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Food Chemistry 98 (2006) 127–135

Food Chemistry

www.elsevier.com/locate/foodchem

Residue contents of DDVP (Dichlorvos) and diazinon applied on cucumbers grown in greenhouses and their reduction by duration of a pre-harvest interval and post-harvest culinary applications

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Received 14 March 2005; received in revised form 20 May 2005; accepted 20 May 2005

Abstract

Controlled applications of DDVP (Dichlorvos) and diazinon were carried out on cucumbers grown in two different greenhouses at different times. The first group of samples was collected 4 h after the application and the second group was collected 4 days later, which was the mean cropping period applied in this region following maturation of the cucumber plant. Additionally, control samples were collected before application. The effects of washing, peeling and predetermined storage periods, at 4° C for 3 and 6 days, on the reduction of residue levels in the plant tissues were investigated in the two groups. A gas chromatographic method, using acetone, dichloromethane and petroleum ether, as extraction solvents was used to analyse residual DDVP and Diazinon in cucumbers, with obtained recoveries greater than 81%. DDVP and Diazinon were determined by gas chromatography–electron capture detection (GC–ECD) equipped with a 5% phenylmethylpolysiloxane-coated fused-silica capillary column.

Results showed that residue levels in samples, which were collected after 4 days following the pesticide application, were significantly lower than the samples collected after 4 h subsequent to the pesticide application. Culinary applications, such as washing and peeling and refrigeration storage, were also effective in reducing the residue levels. 2005 Elsevier Ltd. All rights reserved.

Keywords: Pesticide residues; Cucumber; Culinary applications; PHI; DDVP; Diazinon

1. Introduction

Pesticides are chemical substances that are widely used against plant pests and diseases. The use of pesticides in commercial agriculture has led to an increase in farm productivity [\(Krol, Arsenault, Pylypiw, & Mattina, 2000\)](#page-8-0). Pesticides are essential in modern agricultural practices but, due to their biocidal activity and potential risk to the consumer, the control of pesticide residues in foods is a growing source of concern for the general population (Torres, Picó, & Mañes, 1996). Governments and international organizations are regulating the use of pesticides and are setting the acceptable MRLs in foods. When these compounds are applied according to good agricultural practices, MRLs are not exceeded, but their incorrect application may leave harmful residues, which involve possible health risk and environmental pollution. Teratogenic, carcinogenic and toxic properties of these compounds have been reported in the literature ([Bernard &](#page-7-0)

Abbreviations: ADI, acceptable daily intake; AOAC, association of official analytical chemists; ECD, electron capture detection; LD_{50} , lethal dose 50; MRL, maximum residue level; PHI, pre-harvest interval.

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^{0308-8146/\$ -} see front matter © 2005 Elsevier Ltd. All rights reserved. doi:10.1016/j.foodchem.2005.05.064

[Gordon, 2000; Jalilian, Sattari, Bineshmarvasti, Shafiee,](#page-7-0) [& Daneshtalab, 2000](#page-7-0)). The presence of their residues in fruits and vegetables can be a significant route to human exposure [\(Council Directive 90/642/EEC, 1990\)](#page-8-0).

Especially in developing countries, residue problems are gaining increasing importance, due to the lack of government inspections and awareness of the producer and consumer about this matter. As a consequence, food consumers are face-to-face with food products which have high residue levels.

Residual pesticides on the foodstuffs, related to the kinds and properties of the pesticides, decrease by various culinary application or with time. There is clear evidence that culinary treatments, including washing, peeling and cooking, can have a huge effect on the removal or degradation of the pesticide ([USEPA, 2000\)](#page-8-0). Several investigators have found that levels of DDVP and diazinon residues were reduced by the pre-harvest intervals and/ or culinary applications, such as washing, peeling and storage [\(Bognar, 1977; Kawar, de Batista, & Gunter,](#page-8-0) [1973; Krol et al., 2000; Love & Ferguson, 1977; Pardue,](#page-8-0) [Hansen, Baron, & Chen, 1970; Schattenberg, Geno, &](#page-8-0) [Hsu, 1996; Sugibayashi et al., 1996; Tsumura-Hasegawa,](#page-8-0) [Tonogai, Nakamura, & Ito, 1992; Webley, 1993; Wen,](#page-8-0) [Shimamoto, Nishihara, & Kondo, 1985\)](#page-8-0).

The first pesticide under investigation in this study, diazinon, has been used in agriculture as a nematicide and insecticide against soil insects and pests of fruits, vegetables, tobacco, forage, field crops, rangelands and pasture. It is also used to keep greenhouses and mushroom houses free of flies. Diazinon is an organophosphorus pesticide, of moderate mammalian toxicity, which is active against a variety of agricultural and public health pests ([WHO/FAO](#page-8-0)). It is readily absorbed by the gastrointestinal tract, through the intact skin and by inhalation. It is converted in vivo to the oxygen analogue diazixon, which then inhibits cholinesterase [\(WHO,](#page-8-0) [1998\)](#page-8-0). The LD_{50} is 300 to 400 mg/kg for technical grade diazinon in rats ([Gallo & Lawryk, 1991; Kidd & James,](#page-8-0) [1991\)](#page-8-0). ADI is 0.002 mg/kg/day [\(Lu, 1995\)](#page-8-0). The structure and chemical names of diazinon is shown in Fig. 1.

The other pesticide, DDVP, is an organophosphate compound used to control household, public health,

O,O-diethyl O-2-isopropyl-6-methyl-pyrimidin-4-yl phosphorothioate (IUPAC) O,O-diethyl O-[6-methyl-2-(1-methylethyl)-4-pirimidinyl]phosphorothioate (CA)

Fig. 2. Structure and chemical names of DDVP.

and stored product insects. It is effective against mushroom flies, aphids, spider mites, caterpillars, thrips and white flies in greenhouses, outdoor fruit, and vegetable crops [\(Anonymous, 1996\)](#page-7-0). The oral LD_{50} for DDVP is 61–175 mg/kg in mice, 100–1090 mg/kg in dogs, 15 mg/ kg in chickens, 25–80 mg/kg in rats, 157 mg/kg in pigs, and 11–12.5 mg/kg in rabbits [\(Anonymous, 1995; Gallo](#page-7-0) [& Lawryk, 1991; Kidd & James, 1991\)](#page-7-0). ADI is 0.004 mg/ kg/day ([Lu, 1995](#page-8-0)). DDVP primarily affects the nervous system through cholinesterase inhibition, i.e., the blockage of an enzyme required for proper nerve functioning. In addition, the EPA has classified it as toxicity class I – highly toxic, because it may cause cancer and there is only a small margin of safety for other effects [\(Anony](#page-7-0)[mous, 1996](#page-7-0)). DDVP has been classified as a possible human carcinogen because it caused tumors in rats and mice in some studies [\(EPA, 1988, 1991\)](#page-8-0). The structure and chemical names of diazinon is shown in Fig. 2.

Effects of washing, peeling and storage, for different periods applied with the aim of reduction of DDVP and diazinon (widely used in greenhouses in Antalya, Turkey) residues in cucumber samples, are presented in this study. These effects were evaluated in cucumbers collected after two pre-harvest time intervals following the pesticide application. The first group of cucumbers was collected 4 h after the pesticide application and the second group was collected after 4 days. The recommended pre-harvest intervals were 5 days for DDVP and 21 days for diazinon, as suggested in their respective prospectuses. However, cucumber samples in the second group were collected 4 days after the pesticide application because, in this region, cucumber fruits were harvested in 4 day periods following maturation of the cucumber plant.

2. Materials and methods

2.1. Chemicals

The dichloromethane, acetone and petroleum ether (for analysis of pesticide residues) used in the study were purchased from Merck (Darmstadt, Germany). Pesticide analytical standards were purchased with the purity certified from Dr. Ehrenstorfer (Augsburg, Germany). Concentration of these standards was $10 \text{ ng}/\mu l$ in cyclohexane. More dilute solutions were prepared just Fig. 1. Structure and chemical names of diazinon. before use. Anhydrous sodium sulphate and sodium chloride for residue analysis was obtained from Merck (Darmstadt, Germany). Commercial DDVP (Didifos, %50 EC) that was used on cucumber plants was received from Hektas- (Kocaeli, Turkey) and commercial diazinon (Bazinon %20 EM) was received from Koruma Tarım A.Ş. (İzmit, Turkey).

2.2. Apparatus

Experiments were carried out by using an HP 5890 Series 2 Plus GC System (Hewlett Packard, USA) equipped with a 63 Ni ECD system. Chromatographic separation was achieved by using a DB-5 30 m \times 0.25 mm ID, $0.25 \mu m$ film thickness analytical column from J&W Scientific (Folsom, CA, USA). Nitrogen (purity 99.999%) was used as carrier gas. All data were collected on HP Chemstation software.

2.3. Instrumental conditions

The injector and detector temperatures were kept at 250 and 300 \textdegree C, respectively, throughout the analysis. The column temperature was raised from 70° C (hold 2 min) to 150 °C at 25 °C/min, then to 200 °C at 3 °C/ min, and finally to 280 °C (hold 10 min) at 8 °C/min. Total time for the GC analysis was 43.95 min. A split/ splitless injector operated in the splitless mode was used. The carrier was nitrogen at 14.1 psi column head pressure. The flow of carrier gas was applied as 30 ml/min. The injection volume was 1μ l. GC analysis conditions are presented in Table 1.

2.4. Preparation of commercial pesticides and application in greenhouse

Commercial DDVP and diazinon were diluted in water and mixed. In this way, a sufficient quantity of suspension was obtained for application on the field area. The concentration of each pesticide was 200 ml/

*Analysis (Notice that treated cucumber samples after mentioned processes were analyzed as separate each other)

Fig. 3. Trial schedule.

100 l in water. The prepared emulsion was applied to the cucumber plant using a sprayer.

2.5. Sample collection and storage

Cucumber samples, used in analysis, were grown in two different commercial greenhouses in Antalya. The absence of residual pesticides on samples was confirmed by residue analysis prior to the application of commercial pesticides.

Mature cucumber samples were collected after application of commercial pesticide emulsions according to Section 2.4, and operated as in the procedure shown in Fig. 3.

The collected samples were transferred to the laboratory and analyzed immediately. Samples which required a washing procedure were washed for 15 s by rubbing under running tap water. Samples that required a peeling procedure were peeled with a knife which was previously submerged in acetone for a short time. Samples which were subjected to a storing procedure were kept at $+4$ °C in the refrigerator in polyethylene bags.

2.6. Analytical procedure

2.6.1. Preparation for analysis

All glassware, filter papers and auxiliary equipment (such as knife) were cleaned and rinsed with extra-pure acetone prior to the residue analyses and recovery studies. In this way, interference caused by materials which contaminate the analyte from this apparatus was avoided.

Fig. 4. Gas chromatogram of DDVP standard (min) (concentration: 1 μ g/ml) and calibration curve (0.05–2 μ g/ml).

Working solutions were obtained by appropriate dilutions with acetone and stored in a refrigerator $(4 °C)$ (2 months of maximum storage time). No degradation was observed for the compounds in the mentioned storage times. Various standards of pesticides $(0.05-2 \text{ µg/ml})$ were prepared and injected into the GC system under the conditions stated in [Fig. 3](#page-2-0) and the retention times and areas were recorded. Calibration curves were prepared for these concentrations (Figs. 4 and 5). In this method, detection limits of 0.01 ng/ml for both DDVP and diazinon were determined.

2.6.2. Recovery studies

The method was optimized by recovery studies before the determination of kinds and quantities of pesticides on collected samples. Recovery studies were carried out by spiking fresh samples, which did not contain any pesticides, with known volumes of the appropriate working mixtures of pesticides. The same extraction procedures and GC conditions as applied for sample analyses were used for recovery studies. In this way, recoveries obtained were 94.4% for DDVP, 81.4% for Diazinon.

Fig. 5. Gas chromatogram of diazinon standard (min) (concentration: 1 μ g/ml) and calibration curve (0.05–2 μ g/ml).

2.6.3. Extraction procedures

An AOAC (1985) method was used in this study. According to this method, a non-fatty test portion is blended with acetone and filtered; pesticides are transferred from aqueous filtrate to organic phase by shaking with petroleum ether and CH_2Cl_2 ; after drying, organic phase is concentrated in the presence of petroleum ether and then acetone to remove CH_2Cl_2 ; an aliquot of concentrated organic phase is injected into various GC systems for determination of a wide variety of pesticide residues.

About 2 kg of vegetable samples were chopped, mixed and homogenized. A test portion of 100 g was weighed and transferred into a high-speed blender jar with 200 ml of acetone, and blended for 2 min at high speed. The homogenate was filtered through a 12 cm Buchner funnel fitted with filter paper using a vacuum system. Extracts were collected in 250 ml volumetric flasks.

The amount of extract was recorded and 80 ml of extract was transferred into a 11 separatory funnel. To form a secondary phase, 100 ml of petroleum ether and 100 ml dichloromethane were added. The separatory funnel was shaken vigorously for 1 min and separation of the two phases was observed. The lower aqueous layer was transferred to a second 11 separatory funnel. The upper organic layer in the first separatory funnel passed through anhydrous sodium sulphate placed over a filter paper fitted funnel into a 500 ml rotary evaporator flask. Into the second 1 l separatory funnel, 7 g NaCl were added and the funnel was shaken vigorously for 30 s until most of the NaCl was dissolved. Hundred millilitre of dichloromethane were added, shaken 1 min and the separation of two phases was observed. The lower organic phase was dried by passing through the same sodium sulphate. Hundred millilitre of dichloromethane were added to the extracted aqueous phase and dried as above. Sodium sulphate was rinsed with ca. 50 ml dichloromethane. All extracts were collected in a rotary evaporator flask.

The extract was concentrated using a rotary evaporator (45 °C). When the liquid level in the rotary

evaporator flask was ca. 2 ml, 100 ml petroleum ether were added and the mixture reconcentrated to ca. 2 ml. The concentration step was repeated with further addition of 50 ml petroleum ether. After the addition of 20 ml of acetone, the mixture was reconcentrated to ca. 2 ml. Care was taken to avoid absolute dryness during the concentration steps. The contents of the flask were then completed to a volume of 7 ml with acetone.

2.6.4. Calculation of equivalent test portion weight

Calculated equivalent test portion weights in the final solution are found as follows:

mg test portion equivalent

$$
\mu l \text{ final extract}
$$

$$
= \left(\frac{80}{200 + W - 10}\right) \left(\frac{1}{\text{ml final volume}}\right) 100,
$$

where $200 = \text{ml}$ acetone blended with 100 g test portion; $W =$ amount (ml) H_2O present in test portion; $10 =$ adjustment for water–acetone volume contraction.

2.7. Statistical evaluation

All analyses were performed on duplicate samples and the results were statistically analyzed by ANOVA $(P < 0.01)$. Significant means were subjected to analysis by Duncan's multiple range test ($P \le 0.05$). All statistical analyses were performed using the Statistical Analysis System [\(SAS Institute, 1998](#page-8-0)).

3. Result and discussion

3.1. Results of the DDVP application

The results of DDVP residue analyses are presented in Table 2. The variance analysis was applied to these results. Duncan multiple range test of significantly different means are presented in [Table 3.](#page-5-0)

According to results of variance analysis, significant reductions in residue levels for DDVP were obtained through both the pre-harvest time and processes which

Values are given as means \pm standard error.

^a Mean residue contents in samples which were collected 4 h after the pesticide application.

^b Mean residue contents in samples which were collected 4 days after the pesticide application.

Table 3

^A Values in a column followed by different letters are significantly ($P < 0.05$) different (Duncan's multiple range test). Values are means \pm standard error.

were aimed at decreasing pesticide residues ($P \le 0.01$). Significant interactions of these two parameters were observed on the reduction of residues ($P \le 0.01$).

According to the results of Duncan's multiple range test shown in Table 3, when compared to the samples which were collected 4 h after the pesticide application, DDVP residues were significantly ($P \le 0.05$) reduced in samples which were collected 4 days following the pesticide application. But no significant effects were observed on the residue level by the washing process ($P \le 0.05$). These results agree with [Sugibayashi et al. \(1996\)](#page-8-0) who reported that DDVP residues were reduced by the washing procedure. No statistical differences were observed between effects of peeling and storing (3 days at 4° C) on the reduction of residue levels ($P \le 0.05$). Thus, the effects of these processes were similar. In addition, the results of Duncan multiple range test shows that the most effective process for reduction of residues of DDVP was storing for 6 days at 4° C.

Fig. 6 shows percentages of detected average residues after different processing applications, on the cucumber samples. Percentage average residues determined after collection of cucumber fruit after the pesticide application and following different pre-harvest intervals are shown in Fig. 7. Also, process and time effects are presented together in Fig. 8.

The initial DDVP residue level was decreased 22.4% by the washing procedure, 57.2% by the peeling procedure, 48.1% by the storage procedure at $+4$ °C for 3

Fig. 6. Percentages of detected average residues after different processes in cucumber samples to which commercial DDVP was applied.

Fig. 7. Percentages of detected average DDVP residues after the pesticide application on samples collected following different preharvest intervals (PHI).

Fig. 8. Variation of detected average DDVP residues after different processes in cucumber samples which were collected 4 h and 4 days after pesticide application.

days and 70.8% by the storage procedure at $+4$ °C for 6 days. On the other hand, a 4 day pre-harvest interval resulted in a 93.2% reduction of DDVP residue levels in cucumber samples compared to cucumber in which no process was applied (Figs. 6 and 7).

3.2. Results of the diazinon application

The results of the diazinon residue analyses are presented in [Table 4](#page-6-0). The variance analysis was applied to these results. The Duncan multiple range test of significantly different means is presented in [Table 5](#page-6-0).

Values are given as means \pm standard error.

Mean residue contents in samples which were collected 4 h after the pesticide application.

^b Mean residue contents in samples which were collected 4 days after the pesticide application.

Table 5

Results of Duncan's multiple range test for means of diazinon residues in cucumber samples

^A Values in a column followed by different letters are significantly ($P < 0.05$) different (Duncan's multiple range test). Values are means \pm standard error.

According to results of variance analysis, significant reductions in residue levels of diazinon were obtained through both the pre-harvest time and processes which aimed at decreasing pesticide residues ($P \le 0.01$). Significant interactions of these two parameters were observed on the reduction of residues ($P \le 0.01$). The results also agree with [Pardue et al. \(1970\)](#page-8-0) who reported that diazinon residues were reduced by the pre-harvest interval.

According to the results of Duncan's multiple range test, diazinon residues were significantly reduced in samples which were collected 4 days following the pesticide application ($P \le 0.05$). Cucumber samples which were not subjected to any process had the highest level of residues. But no significant effects were observed on residue level by the washing process ($P \le 0.05$). These results agree with [Wen et al. \(1985\)](#page-8-0) who reported that diazinon residues were reduced by the washing procedure. Although no statistical differences were observed between the effects of peeling and storage for 6 days at 4 °C processes on the reduction of Diazinon residue levels ($P < 0.05$), these parameters were both effective in reducing diazinon residues.

Fig. 9 shows percentages of detected average residues after different processing applications on the cucumber samples. Percentage average residues, determined after collection of cucumber fruit, after the pesticide application and following different pre-harvest intervals, are shown in Fig. 10. Also, the process and time effects are presented together in [Fig. 11](#page-7-0).

The initial Diazinon residue level was decreased 22.3% by the washing procedure, 67.3% by the peeling procedure, 35.8% by the storage procedure for 3 days

Fig. 9. Percentages of detected average residues after different processes in cucumber samples which commercial diazinon was applied.

Fig. 10. Percentages of detected average diazinon residues after the pesticide application on samples collected following different preharvest intervals (PHI).

at $+4$ °C and 64.8% by the storage procedure for 6 days at $+4$ °C. On the other hand, a 4 day pre-harvest interval resulted in a 69.3% reduction of diazinon residue

Fig. 11. Variation of detected average diazinon residues after different processes in cucumber samples which were collected 4 h and 4 days after pesticide application.

levels in cucumber samples to which no process was applied [\(Figs. 9 and 10\)](#page-6-0).

It can be seen from [Fig. 10](#page-6-0) that the pre-harvest interval (4 days), following application of pesticides, has an obviously decreasing effect on diazinon residues in cucumbers.

4. Conclusions

DDVP is a contact and stomach insecticide with fumigant and penetrating action [\(WHO, 1989](#page-8-0)). In this study, DDVP residues were obviously reduced in samples which were collected 4 days after the pesticide application when compared to the samples which were collected 4 h after the pesticide application. DDVP is rapidly lost from plant surfaces by volatilization ([FAO/WHO, 1968, 1971\)](#page-8-0). So, the recommended pre-harvest intervals were only 5 days for DDVP for cucumbers, as suggested in the related prospectus. It is known that DDVP concentrations are more slowly reduced in greenhouse conditions than outdoors ([WHO/FAO, 1970\)](#page-8-0). The samples subjected to refrigerated storage at $+4$ °C for 3 and 6 days had lower DDVP residue contents than the samples which had a shorter PHI (4 days). This can be attributed to lower rates of physiological elimination reactions under refrigerated storage for samples with a shorter PHI (4 days). No significant reduction was observed in cucumber samples which were subjected to a washing process. This may be attributed to the partial apolar characteristic of DDVP ([Chen, Su, &](#page-8-0) [Jen, 2002\)](#page-8-0). Polar, water soluble pesticides are more readily removed than low polarity materials [\(Elkins, 1989\)](#page-8-0). This probably reflects, not only their higher solubility in the wash, but also their reduced propensity to move into waxy layers. Removal of the skin, by peeling, leaves cucumber tissue below the waxy layer, therefore resulting in reduction of DDVP residues by more than 55%.

Based on the obtained data, refrigerated storage for 6 days at $+4$ °C was the most effective way to reduce the

DDVP residues of the cucumber samples. Peeling and refrigerated storage at $+4$ °C for 3 days also decreased DDVP residues. No statistical differences were found between the effects of these processes.

When comparing diazinon with DDVP, it is clear that pre harvest intervals were less effective in reducing the diazinon residue contents than they were for DDVP. A pre-harvest interval (of 4 days) reduced DDVP in cucumbers by 93.2%, whereas the residue of diazinon was only reduced by 68.3%. This can be interpreted as being due to the higher stability and lower fumigant character of diazinon compared to DDVP. This is in accordance with the recommended pre-harvest interval for diazinon being 21 days for cucumbers. The washing process was not sufficiently effective in reducing diazinon residues. It has been indicated that diazinon is slowly hydrolysed by water ([WHO/FAO\)](#page-8-0). In this study, peeling and storage for 6 days at $+4$ °C in a refrigerator were the most effective processes for the reduction of residues of diazinon applied on cucumber plants. Samples kept under refrigerated storage at $+4$ °C for 3 and 6 days had lower diazinon residue contents than the samples which had a pre-harvest interval (4 days). This can be attributed to lower rates of physiological elimination reactions under refrigerated storage for samples with a PHI. This result was a similar to that with DDVP residue determinations.

From the above results, it is clear that the advantages of the application of pesticides in agriculture in producing better crops must be weighed against the possible health hazard arising from the toxic pesticide residues in food. Pesticides should be applied correctly, according to good agricultural practice, using only the required amounts. Culinary applications are necessary to decrease the intake of pesticide residues. It can be concluded that processes, such as controlled dose setting for the use of these pesticides, controlled greenhouse treatments, harvest and storage processes, and culinary applications before consumption, have a crucial role in the reduction of residual pesticides which pose a serious threat to human health and the environment.

Acknowledgements

The authors thank the Research Found of Akdeniz University for a Grant (Project 2003.02.0121.19).

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