RESEARCH ARTICLE

• Arch Hyg Sci 2018;7(3): 165-173
• Journal Homepage: http://jhygiene.muq.ac.ir

Food Safety Evaluation of Imidacloprid Residue in Grape Berries at a Different Dose of Spraying

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A-R-T-I-C-L-EI-N-F-O

Article Notes:
Received: Jun 30, 2018
Received in revised form: Agu 20, 2018
Accepted: Sep 19, 2018
Available Online: Oct 7, 2018

Keywords:
Food Safety,
Confidor,
Grape,
Toxin,
Imidacloprid,
Maximum Residue Limit (MRL);
Iran

A-B-S-T-R-A-C-T

Background & Aims of the Study: Grape, a crucial agriculture crop of Malayer, is affected by Vine cicada, Psalmocharias alhageos. Imidacloprid, a neonicotinoid insecticide, provides good management of this insect. The aim of the current study was to residue persistence of imidacloprid on grapes, to estimate its residue deposit, the half-life of degradation and safe pre-harvest consumption time.

Materials & Methods: Residues of imidacloprid were estimated in grape following two spraying of recommended (80.0 g a.i. ha^{-1}) and doubles the application rate (160.0 g a.i. ha^{-1}). Samples were collected at 1 h to 21 days after spraying of imidacloprid. The analyses were done by the (QuECHERS) technique using HPLC-UV.

Results: The average initial concentrations of imidacloprid on grapes were found to be 10.58 and 17.56 mg kg^{-1} at single and double dosages, respectively. These residues of imidacloprid decreased to be the extract of 97.8% and 98.0%, respectively, at single and double dosages in 15 days, with a half-life period of 2.21 and 2.94 days. Residues of imidacloprid on grapes were less than its MRL value after 7 and 10 days of it's spraying at the recommended and double dosage. Residues of imidacloprid in grape berries at harvest were discovered to below the determination limit.

Conclusions: Consequently, a waiting time of 7 and 10 days is usually recommended for safe consumption of grapes once imidacloprid spraying. Acceptable daily intake (ADI) of imidacloprid is 0.06 mg kg^{-1} body weight day^{-1}. According to the results of this study, the employment of imidacloprid on the grape crop looks to be toxicologically acceptable.

Please cite this article as: Hassanzadeh N, Bahramifar N, Mohammad Zaheri F. Food Safety Evaluation of Imidacloprid Residue in Grape Berries at a Different Dose of Spraying. Arch Hyg Sci 2018;7(3):165-173

Background

In Malayer city (West Iran), grape (*Vitis vinifera L.*) is the most significant farming crop and the space under grape cultivation is more than 10000 ha. Among all the fruit crops, the grape has emerged as the most successful commercial crop in the recent years (1). Vine cicada, *Psalmocharias alhageos* (Hem., Cicadidae), is one of the most important pests of vineyards in Malayer. Main harm of P. *alhageos* is caused by long feeding time of nymphs on the vine roots and laying eggs of females under the skin of the shoots (2,3).

Imidacloprid 1-(6-chloro-3-pyridylmethyl)-N-nitroimidazoli- din-2-ylideneamine (fig.1), is a systemic neonicotinoid insecticide with soil, seed and foliar uses for the mastery of sucking pests, because the active ingredient is stored in plant tissues for 2 or 3 months (4–6,7). It is widely used in Malayer vineyards for the control of *Psalmocharias alhageos* (2)

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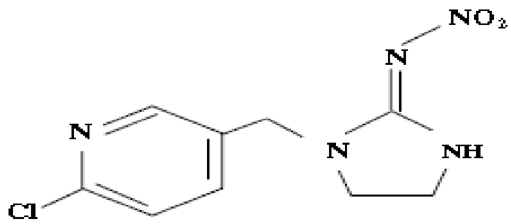

Figure 1) Structure of imidacloprid

Their chemical properties, particularly their high water solubility and partitioning properties (low log KOW) and low soil adsorption (log KOC), promote movement of these insecticides through the surface and subsurface runoff and result in extended persistence under simulated environmental conditions (8).

It is commonly used on rice, cereals, maize, potatoes, vegetable, sugar beet, fruit, cotton, hops and turfs and is very general once used as a seed or soil treatment. Imidacloprid residues have reportable in numerous agriculture crops (9–12). However, when the appliance of pesticides in agricultural fields, residues will be absorbed and continue plants, inflicting a possible hazard for human health (5). To minimize the adverse result of pesticides residues on human and setting, the food commodities treated with pesticides are strictly controlled by the authorities worldwide.

Imidacloprid is intended to be effective by contact or intake. Imidacloprid acts on many forms of post-synaptic nicotinic neurotransmitter receptors within the system. In insects, these receptors are placed only within the central nervous system. Following unreturn binding to the receptors, nerve impulses are spontaneously discharged initially, followed by failure of the neuron to propagate any signal. Sustained activation of the receptor results from the inability of acetyl cholinesterases to break down the chemical (7,13). Imidacloprid is extremely low in toxicity via dermal exposure, and moderately poisonous if eaten; but upon inhalation, its toxicity is variable (14,15). Toxicological studies of imidacloprid are limited and acceptable daily intake (ADI) was before reported as 0.06 mg/kg/day (14).

Due to the high consumption of imidacloprid in agriculture, now a day, there has been a growing interest in detective work and quantifying of insecticide residues in agricultural produce supposed for human consumption (16). The increasing society concern over the potential health risk associated with exposure to pesticides has led to the strict regulation of maximum residue limits (MRLs) of pesticide residues in food crops. When these pesticides are applied according to good agricultural practices, MRLs are not exceeded, but their wrong application may leave harmful residues, which involve possible health risk and environmental pollution (17,18).

Aims of the study:

Based on the high use of imidacloprid in the infected vineyards to cicada in Malayer, therefore, it is necessary to ensure that the residue concentration of imidacloprid in grape does not pose any risk to consumers.

The present studies were undertaken to determine the residues of imidacloprid on grape following its applications at the minimum effective and double the minimum effective dose. Data were obtained to cover a range of pre-harvest intervals (PHIs) 21 days and the results were compared with the Codex Maximum Residues Limits (MRLs) proved for this insecticide in grape. The purpose of this study was to imidacloprid residue on grapes, to determine its residue deposit, the half-life of degradation, safe pre-harvest interval and harvest time residues for use of this fruit after its multiple spraying.

Materials & Methods

Preparation of the Suspension of imidacloprid and their Application in vineyards

Field experiments were conducted during 2017 on vineyards cultivar at Malayer, Iran. At first, the vineyards infected with Vine cicada were chosen in late July. At this stage, the nymphs of cicada existed from the soil, so Imidacloprid poisoning was sprayed onto the vine trees.

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Three replicate have been selected for each treatment i.e. control, recommended dose and double the recommended dose. The treatments were untreated control, effective and double effective dose of Confidor 200 SL at 80 and 160 g a.i. ha^{-1}. For every treatment 10 plants were choose. The spray extent taken was 1000 L ha^{-1}. Grape control samples have been sprayed with water.

Chemicals and Reagents

Pesticide analytical standard of imidacloprid (purity P≥99.9%) was purchased with the purity certified by using Dr Ehrenstorfer Inc. (Augsburg, Germany). Individual pesticide stock solution (1000 µg ml^{-1}) have been prepared in pure acetonitrile (MeCN) and stored at −14 °C. Intermediate and working standard solutions of imidacloprid were prepared in acetonitrile. Calibration solutions were prepared with different concentrations simply earlier than the use. HPLC grade acetonitrile and Analytical grade anhydrous magnesium sulfate ($MgSO_4$) (99%) and sodium chloride (NaCl) (99%), were purchased from Merck (Darmstadt, Germany). Graphitized carbon black (GCB, 400 meshes) was obtained from Supelco. All glassware turned into rinsed with high purity acetone earlier than the use.

Sample Collection

On every sampling day, about 200 g grapes had been collected from each vine. The samples from three replicate treatments were pooled to make a sample size of 5 kg. The treated grapes have been analyzed at 0 (1 h), 1, 3, 5, 7, 10, 13, 16, 19 and 21 days of the application of the insecticide. Grapes were gathered at harvest time. Matured grapes had been analyzed at harvest. The accumulated grapes were placed in polyethylene bags and transferred to the laboratory after harvest and analyzed straight away.

Extraction procedure

The quick, easy, cheap, effective, rugged and safe (QuEChER) technique involves an acetonitrile partitioning and dispersive solid-phase extraction (d-SPE) which let in the simultaneous evaluation of a large range of insecticides in a variety of meals matrices. This technique was done as defined by other authors (5,19). According to this technique, the sample (1.00 kg of grapes) was chopped and homogenized for 5 min at high speed in a laboratory Homogenizer. A correctly weighed amount of 10.0 g of homogenized sample was placed into a 50 ml centrifuge tube with 10 ml of acetonitrile. The screw cap was closed and the tube was shaken vigorously for 1 min by hand, ensuring that the solvent interacted well with the entire sample. Then 4.00 g of anhydrous $MgSO_4$ and 1.00 g of NaCl was added, repeating the shaking process again for 1 min to prevent coagulation of $MgSO_4$. After centrifuging at 5000 × g for 5 min in 4◦C, the upper layer was cleaned by dispersive solid-phase extraction with 0.5 g of GCB and 1.50 g of anhydrous $MgSO_4$. The mixture was then shaken for 1 min and centrifuged for 5 min at 5000 ×g. The extract was filtered through a 0.45 µm PTFE filter and transferred to a vial. The cleaned extract sample was concentrated to 1.0 ml with a gentle stream of ultra-pure nitrogen gas and then 20 µL of this solution become injected into HPLC.

Apparatus

The residues of imidacloprid have been determined using HPLC (Shimadzu Corporation, Kyoto, Japan), version LC 10A Dual Pump with the UV-VIS detector using the reversed phase C-18, (Purospher Star RP-18, 250-4 mm i.d., 5 mm; Merck, Darmstadt, Germany) column. The mobile phase consisted of acetonitrile:water (40:60, v/v) with the solvent flow rate of 1mL min^{-1} and the detector was set at a wavelength of 270 nm. The extent becomes 20 mL. The residues of imidacloprid were estimated in different substrates by comparison of the peak height of the sample with that of standard imidacloprid run under identical conditions. The percent recovery study of pesticide at different fortification levels was evaluated in order to assess the extraction efficiency of the method. Grape fruits from

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control plots had been spiked with imidacloprid at stages of 0.05, 0.10 and 0.20 mg kg^{-1} in triplicate. The same extraction method and HPLC conditions were applied to both the sample analyses and recovery studies. The mean recoveries of imidacloprid fortified at these levels were found to be consistent and more than 89%, with relative standard deviation (RSD) values below 13%. The limit of detection (LOD) of 0.05 mg kg^{-1} was obtained.

Results

The percent recovery of imidacloprid in grape berries is given in Table 1. By following the analytical method defined the recovery of imidacloprid residues in grape berries was in the range of 87-93.2%.

Table 1) Percent recovery of imidacloprid from spiked samples of grape

Substrate	Level of fortification (mg kg^{-1})	Recovery, % ^{a} (Mean±SD)
	0.05	87 ± 4.32
Grape samples	0.10	89 ± 3.58
	0.20	93 ± 5.04

^{A} Each value in the mean±standard deviation of the three replicates determinations

Following application of imidacloprid (Confidor 200SL) at 80 and 160 g ha^{-1} 1,000 L^{-1} of water led to common initial deposits of 10.58 and 17.56 mg kg^{-1} of imidacloprid on grape berries at single and double dosages, respectively. The results of imidacloprid residue analyses and the percent dissipation at different periodic intervals at single and double dosages are provided in Tables 2 and 3. These residue levels of imidacloprid dissipated to the extent of greater than 88% for the duration of one week.

Imidacloprid residue concentrations in grapes obtained in the dissipation observe with the corresponding first-order decay fits are supplied in Figs. 2 and 3. The half-life of imidacloprid in specific matrices was calculated the use of the first order rate equation: $C_t=C_0e^{-kt}$.

In which C_t represents the concentration of the pesticide residue at time t, C_0 represents the initial concentration and k is the rate constant per day. The half-life ($t_{1/2}$) was determined from the k value for each experiment, where $t_{1/2}=\ln 2/k$. The fitness of the data to first order kinetics was confirmed by testing the statistical significance of a correlation coefficient.

The degradation kinetics of this insecticide deposit were well described by first-order decay equation, ($C(t)= 13.578\times e^{-0.333\times t}$, $R^2=0.95$) and ($C(t)=73.558\times e^{-0.827\times t}$, $R^2=0.95$) for Imidacloprid at single and double dosages, respectively.

Table 2) Residues of imidacloprid on grape following its application at 80 g a.i. ha^{-1}

Days after treatment	Residue level (mg kg^{-1}) Replication	Mean±SD	Dissipation (%)
Before	ND* ND ND	ND	-
0 (1 h after	11.24 10.25 10.26	10.58±2.42	-
1	7.25 8.15 8.85	8.08±1.25	23.65
3	3.1 4.96 4.25	4.10±1.01	61.25
5	3.45 3.14 2.98	3.19±0.82	69.85
7	0.98 1.02 1.58	1.19±0.82	88.75
10	0.68 0.85 0.96	0.83±0.42	92.15
13	0.28 0.45 0.35	0.36±0.15	96.59
16	0.08 0.08 ND	0.08±0.01	99.24
19	ND ND ND	ND	-
21	ND ND ND	ND	-

In keeping with our experimental results, the half-lives ($T_{1/2}$) of Imidacloprid are 2.21 and 2.94 days if applied on grape berries.

The theoretical dissipation models set up through regression between time after spray

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application and the corresponding residues in grape, correlation coefficient, and half-lives are provided in Table 4. The dissipation rate constants of 0.333 and 0.827 day^{-1} corresponded to half-lives of 2.21 and 2.94 days, advised that the dissipation was dependent of the initial dose of imidacloprid and followed primary-order rate kinetics. Those consequences are consistent with the result of (11,20).

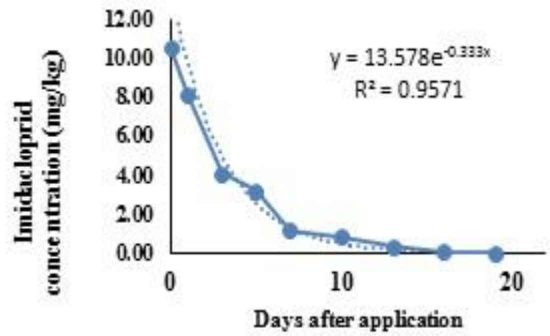

Figure 2) Dissipation of imidacloprid from grape following its application at 80 g a.i. ha^{-1}

Table 3) Residues of imidacloprid on grape following its application at 160 g a.i. ha^{-1}

Days after treatment	Residue level (mg kg^{-1})		Dissipation (%)
	Replication	Mean ± SD	
Before applying	ND*	ND	-
	ND		
	ND		
0 (1 h after spray)	18.01	17.56±2.44	-
	17.95		
	17.00		
1	10.84	10.65±2.01	39.35
	10.54		
	10.56		
3	5.89	6.22±1.54	64.57
	5.94		
	6.84		
5	3.98	3.42±1.87	80.52
	3.28		
	3.00		
7	1.95	2.35±1.08	86.61
	2.62		
	2.48		
10	0.91	0.94±0.04	94.64
	1.01		
	0.89		
13	0.41	0.37±0.06	97.89
	0.38		
	0.31		
16	0.05	0.05 ± 0.01	99.71
	0.05		
	0.05		
19	ND	ND	-
	ND		
	ND		
21	ND	ND	-
	ND		
	ND		

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Table 4) Theoretical dissipation models for Imidacloprid in grape after foliar treatment

Treatment (g ai kg^{-1})	Correlation Coefficient	Dissipation model	Half-life (Days)
80	-0.9571	Y=13.578-0.333X	2.21
160	-0.958	Y=73.558-0.827X	2.94

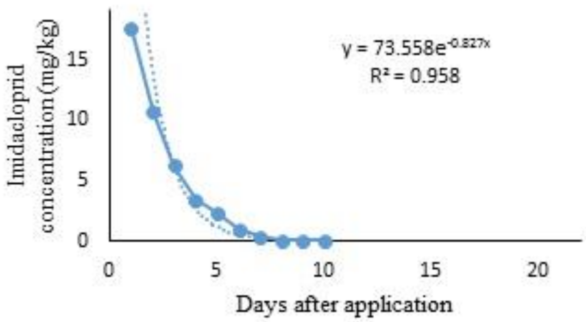

Figure 3) Dissipation of imidacloprid from grape following its application at 160 g a.i. ha^{-1}

Discussion

Residue levels of imidacloprid in samples which were collected after the application of the insecticide in the course of a duration of 21 days showed a gradual and significant ($p<0.05$) decrease in content for this insecticide. These results agree with other research (4,20,21) that simply residues of imidacloprid on grape leaves was rapidly lost in 21 days of its application at the recommended and used dosage. The statistics revealed that there is a speedy lack of this pesticide from the primary few hours/days after application to the end of the periodic interval because the pesticide residues are rapidly lost from plant surfaces by volatilization or a few different manners (9,22). Similar preliminary speedy losses were suggested for systemic pesticides (23,24).

According to our experimental consequences, the half-lives ($t_{1/2}$) of Imidacloprid are 2.21 and 2.94 days if applied on grape berries. Many researchers have calculated the half-lives of imidacloprid in different fruit (12,20,25,26). The study revealed that the dissipation rate was dependent on initial doses and the half-life (t½) values of imidacloprid in grapes.

The PHI is defined as the interval between the last application of a pesticide to a crop and harvesting of that crop. This interval is used to permit for degradation of pesticide residues inside the harvested crop to appropriate levels, which are defined by means of MRLs or criminal limits (12). Maximum residue limit (MRL) of imidacloprid on grapes is fixed at 1.0 mg kg-1 both by Codex Alimentarius Commission (27) and European Union (28). Considering this value the secure pre-harvest interval of imidacloprid on grapes is 7 and 10 days at endorsed and double dosage. But for calculation of pre-harvest interval, the LOQ of 0.05 mg kg^{-1} was taken into consideration; Based on the persistence study and LOQ of 0.05 mg kg^{-1}, the pre-harvest interval was worked out to be 21 days, following utility at the recommended and double the recommended doses. Residues of imidacloprid on grape berries were much less than its MRL value after 7 and 10 days of its application at the recommended and double dosage. Consequently, duration of 10 days is suggested for secure consumption of grapes. The use of pesticides on food crops results in unwanted residues, which may also constitute barriers to exporters and domestic consumptions when they exceed MRLs. The residues of imidacloprid were also estimated in grape berries at harvest. Residues from both the treatments of 80 and 160 g a.i. ha-1 have been below the quantifiable restrict of 0.05 mg kg^{-1} on grape berries at harvest.

Within the grape berries, imidacloprid residues higher than their MRL values had been observed within the preliminary days. When the

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pre-harvest intervals between pesticide applications and harvest are not respected by the farmers, the risk of having higher pesticide levels is not negligible. In this situation, the higher levels of pesticide residues can contain huge consumer health dangers and environmental pollution. It is cautioned that a waiting time of 10 days must be determined before intake of fresh grapes, because it may be secure for the purchaser's health. From the above results, it is clear that the advantages of the application of pesticides in agriculture in generating better crops must be weighed against the possible health threat arising from the poisonous pesticide residues in meals. Insecticides need to be applied effectively, according to correct agricultural exercise, the usage of most effective the specified doses. Arora et al. (2009) studied the persistence of imidacloprid on grape leaves and have evaluated only the harvest time residues on grape berries. They have reported that at harvest, i.e. 25 days after the last spray, residues in grape berries were below determination limit of 0.05 mg kg^{-1} which is in agreement with the results obtained in our study (4). Also, an observed on the dissipation of imidacloprid in Orthodox tea and its transfer from made tea to infusion required a ready duration of 7 days after pesticide utility at a recommended dose for tea (29). In the present study, imidacloprid could not be detected beyond 21 days when applied at the two doses. Grapes grow in bunches of a large number of fruits, and accumulation of pesticides within these bunches could lead to higher residue concentrations as compared with those found in other fruits. Due to the fact grapes have a skinny outer epicuticular layer; pesticide penetration to the pulp may be higher than in fruit with a thicker skin. In an earlier study, when grapes were treated with imidacloprid, residues persisted for 60 days (20). The systemic nature of imidacloprid and the structure form of grapes (smooth-skinned berries developing in clusters) may be the cause for longer persistence on the grapes (25). Also, Degradation of imidacloprid on grapes followed first-order kinetics. Imidacloprid residues degraded with a half-life of about 3 days on grapes after both treatments (60 and 180 g a.i. ha^{-1}). While pesticides remaining on the surface of the plant may degrade rapid, those absorbed into the plant might also degrade slowly, which could cause the long persistence of imidacloprid on grapes.

The application of the imidacloprid (Confidor 200 SL) at 80 and 160 g ha^{-1} 1,000 L^{-1} water leafs residues of imidacloprid in grape berries below its determination limit of 0.05 mg kg^{-1} which is quite low as compared to its MRL of 1.00 mg kg^{-1}. Furthermore, acceptable daily intake of imidacloprid is 0.06 mg kg^{-1} body weight day^{-1} which means that an adult of 60 kg and a child of 10 kg can safely tolerate intake of 3,600 and 600 µg of imidacloprid without any appreciable risk to their life. Assuming a consumption of 200 g grapes contaminated at 0.06 mg kg^{-1} discovered within the present look at application rate, it will lead to intake of 12 µg imidacloprid, which is quite safe for a child as well as an adult and constitutes only 0.02% and 0.003% of ADI value. However, the results from this study make imidacloprid an ideal insecticide that can be safely used on grapevines, which infected with Psalmocharias alhageos.

Conclusion

Grapes are grown in a variety of soil and climatic situations in Malayer and are susceptible to many pests and illnesses. Because grapes grow in bunches and have thin skins, pesticide residues on them are relatively high and residues that accumulate on the surface of the fruit can easily transfer to the pulp. Unlike fruit for which the peel is removed before consumption, the grape is generally consumed together with the peel. Moreover, consumption of insecticides from grapes may be high as compared with fruits with tough

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outer skins. Therefore, pesticides with brief PHIs are required to allow residues to dissipate below the pre-scribed MRLs. Imidacloprid is widely used for the treatment of grapes that infected with a cicada. Consequences obtained from this from this examine confirmed that residues of imidacloprid stay in the grapes, in particular after a few days of spraying of pesticides. The PHIs had been 7 and 10 days for treatment at the recommended (80 g a.i.ha^{-1}) and double (160 g a.i.ha^{-1}) doses, respectively. Therefore, residue-free produce can be obtained at harvest if the combined formulation is used on grapes according to good agricultural practices. According to the results of this study, the usage of imidacloprid on the grape crop (at those recommended and double dosage) appears to be toxicologically perfect.

Footnotes

Funding/Support:

This work was financially supported by the Research Institute for Grapes and Raisin (RIGR) of the Malayer University (84/5-1-295). By this way, thanks to the authors of this article Acknowledgments from the University.

Conflict of Interest:

The authors declared no conflict of interest.

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