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Dissipation and Residues of Penconazole in Grape Fruits

Ehab Hassan[1,*], Nevien Ahmed[1], Mohamed Arief [2]

[1]Central Agricultural Pesticide Laboratory, Pesticide Residues and Environmental Pollution Department, Agriculture Research center, Dokki, Giza, Egypt
[2]Department of Chemistry, Faculty of Sciences, Benha University, Benha, Egypt
*Corresponding author: ehabhassan3006@yahoo.com

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Abstract The main objective of this study was to understand the residue and persistence behavior of Penconazole fungicide in grape fruit samples. The residues were analyzed by GC. The average initial deposit of in grape fruit was observed to be 1.04 mg kg^{-1} at single application rate. The recoveries of Penconazole on grape fruit were observed from 91.04% to 92.13% at fortification levels of 0.1, 0.5 and 1.0 mg kg^{-1}. The reported limit of quantification (LOQ) was found to be 0.02 mg kg^{-1}. The dissipation experiments showed the half-lives ($T_{1/2}$) of Penconazole were around 1.56 days. According to the maximum residue limit (MRL) the pre-harvest interval (PHI) of Penconazole on grape was 14 days after the treatment.

Keywords: *penconazole, residues, dissipation, grape*

1. Introduction

Pesticides will continue to be used in the production of food and fiber especially in the developing countries. Drastic reductions of pesticide usage will increase the production cost and lower the quality of the agriculture productivity. It is well recognized that there are risks attached to the consumption of pesticide-treated crops because of the presence of residues on them [1] and [2]. Therefore, the rational recommendation of a pesticide requires that it must not only provide an effective control of pests but at the same time its residues on the commodity must also be toxicologically acceptable. Penconazole [(RS)-1-[2-(2,4-dichlorophenyl)pentyl]-1H-1,2,4-triazole] is a systemic triazole fungicide used for the preventative and control of powdery mildew [3]. This fungicide is normally sprayed directly onto plants and is rapidly absorbed and distributed to the interior of the leaves [4]. Grape (Vitis vinifera) is one of the most widely-grown fruit crop in the world. Thompson Seedless grape cultivar ranking as the most important table grape variety grown in Egypt. Worldwide, the planted areas of grapes are estimated by 24 million feddan and the total yield exceeds than 60 million ton annually. In Egypt, the grape is planted in different type of soils and represents the second position between fruit crops after citrus. In the earliest writings and archives associated to all sorts of agricultural and religious activities, grapes and its products were given a significant place [5]. The grape crop is frequently infested by a number of diseases at all stages of its development. The crop is often applied with chemical pesticides to offer protection from severe damage. Limited data have been reported concerning the dissipation of Penconazole fungicide in grape fruit and, as a result, no published data are available concerning the fate of Penconazole in grape. Therefore, the aims of the present study were to evaluate the dissipation of Penconazole residues as a function of time and to calculate the PHIs on treated grape.

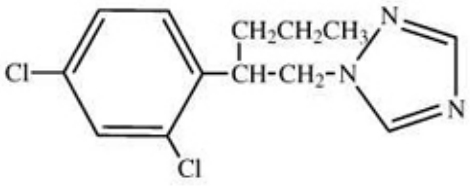

Figure 1. molecular structural of penconazole

Table 1. Recoveries and relative standard deviations for Penconazole in grape at various fortification level

Fortified level (mg kg^{-1}) (n*=5)	Grape Fruits	
	Recovery	RSD
1	91.97	5.2
0.5	92.13	8.1
0.1	91.04	9.3

* Number of replicates

2. Material and Method

Penconazole analytical standard and the formulation (10% EC) were kindly supplied by Syngenta (Cairo, Egypt). All organic solvent were HPLC grade and supplied by Merck Ltd. Deionized water was prepared by a Milli-Q water purification system. Primary secondary amine (PSA, 40 lm Bondesil) was purchased from Supelco (Supelco, Bellefonte, USA). Anhydrous magnesium sulfate was of analytical grade and purchased from Merck Ltd. Sodium chloride was analytical grade and purchased from El Naser pharmaceutical chemical Com. (Egypt). Anhydrous magnesium sulfate and Sodium chloride were activated by heating at 250°C for 4 h in the oven before use and kept in desiccators. A gas liquid chromatography (Hewlett-Packard Model 6890) equipped

with an electron capture detector GC/ECD,GC analysis was conducted on a HP-5(Agilent, Folsom, CA) fused silica capillary column of 30m length, 0.32 mm id., and 0.25mm film thicknesses. The oven temperature was 210°C. injector and detector temperature were maintained at 300 and320°C, respectively. Nitrogen was used as a carrier at flow rate of 3ml/min. Grape trees were cultivated in plots, each plot contained 20 vines. Plots were arranged in complete randomized block design at Quesna district, El-Menofia Governorate, Egypt, on 2 August 2011. Common agricultural and fertilization practices were used. No insecticide sprays were applied to the test plots prior to or during this experiment. Mature plants were sprayed by commercial formulation of penconazole (Topas® 10 % EC) at the recommended dose (25 mL/100 L) using knapsack sprayer motor. The spray solution was prepared in accordance with the manufacture recommendation. The control plots were left unsprayed. There was no rainfall at any time during the experimental period. The average daily temperature during the experiment was from 25 to 39°C. Sampling was performed by randomly collecting from various places of the experimental plots according to the FAO/WHO recommendations [6]. Three replicates were made and fruit samples were taken 0, 1, 2, 3, 7, 10, 14,17 and 21 days after application. Random samples of about 1 kg were collected from each plot and the samples were transferred immediately to the laboratory in an ice box. The samples were comminuted using the laboratory blender and representative homogenized (10 g) of each was then placed into 50 mL polyethylene tube. Samples were extracted and cleaned up immediately after sampling using QuEChERS methodology [7]. 10mL of acetonitrile was added into each tube. The samples were well shaken using a vortex mixer at maximum speed. Afterwards, 4 g of anhydrous magnesium sulfate and 1 g of sodium chloride were added, then extract by shaking vigorously on vortex for 5 min and centrifuged for 10 min at 4,000 rpm. An aliquot of 1 mL was transferred from the supernatant to a new clean 15 mL centrifuge tube containing 25 mg PSA and 150 mg anhydrous magnesium sulfate. The samples were again vortexed for 3 min and then centrifuged for 10 min at 4,000 rpm. An aliquot of 1 mL was concentrated to dryness. The residue was redissolved in 1 mL of ethyl acetate and filtered through a 0.2 lm PTFE filter (Millipore, USA) prior to GC. An aliquot (1 lL) of the final extract was injected into the GC/ECD as described by Gamon et al. [8]. The retention time of penconazole was about 4.41 min. Residues were estimated by comparison of peak area of standards with that of the unknown or spiked samples run under identical conditions. Untreated grape samples were homogenized before begin spiked with penconazole. Recovery assays were performed in the 0.1–1.0 mg kg^{-1} range. The samples were processed according to the above procedure. At each fortification level, five replicates were analyzed. The degradation kinetics of the invistigated pesticide in grape fruits were determined by plotting logarithm residue concentration against time. The rate of degradation (K) and half-life ($t_{1/2}$) values were determined from the following equation of Hoskin(1961) [10].

Rate of degradation (K) = 2.303 X slope

Half-life (t1/2) = 0.693/K

Statistical analyses were done using the Statistical Package for Social Sciences (SPSS 10.0).

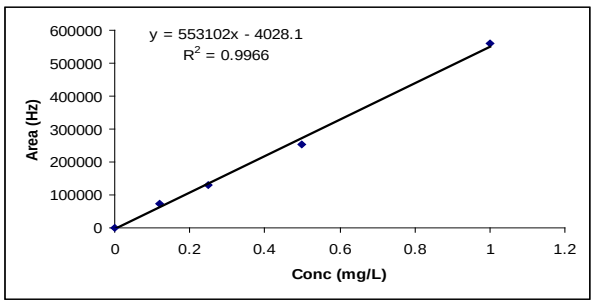

Figure 2. Standard calibration curve of penconazole

3. Result and Discussion

A standard calibration curve of penconazole was constructed by plotting analyte concentration against peak area Figure 2. The detector response was linear in the range of analysed penconazole by the given method with correlation coefficients [0.9966. Blank grape samples were used to establish the detection (LOD) and quantification (LOQ) limits for penconazole by GC. The LOD and LOQ were determined as the sample concentration of penconazole at signal to noise ratio of 3:1 and 10:1, respectively. The LOD and LOQ were estimated to be 0.01 and 0.02 mg kg^{-1}, respectively. Recovery results are shown in Table 1. The recoveries obtained from grape ranged from 91.04% to 92.13%. The relative standard deviation (RSD) was <7.5%. These results demonstrate the good performance of the method.

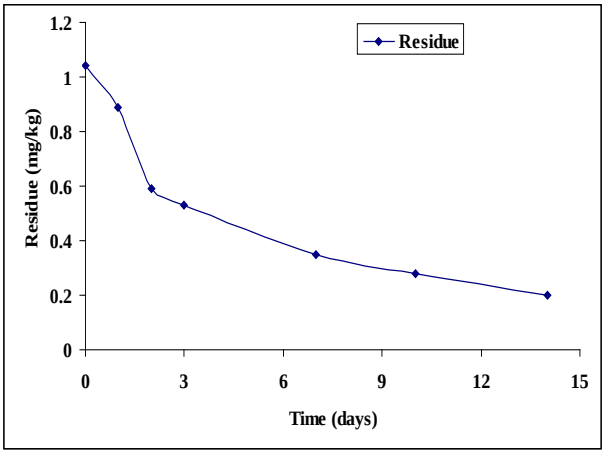

Figure 3. Dissipation of penconazole in grape fruits

The dissipation results of penconazole in grape were shown in Figure 2. The average residue of penconazole recorded was 1.04mg kg^{-1} immediately following application of the recommended dosage. The penconazole residues were decreased with the time. The residues were dissipated to an extent of 14.42 % after 1 day showing residues of 0.89 ± 1.22 mg kg^{-1}. Following that period, the residual amount of penconazole dissipated by 43.27%, 49.04%, 66.35 % and 73.08 % after 2, 3, 7 and 10 days after spraying, with average deposits of 0.59± 0.58, 0.53 ± 0.27, 0.35 ± 0.46 and 0.28 ± 1.17 mg kg^{-1}, respectively. Finally, the residue of penconazole in grape was bellow 0.02 mg kg^{-1} 14 days after the treatment. The dissipation of the pesticide in/on crops depends on the climatic condition, type of application, plant species, dosage, the interval between application, and harvest [9]. Half-life

value (T1/2) for degradation of penconazole on grape fruits was calculated [10] and observed to be 1.56 days, at the recommended dosage. European Union MRL for penconazole in grape was 0.2 mg kg^{-1}. Residues of penconazole on grape fruits were less than its MRL value after 14 days of its application at the recommended dosage.

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