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RESEARCH ARTICLE

Disappearance and hazard quotient of chlorpyrifos-methyl, fipronil, and imidacloprid insecticides from dates

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Abstract Control strategies of red palm weevil (RPW) mainly depend on the use of insecticides. However, the residue of insecticides in dates might cause poisoning risk to the consumers. Therefore, the present study aimed to measure the disappearance rates of the extensively applied insecticides on date palm trees. Residues of chlorpyrifos-methyl (CPM), fipronil (FIP), and imidacloprid (IMD) insecticides in and/or on dates were extracted and cleaned-up using the quick, easy, cheap, effective, rugged, and safe (QuEChERS) method. The residues of CPM, FIP, and IMD were determined by GC-FPD, GC-ECD, and HPLC, respectively. Recoveries of CPM, FIP, and IMD ranged from 86.3 to 98.2%. CPM on dates degraded faster than FIP and IMD. However, after 21 days of the last spray application, 74, 50, and 67% of CPM, FIP, and IMD disappeared, respectively. Residues of CPM and IMD insecticides posed no risk quotient, but FIP caused a risk to humans, depending on the consumption pattern of dates. Insecticides with a fast degradation pattern should be incorporated into the management strategy of RPW to reduce the amount on residues in dates.

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1 Introduction

The date palm (*Phoenix dactylifera*) is a historical plant originated from tropical and subtropical areas, especially the Arabian Peninsula (Ahmed et al. 1995; El-Juhany 2010). The major producing countries in 2014 were Egypt with 1.5 million metric tons (MMT), Iran (1.2 MMT), Algeria (0.93 MMT), Saudi Arabia (0.77 MMT), and Iraq (0.66 MMT). The worldwide production was 7.60 MMT (FAOSTAT 2017). Dates production and industrial processing are increasing worldwide and they are consumed mainly as fresh fruits. Consumption of date fruits provides consumers with significant amounts of phytochemicals, proteins, vitamins, and minerals and other nutrients that have abundant health benefits (El-Juhany 2010; Vayalil 2012).

Worldwide, the annual yield of dates is considerably reduced by insect pests particularly important is the infestation with the red palm weevil (RPW) (Jaradat and Zaid 2004). The RPW, *Rhynchophorus ferrugineus* (Olivier), is a very destructive pest for date trees in the Middle East and North Africa. Integrated management programs include several strategies:

- Pheromone-trapping (Zada et al. 2002; Oehlschlager 2007),
- release of sterile males of *R. ferrugineus* (Krishnakumar and Maheswari 2007),
- biocontrol agents (*Bacillus sphaericus* (Salama et al. 2004),

- *Beauveria bassiana* (Sewify et al. 2009),
- the nematode *Steinernema carpocapsae* in a chitosan formulation (Llácer et al. 2009), and
- the primarily use of chemical insecticides (Al-Shawaf et al. 2011, 2013; Barranco et al. 1998; Giblin-Davis et al. 2013; Kaakeh 2006; Murphy and Briscoe 1999; Rifai et al. 2000; Shar et al. 2012).

However, data on pesticide residues in and/or on dates were investigated in few studies. For example, the residues of abamectin in dates were less than the maximum residue limits (MRLs) after 14 days of spray, while amitraz and flufenoxuron residues were detected even after 30 and 60 days of spray, respectively (Kamel et al. 2007). Also, Abd Rabou et al. (2015) found that the residues of chlorpyrifos, diazinon, and FIP insecticides were below the MRLs after 30 days of injection into trees' trunk.

Therefore, the recommendation of the use of specific insecticide to control RPW should take into consideration its residue in date fruits to avoid the risk of adverse effects to consumers. Consequently, the present study aimed to (1) measure the residue amounts of extensively used insecticides against RPW including chlorpyrifos-methyl (CPM), fipronil (FIP), and imidacloprid (IMD) following different intervals of the last field application and (2) estimate the expected risk to human after consumption of contaminated date fruits.

2 Materials and methods

2.1 Insecticides and chemicals

Chlorpyrifos-methyl (CPM; Gentel 50% EC, Dow AgroSciences), fipronil (FIP; fipronil 20% SC, MAC-GmbH, Germany), and imidacloprid (IMD; Sinodor 70% WG, Jiangsu Guoxin Group Ltd., China) were purchased from local suppliers. All solvents and chemicals were of HPLC-grade and purchased from reputed chemical suppliers in Egypt.

2.2 Field application of insecticides

2.2.1 Foliar spray application

The date palm trees used in this study were about 15 years old (Zaghloul variety). The experimental units (date palm trees) were treated once a week for 3 weeks by foliar application with the recommended rates of each insecticide (MALR 2016) of CPM (3 ml/l of Gentel 50% EC = 1500 mg/l a.i.), FIP (3 ml/l of FIP 20% SC = 600 mg/l a.i.), or IMD (3 g/l of Sinodor 70% WG = 2100 mg/l a.i.). The application was carried out using a Knapsack sprayer (CP-3; Cooper Pegeler, Spraying Technology, UK) equipped with one nozzle. The sprayer was calibrated for pressure (15 psi) and nozzle following the method described by Landgren (1987). Each treatment had three replicates of five trees. Control trees were sprayed with an equal volume of water.

2.2.2 Application of insecticides via trunk injection

The same palm tree variety (Zaghloul) was injected with the field application rates of CPM, FIP, or IMD (as mentioned before) once every 5–7 days into 2.5 cm diameter holes (MALR 2016). Each treatment had three replicates with five trees within each replicate. Control trees were injected with an equal amount of water. Inside the tree, holes were drilled 1.5 cm above the insect attacking point on the same side of infestation with a diameter of 2.5 cm and 10 cm deep. About 20–25 ml of each insecticide was injected per hole (the volume that filled the hole). Then, the holes were closed with cotton plugs and covered with plastic masking tape in order to prevent any infection. All agricultural practices were done as that followed in the commercial production of palm date crop.

2.3 Date samples collection and preparation

Approximately 2 kg of date palm fruits were randomly collected from each tree 1 h (initial time) and 1, 2, and 3 weeks after the last foliar spray of the insecticides. Samples were transferred to the laboratory at 4 °C. Seeds were discarded and flesh was homogenized and stored at −20 °C until analysis. The insecticide residues were extracted upon reaching the laboratory within 5 h of collection.

2.4 Insecticides extraction and clean up

Extraction and clean up of CPM, FIP, and IMD residues was done by using a modified QuEChERS method (Paya et al. 2007). A sample of 10 g of date flesh was extracted with 10 ml of acetonitrile (acidified with 1% acetic acid) in 50 ml falcon screw cap tubes. Tubes were vortexed for 1 min. Then 1 g of sodium chloride and 4 g of magnesium sulfate anhydrous were added. Samples were vortexed for 10 min and then centrifuged for 10 min at 4000 rpm and 4 °C. Aliquots of 1 ml of the supernatant were transferred into 15 ml centrifuge tubes for clean up by dispersive solid-phase extraction with primary secondary amine (PSA; 25 mg), graphite carbon black (GCB; 10 mg), and $MgSO_4$ (150 mg). The tubes were vortexed for 10 min and then centrifuged as mentioned before. The supernatant was filtered through 0.22 μm PTFE filter (Millipore, USA) into a 1.5 ml amber HPLC glass vial, and kept at −20 °C until analysis of CPM and IMD. For FIP, 1 ml of the supernatant after centrifugation was dried at 35–40 °C under a gentle current of nitrogen and the residue was re-dissolved in 2 ml of toluene. Then samples were filtered in the PTFE filters as mentioned above.

2.5 Analysis of CPM and FIP residues

Quantitative analysis of CPM residues was done using a gas chromatography system with a DB1701 (30 m × 0.25 mm × 0.25 μm; Agilent Technologies Ltd., CA, USA) column and equipped with flame photometric detector (GC–FPD) (Agilent 7890, Agilent Technologies, CA, USA). Gas chromatography conditions were: injection port temperature 220 °C: column temperature: maintained at 160 °C for 2 min and programmed at 15 °C/min to 260 °C, and held for 10 min at 240 °C. The detector temperature was 250 °C, flammable gas flow rates were 100 (hydrogen) and 75 (air) ml/min, and the flow rate of the carrier gas was 3 ml/min (nitrogen) (Abbassy et al. 2015).

FIP residues were determined using a gas chromatography system with an electron-captured detector (GC–ECD) (Agilent 7890, Agilent Technologies, CA, USA) that was equipped with HP5 (30 m × 0.25 mm × 0.25 μm) column. The column temperature was maintained at 160 °C for 2 min, programmed at 15 °C/min to 260 °C, and held for 10 min. Injection port temperature was 300 °C and the detector temperature was set up at 320 °C. The carrier gas was nitrogen at a flow rate of 3 ml/min (Kurz et al. 2013). The residue amounts of CPM and FIP in date samples were calculated using standard curves depicted by standard materials of both insecticides.

2.6 Determination of IMD residues

The estimation of IMD concentration in date fruit samples was done using an Agilent 1260 High Performance Liquid Chromatography (HPLC) system (Agilent Technologies, CA, USA) that was equipped with a quaternary pump, an auto sampler injector, thermostat compartment for the column, and photodiode array detector. The chromatography column was Zorbax C18 XDB (250 mm × 4.6 mm × 5 μm film thicknesses; Agilent Technologies, CA, USA). The column was kept at room temperature and the flow rate of mobile phase (acetonitrile:water (60:40 v/v)) was 1 ml/min, and the injection volume was 20 μl. IMD was detected at 270 nm. The residue concentrations of IMD were identified by comparing the areas and retention times (RTs) of the sample peaks with the RTs of standard (active ingredient) on a standard curve (Nassar et al. 2015).

2.7 Recovery studies (validation and quality control)

The analysis method was validated before using for the analysis. Untreated date fruit samples were spiked with three concentrations: 0.1, 0.5 and 1.0 mg/kg in five replicates each of CPM, FIP, or IMD prior to extraction and clean up of the insecticides (Table 1). These samples were extracted, cleaned-up, and analyzed as described for unknown samples. The recovery values were calculated using the following formula:

$$\text{Recovery}\ \% = \frac{\mu\text{g pesticide residue/g sample found}}{\mu\text{g pesticide residue/g sample added}}.$$

The method precision parameters in terms of average recovery and relative standard deviation were calculated and assessed following the European Union guidelines (Pihlström 2011).

2.8 Half-life time values

The half-life time values of tested insecticides were estimated following the model described by Su et al. (1999).

$$C_t = C_0 \times (0.5)^{(t/k)}$$

C_t was the insecticide concentration at time t, C_0 is initial concentration, and k is the half-life index or time required for 50% insecticide degradation. The model was linearized by calculating the natural logarithm, $\ln C_t = \ln C_0 + (t/k) + \ln(0.5)$ or $Y = A + B + t$, where $Y = \ln C_t$, $B = [\ln(0.5)]/k$, and $A = \ln C_0$.

2.9 Estimated dietary exposure dose (EED) and risk quotient (RQ)

Dietary exposure calculation and risk assessment were calculated using the following equations:

$$\text{EED} = \text{calculated residue limit (mg/kg)} \times \text{food intake (kg/capita/day)},$$

Table 1 Mean recovery percentage ± RSD of CPM, IMD, and FIP from dates fortified with various levels of each insecticide

Fortification level (mg/kg)	CPM	IMD	FIP
0.1	96.70 ± 2.42	89.70 ± 2.24	86.30 ± 2.15
0.5	98.15 ± 2.45	92.50 ± 2.31	89.50 ± 2.24
1.0	91.71 ± 2.29	90.30 ± 2.26	93.80 ± 2.35

Table 2 Half-life ($t_{1/2}$; day), maximum residue limits (MRL; mg/kg), and acceptable daily intake (ADI; mg/kg/day)

Insecticide (application method)	$t_{1/2}$ (days)[a]	MRL (mg/kg)[b]	ADI (mg/kg/day)[b]
CPM (spray)	10.74	0.05	0.01
FIP (spray)	22.94	0.005	0.0002
FIP (injection)	27.70		
IMD (spray)	13.88	0.05	0.06
IMD (injection)	21.12		

[a] $t_{1/2}$ calculated half-life

[b] MRLs and ADIs values were obtained from the Codex Alimentarius International Food Standards website at http://www.codexalimentarius.net/pestres/data/pesticides/details.html?id=202

$$RQ = EED/\text{acceptable daily intake (ADI) (mg/kg bw)}.$$

Based on the summary report of the Food and Agriculture Organization, the average estimated standard dates intake for an Egyptian adult (an average body weight of 60 kg) in 2011 was 42 g (0.042 kg/capita/day). The acceptable daily intake (ADI) for CPM, FIP, and IMD was 0.01, 0.0002, and 0.06 mg/kg/bw/day (Codex Alimentarius Commission 2016) (Table 2). An RQ value higher than 1 indicates that the risk of a pesticide for humans is unacceptable while an RQ value less than 1 represents minimal risk to humans (Zhang et al. 2009).

2.10 Statistical analysis

Results of the residue analysis of CPM, FIP, and IMD were statistically analyzed as a repeated measure design over time with the insecticide residue levels as the main factor over different time intervals using the MIXED procedure of the statistical analysis system SAS (SAS 2013). Significant means of residue amounts were compared using Tukey's studentized range (HSD) test at $p \leq 0.05$.

3 Results and discussion

3.1 Recovery percentages of insecticides and method validation

The mean recoveries of CPM, IMD, and FIP insecticides in/on dates ranged from 92 to 98, 90 to 93, and 86 to 94%, respectively (Table 1). The relative standard deviation (RSD) ranged from 2.15 to 2.45%, which was within the acceptable limits for routine analysis of pesticide residues (Codex Alimentarius Commission 2016). Samples were quantified using five dilutions of standard materials of the tested insecticides with concentrations of 0.01–2 mg/kg.

3.2 Half-life

The half-lives ($t_{1/2}$) for CPM, FIP, and IMD are shown in Table 2. After the spray application, $t_{1/2}$ was 10.74, 13.88, and 22.94 days for CPM, IMD, and FIP, respectively. Whereas after insecticide injection into the tree trunk, $t_{1/2}$ was 21.12 and 27.7 d for IMD and FIP, respectively. CPM residues in dates of injected trees were not detected.

3.3 Insecticide residues in dates

After the injection into the palm trees, CPM residues were not detected in the dates. Results in Table 3 and Fig. 1a showed the residue level of CPM in dates after 1 h (initial time), and 7, 14, and 21 days after the last spray application. The level of CPM was 0.382, 0.322, 0.192, and 0.101 mg/kg after 1 h, and 7, 14, and 21 days after the last application on trees, respectively. That corresponds to 0, 15.7, 49.7, and 73.6% of disappearance, respectively. The residue level of CPM in dates after 21 days of spray was twice as high as its MRL (0.05 mg/kg). Statistical analysis revealed that the residue amount after 1 h and 7 days were similar but significantly different from those after 14 and 21 days, which were not significantly different. These findings are in agreement with the reports by EL-Saeid and AL-Dosari (2010). According to that study, the residue of CPM in dates collected from local markets exceeded the MRLs. However, Al-Samarrie and Abo-Akela (2011) reported that the residues of CPM were detected after 100 days of injecting the insecticide into the tree's trunk. Long-term persistence of other insecticides was also reported in dates; e.g., dimethoate was detected after 60 days of last injection application into the trunk and disappeared after 75 days (Khan et al. 2001).

Table 3 LS-means for residues (mg/kg), estimated exposure dose (EED; mg/kg/bw/day), and risk quotient (RQ; mg/kg) of CPM, FIP, and IMD in dates after different time intervals of the last application of date palm trees

Insecticide application method	Time after application (days)	Residues ± SE (mg/kg)[1]	EED (mg/kg/bw/day)[2]	RQ (mg/kg)[3]	Health risk
CPM					
Spray	Initial (1 h)	$0.382^{a} \pm 0.122$	–	–	–
	7	$0.322^{a} \pm 0.122$	–	–	–
	14	$0.192^{b} \pm 0.122$	0.00013	0.0134	No
	21	$0.101^{b} \pm 0.122$	0.00007	0.0071	No
FIP					
Spray	Initial (1 h)	$3.12^{a} \pm 0.122$	–	–	Yes
	7	$2.04^{b} \pm 0.122$	0.00143	7.1400	Yes
	14	$1.92^{b} \pm 0.122$	0.00134	6.7200	Yes
	21	$1.57^{b} \pm 0.122$	0.00110	5.4950	Yes
Injection	Initial (1 h)	$0.042^{c} \pm 0.122$	–	–	No
	7	$0.052^{c} \pm 0.122$	0.00004	0.1820	No
	14	$0.035^{c} \pm 0.122$	0.00002	0.1225	No
	21	$0.026^{c} \pm 0.122$	0.00002	0.0910	No
IMD					
Spray	Initial (1 h)	$2.86^{a} \pm 0.122$	–	–	No
	7	$2.74^{ab} \pm 0.122$	–	–	No
	14	$2.40^{b} \pm 0.122$	0.00168	0.0280	No
	21	$0.93^{c} \pm 0.122$	0.00065	0.0109	No
Injection	Initial (1 h)	$0.450^{de} \pm 0.122$	–	–	No
	7	$0.612^{cd} \pm 0.122$	–	–	No
	14	$0.383^{de} \pm 0.122$	0.00027	0.0045	No
	21	$0.245^{e} \pm 0.122$	0.00017	0.0029	No

Means were compared using Tukey's studentized range (HSD) test ($p \leq 0.05$). Means with the same superscript letter are not significantly different

[1] Standard error

[2] EED = calculated residue limit (mg/kg)

[3] RQ = EED/ADI (mg/kg bw)

Residue amounts and the disappearance pattern of FIP are shown in Table 3 and Fig. 1a. After last spray application, residue levels were 3.12, 2.04, 1.92, and 1.57 mg/kg after 1 h, 7, 14, and 21 days, which corresponded to 0, 34.6, 38.5, and 49.6% of disappearance, respectively. After injecting FIP into palm trees trunk, the residue levels were reduced compared to the levels after spray application (0.042, 0.052, 0.035, and 0.026 mg/kg, respectively). Moreover, residue amounts after the spray application were not difference after 1 h, 1, and 2 weeks, but there was a statistically difference in the residues after 3 weeks, while there was no statistical difference in FIP residues after 1 h, 1, 2, and 3 weeks.

IMD residues were 2.86, 2.74, 2.40, and 0.93 mg/kg after 1 h, 7, 14, and 21 days, respectively, after the last spray with disappearance percentages of 0, 4.2, 16, and 67.4%, respectively (Table 3; Fig. 1c). Residues in date fruits of IMD-injected trees were less compared to those detected in dates from sprayed trees: 0.450, 0.612, 0.383, and 0.245 mg/kg after 1 h, 7, 14, and 21 days, respectively, with corresponding disappearance percentages of 0, −36.0, 14.9, and 45.6% after the specified time intervals. In the case of spraying IMD, there was a significant difference in the amount of residues after 1 h, 2, and 3 weeks, while residues after 1 h and 1 week were statistically similar. On the other hand, after the injection application, no differences were reported among residues after 1 h, 1 and 2 weeks of last application, but the residues after 1 h and 1 week were significantly different from those after 3 weeks.

Data reported in the current study revealed that the half-life time depends on the application method. The residue level of FIP in dates after 21 days of last spray or injection was 314- and 5.2-

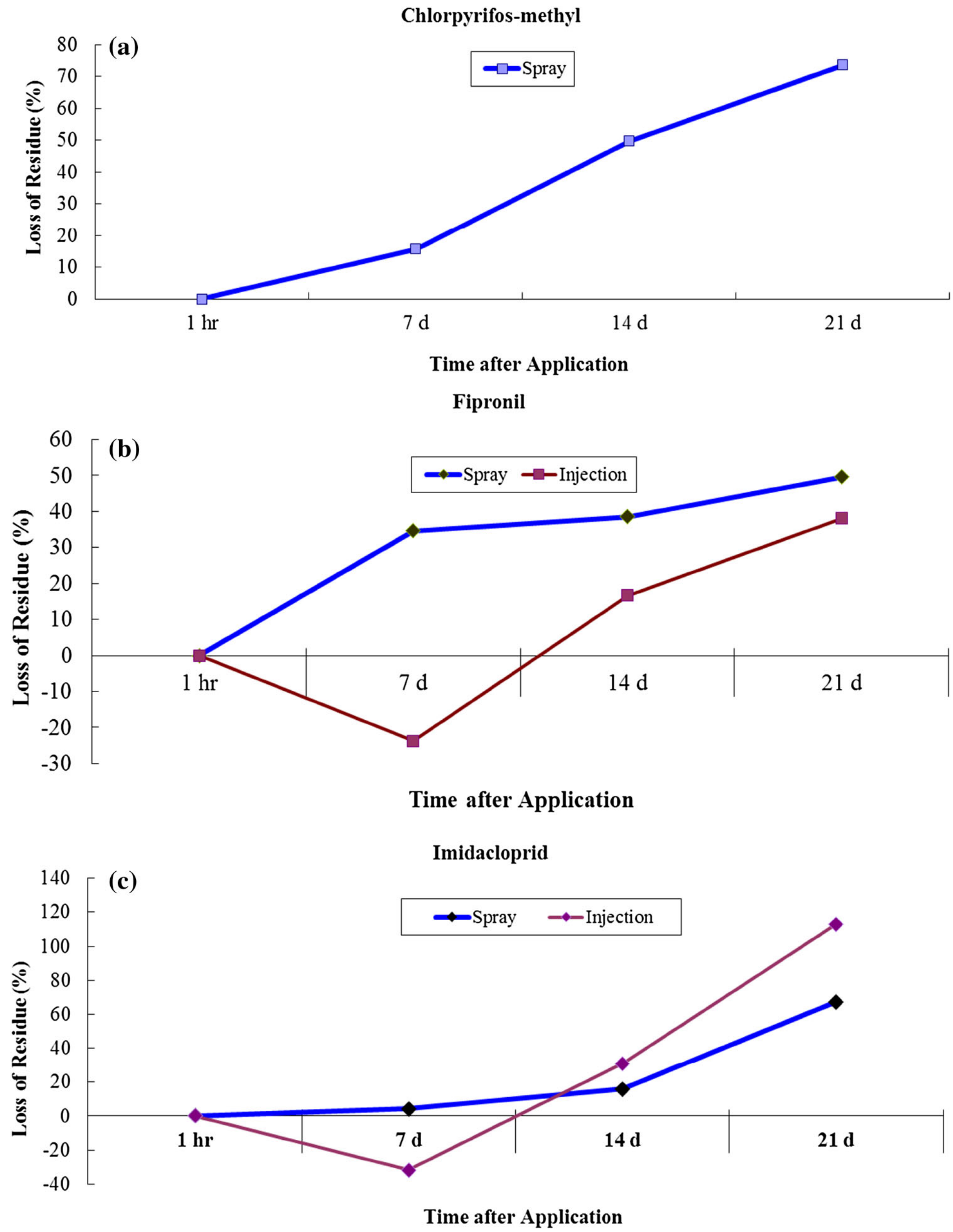

Fig. 1 Percentages of loss of residues of chlorpyrifos-methyl (**a**), fipronil (**b**), and imidacloprid (**c**) insecticides, which were applied onto date palm trees either as injection or general spray from date fruits after different time intervals post application

times higher than its MRL (0.005 mg/kg) (Codex Alimentarius Commission 2016). Also, statistical analysis showed that residue amounts of IMD after spray application were higher than after injection application. The residue amounts of IMD detected in dates after 21 days from the spray and injection were 18.6 and 4.9 times higher than MRL (0.05 mg/kg), respectively (EU Pesticides database 2014). Based on our study results, the half-life time ($t_{1/2}$) of IMP and FIP insecticides were less after the spray compared to injection. Consequently, risk assessment calculation revealed that the studied insecticides pose negligible risk to humans if they have been applied at the recommended rates.

3.4 Risk assessment

The health effects (RQ values) of CPM, FIP, and IMD residues in or on dates were calculated (Table 3). Results showed that the RQ values of CPM and IMD residues in dates from sprayed or injected trees and FIP residues in dates from injected trees were significantly lower than 1. These results suggest that their risk in dates at the recommended dosage was negligible to humans. These results were similar to that of Tchounwou et al. (2002). They reported that there were no health effects of CPM measured in water, milk, orange, or fish samples. On the contrary, the present findings showed that spraying FIP on date trees revealed RQ values significantly greater than 1, which highlights elevated risk to humans.

4 Conclusions

Residue analysis of CPM, FIP, and IMP insecticides showed that CPM disappeared faster than FIP and IMD in date palm fruits. Noteworthy, CPM and IMD revealed no risk whether applied as spraying on or injection into the trees trunk. However, FIP showed contradicting results for the two application methods. Injecting FIP into the trunk with a recommended dose did not show any health risks but spraying it on the palm trees exhibited health risk to human. This study highlights that the integrated pest management program (IPM) would be the suitable strategy to control RPW, which include early detection strategies, resistant varieties, biological agents, and insecticides and aggregation pheromones. More importantly, harvesting and marketing of date fruits should be done after at least 14–21 days from the last spray of the trees.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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