract was passed through a 0.45-um membrane filter, evaporated to dryness using a rotary evaporator, reconstituted in 20 mL methanol, and kept under refrigeration until HPLC analysis (Belguet et al., 2019).

2.6. HPLC determination and calibration

Emamectin benzoate residues were determined using an HPLC system at the Environment and Water Department laboratories, Ministry of Science and Technology. Analysis was performed on a SYKAMN HPLC system (Germany). Separation was achieved on a C18 ODS column (25 cm x 4.6 mm). The mobile phase consisted of water (pH 7.0) and acetonitrile (40:60, v/v) under isocratic conditions at a flow rate of 1.0 mL min-1. Detection was performed using a UV-Vis detector at 210 nm.

Standard solutions of Emamectin benzoate were prepared to construct the calibration curve. Four standard levels (50, 75, 100, and 125; unit to be verified before submission) were used. The calibration curve showed a correlation coefficient (R2) of 0.999, indicating acceptable linearity. The retention time of the standard was compared with the retention time observed in sample extracts to identify Emamectin benzoate residues. The extraction, purification, and quantification procedures followed EPA guidance for Emamectin benzoate residue analysis, with modifications involving the use of ready-made solid phase extraction (SPE) columns (U.S. Environmental Protection Agency (EPA), 1998).

2.7. Statistical analysis

Efficacy data were analyzed according to RCBD using analysis of variance (ANOVA). Means were compared using the least significant difference (LSD) test at $P <= 0.05$. Statistical analyses were performed using GenStat software (VSN International, 2019).

3. Results and Discussion

3.1. Field efficacy and integrated pest management relevance of Emamectin benzoate and Lufenuron against Cydia pomonella

The field results showed significant differences in relative efficacy according to pesticide type, number of sprays, and time after treatment (Table 3). The overall mean efficacy of Lufenuron was 36.19%, whereas Emamectin benzoate recorded an overall mean of 30.08%. Although the overall mean of Lufenuron was higher, Emamectin benzoate showed a faster and more pronounced early effect, particularly at 7 days after treatment. This rapid reduction is important in orchard management because internal fruit feeders such as C. pomonella can cause direct market losses once larvae penetrate the fruit. However, rapid efficacy alone should not be the only criterion for selecting a pesticide; the environmental load, residue persistence, and safety of harvested fruits must also be considered.

The number of spray applications also affected efficacy. The second spray produced a higher overall mean efficacy (34.91%) than the first spray (31.36%). This indicates that repeated application under field infestation conditions improved suppression of C. pomonella. The time interval after treatment was also significant; the highest mean efficacy across treatments was recorded at 7 days after spraying (50.33%), whereas the lowest efficacy occurred at 1 day after treatment (10.50%).

The interaction between pesticide type and time interval showed that Emamectin benzoate reached its highest mean efficacy at 7 days after treatment (62.83%), while Lufenuron showed its highest mean efficacy at 28 days after treatment (54.33%). This pattern agrees with the expected mode of action of the two compounds. Emamectin benzoate is known for relatively rapid neurotoxic action in susceptible larvae, while Lufenuron interferes with chitin synthesis and therefore produces delayed effects that become clearer during larval development and molting. From an environmental management perspective, alternating or integrating compounds with different modes of action may reduce unnecessary repeated applications and may help limit selection pressure for resistance when used within an integrated pest management program.

The three-way interaction among pesticide, spray number, and time interval showed that the highest efficacy (67.66%) was recorded 7 days after the second spray of Emamectin benzoate. In contrast, the lowest value (0.00%) was recorded 1 day after the first spray of Lufenuron. These findings suggest that Emamectin benzoate is more suitable when rapid reduction of active infestation is needed, whereas Lufenuron may be more useful as part of a preventive or integrated pest management program targeting eggs and early larval stages. Reducing the dependence on a single fast-acting insecticide may also support safer residue management by allowing spray schedules to be planned according to pest pressure, pre-harvest interval, and fruit consumption safety.

Table 3. Relative efficacy (%) of Emamectin benzoate and Lufenuron at different post-treatment intervals on Ibrahimi apple trees infested with *C. pomonella.*

Treatment	**Spray**	**1 d**	**3 d**	**7 d**	**14 d**	**21 d**	**28 d**	**Treatment x spray**
Emamectin benzoate	First	10.00	19.66	58.00	31.00	19.66	14.00	25.38
Emamectin benzoate	Second	17.00	28.66	67.66	53.33	23.00	18.66	34.77
Lufenuron	First	0.00	33.00	36.00	45.33	45.66	50.33	35.05
Lufenuron	Second	15.00	23.00	39.66	48.66	39.33	58.33	37.33
Time interval effect	-	10.50	26.08	50.33	44.66	31.91	35.33	-

LSD0.05: treatment = 3.6109; spray = 3.6109; time interval = 6.2543; treatment x spray = 7.2218; treatment x time = 9.8652; spray x time = 9.8652; treatment x spray x time = 12.509.

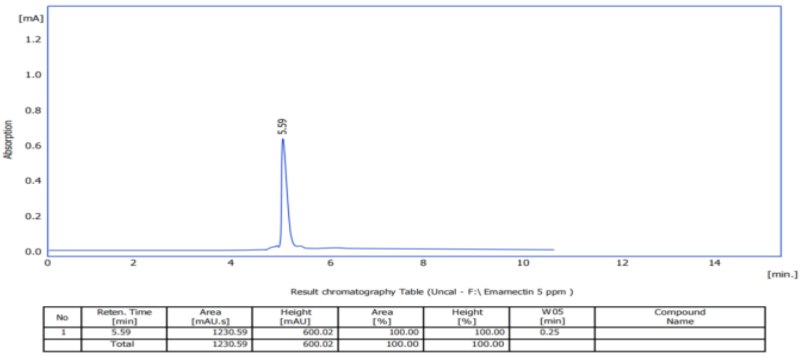
3.2. Environmental implications, residue dissipation, and food safety assessment of Emamectin benzoate on apple fruit

The chromatogram of the Emamectin benzoate standard showed a clear peak at a retention time of approximately 5.59 min, which was used as the reference for identifying the active ingredient in apple fruit samples (Figure 1). The use of HPLC in this part of the study was essential because residue monitoring provides a direct basis for evaluating the food safety aspect of pesticide application and for interpreting whether field control can be achieved without leaving persistent detectable residues on harvested fruits.

Residue analysis showed a rapid decline in Emamectin benzoate concentration over the first 5 days after application (Figure 2). The initial deposit reached 412.6 ug kg-1 at 1 h after spraying and then decreased to 321.58 ug kg-1 after 5 h, 166.00 ug kg-1 after 24 h, 88.9 ug kg-1 after 3 days, and 42.0 ug kg-1 after 5 days. Residues were not detected by the HPLC method at 7, 14, 21, and 28 days after treatment. This decline indicates that the active ingredient did not persist at detectable levels for long periods under the experimental orchard conditions, which is a favorable result for food safety when the recommended waiting period is respected.

The rapid dissipation may be attributed to the chemical properties of Emamectin benzoate, environmental conditions in the orchard, and exposure to temperature, sunlight, and humidity. High temperature and direct solar radiation may accelerate degradation on fruit surfaces, while fruit growth and surface characteristics can also contribute to residue dilution. The calculated half-life was 38.298 h, indicating relatively fast degradation under the conditions of this experiment. These results are consistent with previous reports indicating rapid dissipation of Emamectin benzoate residues in different crops and environmental matrices. Environmentally, rapid dissipation can reduce the period during which residues remain available on fruit surfaces; nevertheless, it does not eliminate the need for responsible application, avoidance of excessive spraying, and protection of non-target organisms within the orchard ecosystem.

The absence of detectable residues after 7 days suggests that a 7-day pre-harvest interval may be appropriate under the conditions of this study. However, for publication and regulatory interpretation, the final manuscript should clearly report the residue unit, the official maximum residue limit (MRL) used for comparison, the limit of detection (LOD), the limit of quantification (LOQ), and recovery percentages from method validation. Without these values, the phrase 'below the permissible limit' should be used cautiously. Therefore, the food safety conclusion should be expressed as follows: Emamectin benzoate residues became non-detectable by the applied HPLC method after 7 days, and this result supports the safety of harvesting after this interval only when it is confirmed against the official MRL and validated analytical performance criteria.



Result chromatography Table (Uncal - F:\ Emamectin 5 ppm)

No	Reten. Time [min]	Area [mAU.s]	Height [mAU]	Area [%]	Height [%]	W05 [min]	Compound Name
1	5.59	1230.59	600.02	100.00	100.00	0.25	
	Total	1230.59	600.02	100.00	100.00		

Figure 1. HPLC chromatogram of the Emamectin benzoate standard.

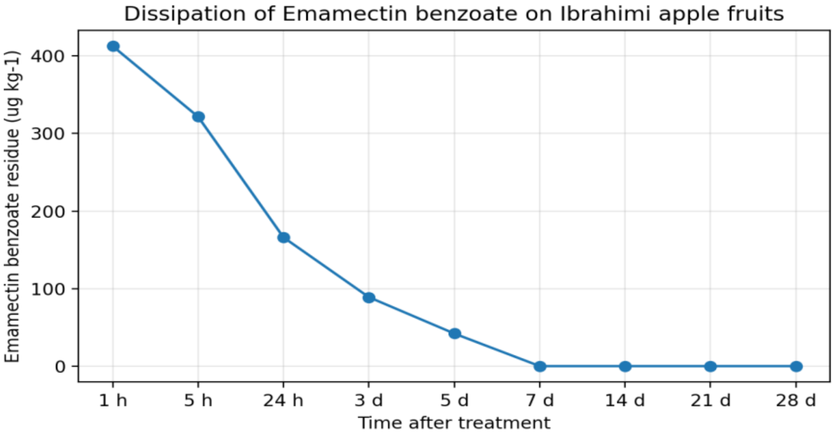


Figure 2. Dissipation pattern of Emamectin benzoate residues on Ibrahimi apple fruit

4. Conclusion

Emamectin benzoate and Lufenuron differed in their field performance against C. pomonella on Ibrahimi apple trees. Emamectin benzoate produced the highest rapid efficacy 7 days after spraying, especially after the second application, whereas Lufenuron showed a delayed effect that increased at later intervals. These results support the use of both compounds within an integrated pest management strategy that balances rapid pest suppression with reduced unnecessary pesticide pressure. From an environmental and food safety perspective, Emamectin benzoate residues declined rapidly on apple fruits, with no residues detected by HPLC at 7 days and later sampling dates. The estimated half-life was 38.298 h. A 7-day pre-harvest interval is suggested under the experimental conditions, but this recommendation should be finalized only after confirming residue units, official MRL, LOD, LOQ, and recovery data.

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