JSAES
Journal of Sustainable Agricultural and Environmental Sciences
Print ISSN : 2735-4377
Online ISSN : 2785-9878
Homepage: https://jsaes.journals.ekb.eg/

Research Article

Persistence and Dissipation Behavior of Selected Pesticide Residues in Grape Berries and Leaves under Field Conditions in Egypt

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Article info: -
- **Received:** 30 July 2025
- **Revised:** 10 August 2025
- **Accepted:** 15 August 2025
- **Published:** 24 August 2025

Keywords:
Grapes; Pesticides Residues; Dissipation rate; Maximum Residue Limits; QuEChERS; HPLC

Abstract:

Pesticide use boosts agricultural productivity, but excessive residues can harm human health. To ensure consumer safety, pesticide residue levels must remain below the established Maximum Residue Limits (MRLs). Therefore, regular monitoring of pesticide residues in fruits is essential for maintaining food safety and public health. In this study, the dissipation rate, half-life ($t_{0.5}$), and pre-harvest interval (PHI) of three commonly used pesticides in grape cultivation, boscalid, penconazole, and dimethomorph, were investigated in grape berries and leaves under field conditions. Residue levels were measured at various time intervals as following: 0 (2 hours), 1, 3, 5, 7, 10, 14, and 21 days post-application. These pesticides were applied to grapevines under open-field conditions.. Sample extraction and cleanup were conducted using the QuEChERS method, followed by analysis through High-Performance Liquid Chromatography (HPLC) coupled with a QTRAP mass spectrometer. The results indicated that the half-lives of boscalid, penconazole, and dimethomorph in grape fruits and leaves were 7.0 and 1.24 days, 5.0 and 6.7 days, and 2.49 and 3.5 days, respectively. Corresponding pre-harvest intervals (PHIs) were 5.0 and 3.0 days for boscalid, 4.0 and 6.0 days for penconazole, and 8.5 and 4.5 days for dimethomorph. Additionally, the estimated degradation rates in grape berries and leaves were as follows: boscalid (0.0991 and 0.1036), penconazole (0.557 and 0.279), and dimethomorph (0.147 and 0.0200), respectively. Rapid degradation (half-lives $\leqslant$7 days) and PHIs $\leqslant$8.5 days suggest low persistence of these pesticides, aligning with food safety standards and minimizing consumer exposure risks.

1. Introduction

Grapes (*Vitis* spp.) are among the most widely cultivated and economically significant fruit crops worldwide. While grapes are often consumed fresh (Evaristo et al., 2022), they are also processed into various products, including wine, juice, jams, vinegar, seed oil, and raisins (Beres et al., 2017). Grapes are highly valued, nutritious, and widely consumed fruits around the globe (Kgang et al., 2023).

In Egypt, grapes rank as the second most important fruit crop after citrus. The total cultivated area reached about 172,533.6 feddans in Egypt with an annual total production of about 1,586,342 tons (FAO, 2020).

Grapevines worldwide are commonly affected by significant fungal and insect pests such as powdery mildew, spider mites, two-spotted spider mites, thrips, and aphids, which cause considerable economic losses by reducing both yield and fruit quality (Banerjee et al., 2008; Wang et al., 2021).

Grapes are widely cultivated across the globe due to their significant economic importance and nutritional benefits, which often require the application of pesticides to safeguard the crop against pests and diseases and to maintain optimal yield and quality (Narenderan et al., 2019). However, the use of pesticides is one of the ways to enhance production efficiency (Badawy et al., 2019; Hesham et al., 2019; Heshmati and Nezemi, 2018). Pesticides are a mixture of chemical compounds for killing, destroying, or mitigating the threat of pests (Pallares et al., 2020; Serefoglu and Serefoglu, 2016). Pesticides are a vital component of modern agriculture, significantly contributing to the maintenance of high crop yields. In intensive, high-input agricultural production systems, the widespread use of pesticides for pest control has become a common practice (Tilman et al., 2002). Nevertheless, this heavy dependence on pesticides poses sustainability challenges due to their unintended long-term negative impacts on both the environment and, more critically, human health (Pimentel, 2005). Certain pesticides are persistent, difficult to degrade, and can remain in food products even after industrial processing (Cámara et al., 2020; Hamed et al., 2024). Health concerns linked to pesticide exposure vary from acute effects, such as nausea and headaches, to more severe chronic conditions, including various types of cancer, reproductive disorders, birth abnormalities, infertility, and disruptions to the endocrine system (Cecchi et al., 2012; Alavanja et al., 2013).

Harvesting crops after pesticide application, especially fruits and vegetables, might lead to high levels of pesticide residues in food commodities, which might have

chronic effects on human health upon consumption. A study analyzing vegetables from various Egyptian governorates found that 72% of samples contained detectable pesticide residues, with 21% exceeding European Union MRLs. Tomatoes and strawberries exhibited the highest frequency of pesticide presence (Abuo El-kasem et al., 2023). In addition, another research focusing on the Sharkia Governorate identified 40 different pesticides in fruits and vegetables, with approximately 40% of the residues exceeding MRLs. Cucumber and apple samples had the highest number of pesticide residues (El-Sheikh et al., 2022).

Although pesticides prevent or minimize crop loss due to pests, their residues in agricultural products are a major concern with regard to food safety (Aydin and Ulvi, 2019; Hamidi et al., 2019; Nazemi et al., 2016; Shoeibi et al., 2013). The analysis of pesticide residues in food is an essential component of food safety and public health monitoring, as it provides valuable data on the extent of human exposure to potentially hazardous chemicals. This analytical process not only ensures compliance with established MRLs but also helps identify trends in pesticide use and dissipation (Vasylieva et al., 2017; El-Shaikh and Ashour, 2022).

To safeguard human health, pesticide residues in agricultural products and foodstuffs must remain below the MRLs established by regulatory authorities, such as the European Commission (European Commission, 2005; Razzaghi et al., 2018). The Pre-Harvest Interval (PHI) refers to the number of days between the last application of a pesticide and the harvest of a crop, during which the pesticide residues degrade to levels below the MRL established for safe consumption. Therefore, conducting pesticide dissipation studies is essential for determining appropriate PHIs, as they provide critical data on the degradation rates and residue dynamics in crops such as grapes, ensuring both regulatory compliance and consumer safety (Horska et al., 2020).

The objective of this study was to investigate the dissipation, persistence, and residue levels of boscalid, penconazole, and dimethomorph in grape berries and leaves, as well as to determine the pre-harvest intervals (PHI) and half-lives ($t_{0.5}$) at the recommended application rates.

2. Materials and Methods

2.1. Tested pesticides and application rates

Three pesticides, each belonging to different chemical groups, were applied to grape berries and leaves in our study at their recommended doses.

2.1.1. Boscalid

Boscalid (Rukan 50% WG; 2-chloro-N-(4'-chlorobiphenyl-2-yl) nicotinamide). Boscalid was applied at the recommended field rate of 100 cm^3/100 L water. The product was supplied by StarChem Industrial Chemicals, Egypt.

2.1.2. Penconazole

Penconazole (Topas 10 % EC; 1-[2-(2,4-dichlorophenyl)pentyl]-1,2,4-triazole). Penconazole was applied at the recommended field rate of 10 cm^3/100 L. The fungicide was obtained from Syngenta, Egypt.

2.1.3. Dimethomorph

Dimethomorph (Diroof 50% WDG; (E)-3-(4-chlorophenyl)-3-(3,4-dimethoxyphenyl)-1-morpholin-4-ylprop-2-en-1-one). Dimethomorph was applied at the recommended field rate of 50g/100 L. The product was provided by Asbayer, Egypt.

2.2. Chemicals and reagents

High-performance liquid chromatography (HPLC) grade solvents, including acetone, acetonitrile, methanol, ethyl acetate, dichloromethane, and acetic acid, were obtained from Merck (Darmstadt, Germany). Ultra-pure water was generated using a Millipore purification system. Anhydrous magnesium sulfate (Merck, Darmstadt, Germany) was activated by heating at 400 °C for 4 hours in a muffle furnace, then allowed to cool and stored in a desiccator until use. Primary secondary amine (PSA, 40 µm Bondesil) sorbents were supplied by Agilent Technologies (Santa Clara, CA, USA). Sodium chloride and sodium sulfate (analytical grade) were purchased from El Nasr Pharmaceutical Chemicals Company (Cairo, Egypt).

2.3. Field Trial and Sample Collection

A field experiment was carried out on 10-year-old Sultana grape trees during the summer growing season of 2023/2024 in Al-Nakrashi village, located in Itay El-Baroud district, El-Beheira Governorate, Egypt. The study was performed in an open vineyard under natural field conditions to evaluate the residue behavior of selected fungicides on the grape plants. The pesticides used in this study included boscalid, penconazole, and dimethomorph. Each was applied individually at the manufacturer's recommended field rate. Applications were performed using a knapsack sprayer equipped with a single nozzle (Model HSPP4202, Ingco, 20 L capacity) to ensure uniform distribution over the grape leaves and berries. The experiment was conducted using a completely randomized block design (CRBD), incorporating both treated plots and an untreated control. The control plots were sprayed with water instead of pesticide solutions to serve as a check control for the recovery target. All treatments, including the control, were replicated three times. Fruit and leaf samples from grapevines were randomly collected at specific intervals following pesticide application: 2 hours (considered as day 0), and on days 1, 3, 7, 10, 14, and 21.

Approximately 2 to 3 Kg of grape berries and leaves were gathered per sampling time, placed in labeled polyethylene bags, and transported to the laboratory in an icebox to preserve sample integrity. Upon arrival, the samples were homogenized and portioned into subsamples (50 g for fruit and 25 g for leaves). These subsamples

were then stored at –20 °C in a deep freezer until pesticide residue analysis was conducted.

2.4. Extraction and clean up

Pesticide residues from grape leaves and berries were extracted using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method (Anastassiades et al., 2003). Ten g of the homogenized sample were mixed with 2.5 mL of water and 2.5 mL of acetonitrile (1% acetic acid) in a 50 mL centrifuge tube. After adding 4 g $MgSO_4$ and 1 g NaCl, the mixture was vortexed and centrifuged at 2500 rpm for 5 min. at 4 °C. A 4 mL aliquot of the supernatant was cleaned using d-SPE with 100 mg PSA and 600 mg $MgSO_4$.

For further purification, 20 mL of acetonitrile and 5 g NaCl were added, followed by centrifugation at 3800 rpm. The organic layer was evaporated at 40 °C under vacuum, and the residue reconstituted in 2 mL acetonitrile. Final cleanup involved 0.3 g $MgSO_4$, 0.05 g PSA, 50 mg C18, and 0.005 g GCB, followed by filtration through a 0.2 µm PTFE filter before HPLC analysis.

2.5. Preparation of standard solution:

A stock solution of each analytic was prepared in acetonitrile at a concentration of 100 µg mL^{-1}. Working standard solutions, used for matrix fortification and instrument calibration, were obtained through serial dilution of the stock solution. All prepared standards were stored at 4 °C until analysis. Calibration curves were generated by plotting the peak area against the corresponding analytic concentrations to ensure accurate quantification

2.6. Determination and Analytical Conditions

2.6.1. HPLC analysis

HPLC analysis was conducted using an Exion HPLC system coupled with a QTRAP 6500+ mass spectrometer (AB SCIEX). Separation was achieved on a Synergi Fusion-RP C18 column (2.5 µm, 100 Å, 3.0 × 50 mm; Phenomenex) maintained at 40 °C. The mobile phase consisted of 5 mM ammonium format at pH 4, prepared in water/methanol (90:10, v/v) as phase A, and in methanol as phase B. A gradient elution was applied, starting with 100% phase A at 0 min, decreasing linearly to 0% phase A by 15 min, maintained at 0% A from 15 to 18 min, then re-equilibrated to 100% A by 20 min. The flow rate was set at 0.3 mL/min, with an injection volume of 2 µL. The total run time for each sample was 20 minutes.

2.7. Method validation

The analytical method was validated by assessing standard performance parameters, following the guidelines outlined in Guidance SANTE 11312/2021. These parameters included linearity, matrix effect, and limit of detection (LOD), limit of quantification (LOQ), accuracy, and precision. Each parameter was evaluated to ensure the reliability, sensitivity, and reproducibility of the method for pesticide residue analysis in grape matrices.

2.8. Determination and analysis conditions

2.8.1. Recovery of residues

Recovery assays were carried out by spiking control to grape berries and leaves samples with boscalid, penconazole, and dimethomorph standards at fourth fortification levels (0.01, 0.5, 2.0, and 5.0 mg/kg). Five replicates of each concentration level were processed. Samples were passed through the entire process of extraction, clean-up, and analysis. The percent of recovery was calculated by the following equation:

Recovery % = (µg) found / (µg) added x 100

The average recovery values of grape samples were used to correct all obtained values of (boscalid, penconazole, and dimethomorph) residues.

2.8.2. Linearity

For the linearity relation, a standard calibration curve of the tested pesticides was established by plotting analyte concentrations against peak areas over a concentration range of 0.001-0.100 mg L^{-1}. Calibration curve of a standard was prepared either in solvent or in blank matrixes. The fit of the calibration was plotted and inspected by calculation of the correlation coefficient, to insure that the fit is satisfactory within the concentration range of the pesticide detected.

2.8.3. Calculation of the dissipation rate, Half-life time ($t_{0.5}$), and pre- harvest intervals (PHI)

The dissipation rate of insecticides was calculated using the following formula (Sivakumar et al., 2025):

Dissipation (%) = [(Residue at initial time − Residue at given time) / Residue at initial time] × 100.

The half-life values ($t_{0.5}$) for boscalid, penconazole, and dimethomorph were estimated according to Moye et al. (1987):

$C_t = C_0 e^{-kt}$,

Where

C_t is the pesticide residue concentration at time t,
C_0 is the initial residue level immediately after application
k is the dissipation rate constant (per day).

The dissipation half-life periods were calculated using the formula:

($t_{0.5} = Ln\ (2)/k$).

PHI was calculated according to the method by Chen et al. (2016) using the equation:

$$\text{PHI} = ln\left(\frac{\text{MRL}}{C0}\right)/K$$

Where MRL is the maximum residue limit, C_0 is the initial residue concentration, and k is the rate of decomposition, calculated as k = 2.303 × b, with b being the slope of the regression line.

2.9. Statistical analysis

Residue data were expressed as means ± standard deviation (S.D.) calculated from five replicate samples. One-way analysis of variance (ANOVA) was performed to assess differences in residue levels across sampling days. The least significant difference (LSD) test at a 5% significance level ($p < 0.05$) was applied as a post-hoc test to determine significant differences between means. All statistical analyses were conducted using SPSS, with a significance threshold set at $p < 0.05$.

3. Results and Discussion

3.1. Recovery of pesticides

The recovery assays were performed on untreated grape berries and leaves spiked with boscalid, penconazole, and dimethomorph at four fortification levels (0.01, 0.50, 2.00, and 5.00 mg/kg), each in five replicates, to evaluate the accuracy of the analytical method. As presented in Table 2, the average recovery percentages for all analytes across both matrices ranged from 83.07% to 96.22%, with associated standard deviations (SD) indicating acceptable repeatability.

For boscalid, recovery in berries ranged from 88.22 % (at 5.00 mg/kg) to 96.22 % (at 2.00 mg/kg), while in leaves it ranged from 84.50 % (at 5.00 mg/kg) to 93.50 % (at 2.00 mg/kg), showing slightly lower recoveries at higher concentrations. Furthermore, penconazole showed slightly lower recoveries in leaves compared to berries, with the lowest value being 80.29 % (at 0.01 mg/kg) and the highest 91.22 % in berries. In leaves, the highest value was 90.50 % (at 5.00 mg/kg), with variability more noticeable at lower levels. Besides, dimethomorph exhibited the most consistent recoveries in berries (83.07–89.37%) across all levels. Leaves showed higher recovery at the highest fortification level (5.00 mg/kg) with 96.00 %, while other levels ranged from 83.00% to 90.91%.

Overall, the data revealed that leaves generally showed slightly more variability in recoveries compared to berries, particularly for dimethomorph and penconazole. The recovery values for all analytes in both matrices fall within the acceptable range (typically 70–120%) as per SANTE/12682/2019 guidelines, confirming the method's accuracy and reliability.

Table 1. Recovery rates of tested pesticides in grape leaves and berries under study.

Fortification Levels (mg/kg)*	Recovery percentages ± SD					
	Boscalid		Penconzole		Dimethomorph	
	Berries	Leaves	Berries	Leaves	Berries	Leaves
0.01	94.34 ± 4.77	90.29 ± 3.71	90.34 ± 4.77	80.29 ± 3.71	83.07 ± 1.62	90.91 ± 2.40
0.50	91.22 ± 1.78	89.50 ± 2.15	89.22 ± 1.78	85.50 ± 2.15	89.37 ± 1.14	83.00 ± 4.34
2.00	96.22 ± 1.78	93.50 ± 2.15	91.22 ± 1.78	82.50 ± 2.15	89.37 ± 1.14	87.00 ± 4.34
5.00	88.22 ± 1.78	84.50 ± 2.15	86.22 ± 1.78	90.50 ± 2.15	89.37 ± 1.14	96.00 ± 4.34
LSD 5%	0.92	1.24	0.73	1.47	1.05	1.85

*Each fortification level is a mean of five replicates

3.2. Method validation

The analytical procedure for detecting pesticide residues was validated following the European Commission guidelines. To confirm the method's reliability, several validation criteria were examined. These parameters were assessed to verify the method's effectiveness in accurately identifying and quantifying residues of boscalid, penconazole, and dimethomorph in grape berries and leaves.

The method validation data are shown in Table 1.The LOD for boscalid, penconazole, and dimethomorph were 0.03, 0.001, and 0.011 mg/kg, respectively. The LOQ for all three pesticides was below their established MRLs, indicating that the developed method is appropriate and reliable for residue analysis, as presented in Table 2.

Table 2. Detection and Quantification Limits (LOD and LOQ) for the Analyzed Pesticides.

Pesticides	LOQ (mg/kg)	LOD (mg/kg)	MRL (mg/kg)
Boscalid	1.00	0.030	5.00
Penconazole	0.1	0.001	0.40
Dimethomorph	0.5	0.011	3.00

LOQ = Limit of Quantification
LOD = Limit of Detection

3.3. Residual levels of boscalid, penconazole, and dimethomorph in grape berries and leaves

The initial residue levels measured two hours after pesticide application revealed concentrations of 7.823 mg/kg on/in grape berries and 8.958 mg/kg on/in leaves for boscalid. For penconazole, the initial deposits were 1.923 mg/kg in berries and 4.158 mg/kg in leaves. Dimethomorph showed initial concentrations of 5.091 mg/kg in berries and 7.261 mg/kg in leaves. These values represent the starting point for assessing dissipation dynamics and determining the persistence of each pesticide in grape plant tissues over time.

The residues and dissipation levels, including the calculated half-lives ($t_{1/2}$), of boscalid, penconazole, and dimethomorph in and on grape berries and leaves at various time intervals following pesticide application were detailed in Tables 3, 4, and 5, as well as Figures 1 through 6.

3.3.1. Residual levels of boscalid

Data in Table 3 and Fig. 1 detail the degradation of pesticide residues in grape berries and leaves over 21 days following application, providing residue levels (mg/kg), dissipation percentages (%), persistence percentages (%), and additional kinetic parameters. For grape berries, the initial residue was 7.823 mg/kg, which decreased to 6.981 mg/kg by day 1 (10.76% dissipation, 89.24% persistence) and further to 0.964 mg/kg by day 21 (87.67% dissipation, 12.33% persistence). Significant dissipation of the pesticide residue was observed over time, with a 39.12% dissipation (to 4.762 mg/kg) by day 3 and a 61.38% dissipation (to 3.021 mg/kg) by day 5. The half-life ($t_{0.5}$) is 7.0 days, indicating the time for residue to halve, with a pre-harvest interval (PHI) of 5.0 days. The degradation rate constant (K) is 0.0991, and the least significant difference (LSD 5%) is 0.81 mg/kg, with a maximum residue limit (MRL) of 5 mg/kg.

For leaves, the initial residue was 8.958 mg/kg, dropping to 7.576 mg/kg by day 1 (15.43% dissipation, 84.57% persistence) and to 0.965 mg/kg by day 21 (89.22% dissipation, 10.78% persistence). Notable reductions include 25.99% dissipation (to 6.630 mg/kg) by day 3 and 44.02% (to 5.015 mg/kg) by day 5. The half-life is 6.7 days, slightly shorter than berries, with a PHI of 6.0 days. The K value is 0.1036, and the LSD 5% is 0.78 mg/kg, with the same MRL of 5 mg/kg.

The data show a consistent decline in residues, with leaves exhibiting a marginally faster dissipation rate (89.22% vs. 87.67% by day 21) and shorter half-life, possibly due to greater exposure or metabolic activity. Both matrices remain below the MRL after the PHI, suggesting compliance with safety standards, though statistical significance (LSD) confirms observed differences are reliable.

Table 3. Initial residue deposit and residue decline of boscalid in grape berries and leaves over 21 days post-application

Days after application	Grape berries			Grape Leaves		
	Residues (mg/kg)*	Dissipation (%)	Persistence (%)	Residues (mg/kg)*	Dissipation (%)	Persistence (%)
Initial (2 hrs)	7.823	00.00	100.00	8.958	00.00	100.00
1	6.981	10.76	89.24	7.576	15.43	84.57
3	4.762	39.12	60.88	6.630	25.99	74.01
5	3.021	61.38	38.62	5.015	44.02	55.98
7	2.743	64.94	35.06	4.034	54.97	45.03
10	2.252	71.21	28.79	3.181	64.49	35.51
14	1.342	82.85	17.15	1.916	78.61	21.39
21	0.964	87.67	12.33	0.965	89.22	10.78
LSD 5%	0.81	7.97		0.78	8.02	
MRL (mg/kg) **	5					
$t_{0.5}$ (days)	7.0			6.70		
PHI (days)	5.0			6.0		
K	0.0991			0.1036		

*Means = mg/kg ± S.D. Values given are the means of five replicates.
** Maximum residue limit according to (European Commission Regulation (EU), 2022)
$t_{0.5}$= Half-Life time, ND= Not Detected, MRL= Maximum Residue Level, PHI (days) = Pre-harvest intervals, K = Rate of decomposition

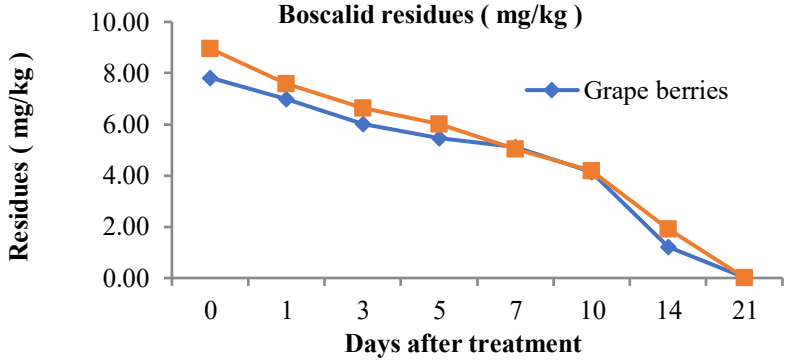

Figure 1. Decline of boscalid residues (mg/kg) in grape berries and leaves over 21 days post-treatment.

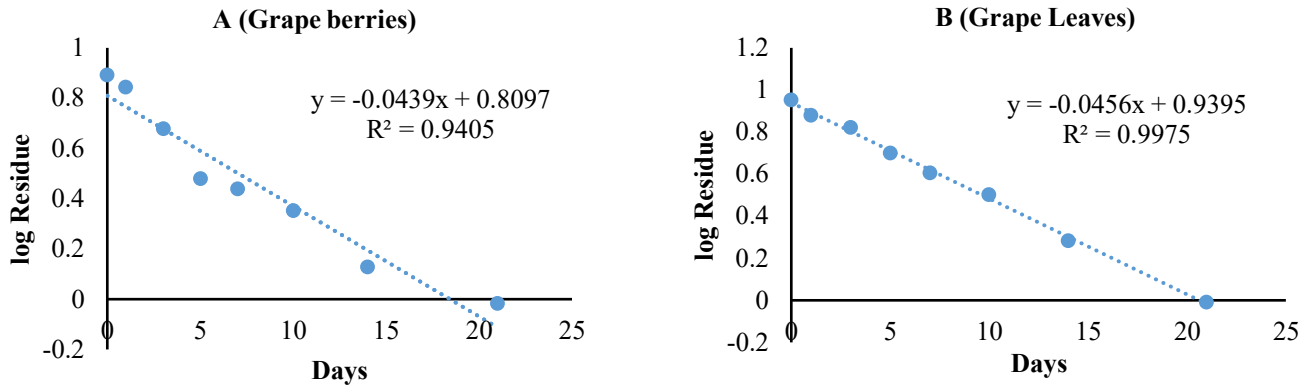

Figure 2. Logarithmic residue-day regression lines of boscalid in grape berries and leaves over 21 days post-application. A) Grape berries and B) Grape leaves. A) Grape berries and B) Grape leaves. Both graphs highlight the kinetic degradation, with R^2 values reflecting the goodness of fit for the linear models.

Data in Fig. 2 illustrated the logarithmic decrease of boscalid residues in grape berries and leaves over 25 days following application. In grape berries, the regression analysis produced a slope of -0.0439 and an intercept of 0.8097, with an R^2 value of 0.9405, reflecting a consistent decline in residue levels from approximately 0.8 log mg/kg to nearly 0 log mg/kg by day 25. In grape leaves, the slope was slightly steeper at -0.0456, with an intercept of 0.9395 and a higher R^2 of 0.9975, indicating a more uniform and slightly faster dissipation from an initial value of about 0.94 log mg/kg to close to 0 log mg/kg by the end of the period. The data points in both cases closely follow the regression lines, supporting a first-order kinetic model for the degradation of boscalid, with the dissipation in leaves being marginally more rapid and consistent.

3.3.2. Residual levels of penconazole

Results obtained in Table 4 and Fig. 3 presented the initial residue deposit and subsequent decline of penconazole in grape berries and leaves over 21 days post-application. In grape berries, the initial residue was 1.923 mg/kg, decreasing to 0.654 mg/kg by day 1 (34.01% dissipation, 65.99% persistence) and further to 0.154 mg/kg by day 14 (91.99% dissipation, 8.01% persistence), with no detectable (ND) residues by day 21 (100% dissipation). For leaves, the initial residue was 4.158 mg/kg, dropping to 2.876 mg/kg by day 1 (30.83% dissipation, 69.17% persistence) and to 0.098 mg/kg by day 14 (97.4% dissipation, 2.6% persistence), also reaching ND by day 21 (100% dissipation). The half-life ($t_{0.5}$) was 1.24 days for berries and 2.49 days for leaves, with pre-harvest intervals (PHI) of 3.0 and 8.5 days, respectively. The degradation rate constant (K) was 0.557 for berries and 0.279 for leaves, indicating a faster degradation in berries. The LSD 5% values (0.23 for berries, 0.49 for leaves) confirm statistical significance, and the MRL was set at 0.4 mg/kg for both. Residues in both matrices fell below the MRL after their respective PHIs, with berries showing a quicker dissipation rate. The higher initial residue and slower half-life in leaves suggest greater retention or slower metabolic breakdown compared to berries.

Table 4. Initial residue deposit and residue decline of penconazole in grape berries and leaves over 21 days post-application

Days after application	Grape berries			Grape Leaves		
	Residues (mg/kg)*	Dissipation (%)	Persistence (%)	Residues (mg/kg)*	Dissipation (%)	Persistence (%)
Initial (2 hrs)	1.923	00.00	100.00	4.158	00.00	100.00
1	0.654	34.01	65.99	2.876	30.83	69.17
3	0.321	83.30	16.7	1.630	60.78	39.22
5	0.298	84.50	15.5	0.423	89.83	10.17
7	0.256	86.69	13.31	0.211	94.93	5.07
10	0.193	89.96	10.04	0.181	95.65	4.35
14	0.154	91.99	8.01	0.098	97.4	2.6
21	ND	100	00.00	ND	100.00	00.00
LSD 5%	**0.23**	**6.58**		**0.49**	**6.41**	
MRL (mg/kg) **	**0.4**					
$t_{0.5}$ (days)	1.24			2.49		
PHI (days)	3.0			8.5		
K	0.557			0.279		

*Means = mg/kg ± S.D. Values given are the means of five replicates.
** Maximum residue limit according to (European Commission Regulation (EU), 2022)
$t_{0.5}$= Half-Life time, ND= Not Detected, MRL= Maximum Residue Level, PHI (days) = Pre-harvest intervals, K = Rate of decomposition

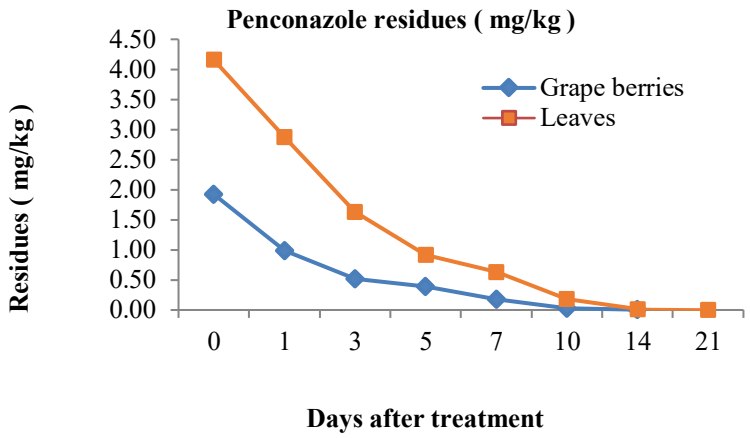

Figure 3. Decline of penconazole residues (mg/kg) in grape berries and leaves over 21 days post-treatment.

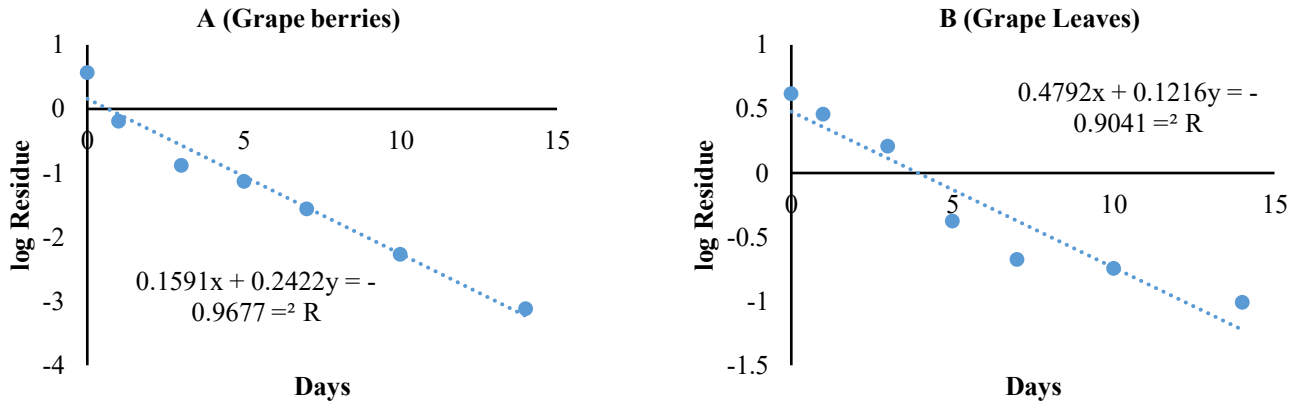

Figure 4. Logarithmic residue-day regression lines of penconazole in grape berries and leaves over 21 days post-application. A) Grape berries and B) Grape leaves. Both graphs highlight the kinetic degradation, with R^2 values reflecting the goodness of fit for the linear models.

Data in Fig. 4 clarified the logarithmic residue-day regression lines for penconazole in grape berries and leaves over 16 days. For grape berries, the regression line is described by the equation y = -0.2422x + 0.1591, with an R^2 of 0.9677, indicating a strong fit. Residue levels start near 0.5 log mg/kg and decline steadily to approximately -3.5 log mg/kg by day 16. For leaves, the regression line follows y = -0.1216x + 0.4792, with an R^2 of 0.9041, showing a good fit but a slower decline. Initial residues are around 0.5 log mg/kg, decreasing to about -1.5 log mg/kg by day 16. The steeper slope for berries (-0.2422 vs. -0.1216) suggested a faster degradation rate compared to leaves.

3.3.3. Residual levels of dimethomorph

Table 3 and Fig. 5 present the initial deposit levels and the progressive reduction of dimethomorph residues in grape berries and leaves throughout the 21 days following application. In grape berries, the initial residue was 5.091 mg/kg, decreasing to 4.891 mg/kg by day 1 (3.93% dissipation, 96.07% persistence) and further to 1.254 mg/kg by day 10 (75.37% dissipation, 24.63% persistence). By day 14, residues dropped to 0.954 mg/kg (81.26% dissipation, 18.74% persistence), with no detectable (ND) levels by day 21 (100% dissipation). For leaves, the initial residue was 7.261 mg/kg, reducing to 4.230 mg/kg by day 1 (41.74% dissipation, 58.26% persistence) and to 0.931 mg/kg by day 10 (87.18% dissipation, 12.82% persistence). By day 14, residues fell to 0.065 mg/kg (99.10% dissipation, 0.90% persistence), also reaching ND by day 21 (100% dissipation). The half-life ($t_{0.5}$) was 5.0 days for berries and 3.5 days for leaves, with pre-harvest intervals (PHI) of 4.0 and 4.5 days, respectively. The degradation rate constant (K) was 0.138 for berries and 0.200 for leaves, indicating a faster degradation in leaves. The LSD 5% values (0.62 for berries, 0.84 for leaves) confirm statistical significance, and the MRL was set at 3 mg/kg for both. Residues in both matrices remained below the MRL after their respective PHIs, with leaves showing a quicker initial dissipation.

Table 5. Initial residue deposit and residue decline of dimethomorph in grape berries and leaves over 21 days post-application

Days after application	Grape berries			Grape Leaves		
	Residues (mg/kg)*	Dissipation (%)	Persistence (%)	Residues (mg/kg)*	Dissipation (%)	Persistence (%)
Initial (2 hrs)	5.091	00.00	100.00	7.261	00.00	100.00
1	4.891	3.93	96.07	4.230	41.74	58.26
3	3.132	38.48	61.52	2.986	58.88	41.12
5	2.678	47.39	52.61	2.640	63.64	36.36
7	1.865	63.37	36.63	1.874	74.19	25.81
10	1.254	75.37	24.63	0.931	87.18	12.82
14	0.954	81.26	18.74	0.065	99.10	0.90
21	ND	100.00	00.00	ND	100.00	00.00
LSD 5%	**0.62**	**10.08**		**0.84**	**9.39**	
MRL (mg/kg) **	**3**					
$t_{0.5}$ (days)	5.0			3.5		
PHI (days)	4.0			4.5		
K	0.138			0.200		

*Means = mg/kg ± S.D. Values given are the means of five replicates.
** Maximum residue limit according to (European Commission Regulation (EU), 2022)
$t_{0.5}$= Half-Life time, ND= Not Detected, MRL= Maximum Residue Level, PHI (days) = Pre-harvest intervals, K = Rate of decomposition

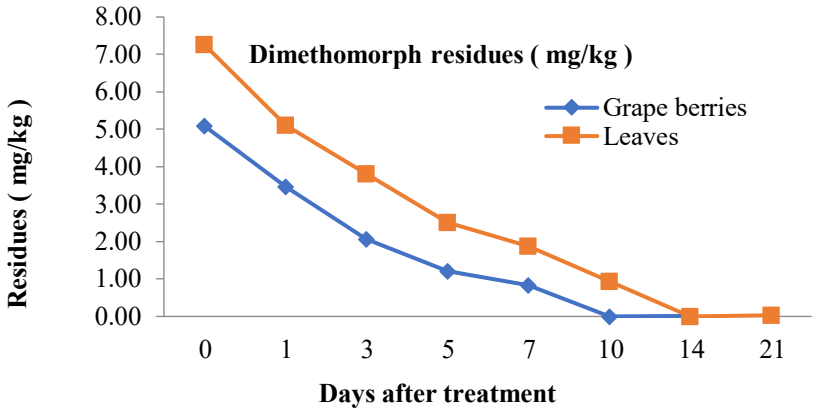

Figure 5. Decline of dimethomorph residues (mg/kg) in grape berries and leaves over 21 days post-treatment.

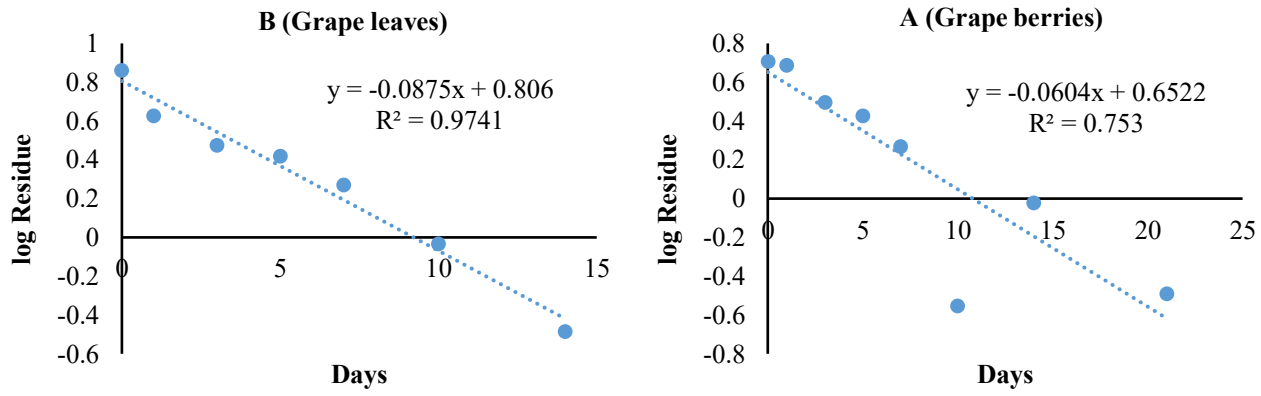

Figure 6. Logarithmic residue-day regression lines of dimethomorph in grape berries and leaves over 21 days post-application. A) Grape berries and B) Grape leaves. Both graphs highlight the kinetic degradation, with R^2 values reflecting the goodness of fit for the linear models.

Data in Fig. 6 illustrated the logarithmic residue-day regression lines for dimethomorph in grape berries and leaves over 25 days post-application. For grape berries, the regression line is defined by $y = -0.0604x + 0.6522$, with an R^2 of 0.753, indicating a moderate fit. Residue levels start near 0.8 log mg/kg and decline steadily to approximately -0.6 log mg/kg by day 25, reflecting a gradual degradation. For leaves, the regression line follows $y = -0.0875x + 0.806$, with a higher R^2 of 0.9741, suggesting a stronger fit and faster dissipation. Initial residues are around 1.0 log mg/kg, decreasing to about -0.6 log mg/kg by day 25. The steeper slope for leaves (-0.0875 vs. -0.0604) indicates a quicker degradation rate compared to berries, supported by the higher R^2 value reflecting a more consistent decline.

4. Discussion

The present study revealed that initial pesticide residues were markedly higher in grape leaves compared to grape berries, a finding consistent with previous studies (Hayar et al., 2021; Hamzawy, 2022). This disparity is likely attributed to the structural and chemical differences between the surfaces of leaves and fruits. Leaf surfaces are typically rougher and a larger surface area, which may enhance pesticide retention. Grape leaves typically show higher initial pesticide residues and slower dissipation compared to fruit, a trend attributed to differences in surface morphology and plant physiology. Specifically, leaves tend to retain more spray due to larger, rougher surfaces and different cuticular wax chemistry, while fruits often have a smoother, waxier cuticle that reduces retention and promotes faster degradation. These observations are consistent with findings by Majed et al. (2021), who reported that imidacloprid residues on vine leaves were 20–70 times higher than those in grapes, attributed to the differing structural and physiological characteristics between leaves and fruits.

The dissipation trends of boscalid, penconazole, and dimethomorph in both grape berries and leaves followed a typical first-order kinetic model, where the residue levels declined exponentially with time. These results support the general pattern observed in many field and vegetable crops, where dissipation behavior is influenced by a variety of environmental and physiological factors. Among these, photodegradation, volatilization, plant metabolism, and dilution due to fruit growth were particularly prominent. As described by Christensen et al. (2003), pesticide degradation may result from chemical, physical, and biological pathways, and in field settings, the dynamics of plant growth, such as expanding biomass, can further accelerate the decline of pesticide residues. Walgenbach et al. (1991) emphasized the role of growth dilution, especially in fruits, which expand significantly over time, diluting the concentration of pesticide residues.

Degradation rates consistently differed between leaves and berries for all compounds. Grape leaves generally exhibited higher initial deposits and slower dissipation, which may be explained by the reduced metabolic activity and absence of significant growth dilution compared to fruit tissues. The slower degradation in leaves also implies a prolonged exposure risk to non-target organisms like beneficial insects or grazing livestock, which should be considered in pesticide safety assessments. Moreover, external environmental factors such as solar radiation, temperature, humidity, and rainfall significantly influence pesticide dissipation rates. As Zepp and Cline (1977) noted, sunlight and temperature play critical roles in pesticide stability. Photolysis under UV exposure can degrade many pesticides, while elevated temperatures may enhance both volatilization and microbial degradation in the plant canopy and soil. This is particularly relevant under Egyptian climatic conditions, which are characterized by high sunlight intensity and temperature during the grape growing season, thus likely contributing to faster degradation observed in this study.

Our findings align well with those reported by Abdallah et al. (2014), who found that the initial residues of chlorfenapyr and difenoconazole were considerably higher in grape leaves than in berries, and both followed first-order kinetic degradation patterns. The reported half-lives of 1.796–4.494 days in berries and 2.359–5.143 days in leaves were in a similar range to those observed in our study for boscalid, penconazole, and dimethomorph.

Morsy et al. (2022) provided further confirmation, reporting compound-dependent variations in half-life and dissipation across grape tissues. Their study showed faster degradation of diniconazole and spinetoram compared to difenoconazole and methoxyfenozide. Their reported pre-harvest intervals (PHIs) of 7–15 days align with safety standards and residue compliance measures required under international regulations such as Codex and EU MRLs. Similarly, Chen and Zhang (2010) found that boscalid dissipated rapidly in strawberries with half-lives of 4.9–6.4 days, and residues declined below the EU-MRL within three days post-application. These findings are directly comparable to our observations in grapes, particularly with boscalid, which showed effective dissipation within the first week after treatment. In cucumbers, boscalid residues dropped to only 5–17% of initial values within six days (Chen et al., 2007), reinforcing its moderate persistence across different crops and climates.

Regarding penconazole, Hassan et al. (2013) documented a short half-life (~1.56 days) in grapes and recommended a PHI of 14 days to remain within the MRL limits. This is consistent with our data, where rapid dissipation was observed. Additional support is found in Babazadeh (2020), who reported a strong linear decline in penconazole residues in cucumber, with an $R^2 > 0.91$, indicating predictable and steady degradation. Similarly, Abdallah et al. (2021) suggested a PHI of 11.4 days for penconazole under standard and double-dose treatments, with terminal residues falling below 0.3 mg/kg after 7 days.

For dimethomorph, our results agree with Chen et al. (2018), who observed rapid dissipation in potatoes, with half-lives between 2.1 and 2.6 days. In grapes, Wang et al. (2018) recorded a half-life of 7.3 days, while Liu et al. (2019) noted considerable variability

across regions and years, with half-lives ranging from 3.69 to 28.88 days. This range highlights how local climate and agronomic practices can influence degradation behavior. Yang et al. (2022) showed translocation of dimethomorph to edible parts of the plant within 48 hours and documented distinct half-lives in tubers, leaves, and soil, emphasizing the need for multi-compartmental monitoring when assessing environmental persistence. Our $t_{0.5}$ for dimethomorph (3.5 days) aligns with Chen et al. (2018) but contrasts with Liu et al. (2019), likely due to climatic differences

4. Conclusion

Our study confirms that pesticide dissipation behavior in grapes is influenced by both intrinsic plant factors and external environmental conditions. The findings support existing literature and provide practical insights for establishing appropriate pre-harvest intervals to ensure food safety. Moreover, the observed kinetic patterns suggest that first-order models remain robust and applicable in predicting pesticide residue behavior under field conditions in arid and semi-arid climates like Egypt.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest.

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