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Fate of Preharvest-Sprayed Dicofol in Date Fruits: Residue Analysis by HPLC-UV

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مصير مبيد الدايكوفول على ثمار نخيل البلح: تحليل المتبقيات باستخدام جهاز التحليل الكروماتوجرافي عالي الأداء في نطاق. الأشعة فوق البنفسجية

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الخلاصة: تم تقدير مصير مبيد الدايكوفول على ثمار نخيل البلح من الصنف السكري و ذلك بعد رش الأشجار بالمبيد بمعدل 200 ملليلتر/100لتر. وقد تم تقدير المتبقيات في ثمار النخيل بعد فترات مختلفة باستخدام جهاز التحليل الكروماتوجرافي عالي في نطاق الأشعة فوق البنفسجية وعلي طول موجة 220 نانوميتر. وتعتمد طريقة التحليل المستخدمة على HPLC))الأداء استخلاص المبيد بواسطة خلات الإيثايل. أوضحت النتائج أن استجابة جهاز HPLC كانت خطية (قيمة معامل الارتباط أكبر من 0.98) في مدى من التركيزات قدرها 0.0-2.0 ميكروجرام من الدايكوفول و أن قيم حدود التعرف وصلاحية الطريقة المستخدمة تساوي 0.24 و 0.80 جزء في المليون علي التوالي. وقد تم حساب نسبة استرجاع المبيد بعد عمل تقوية لعينات ثمار النخيل غير المعامل بمستويات قدرها 0.25 و 0.5 و1 جزء في المليون وكانت نسبة الاسترجاع المتحصل عليها تتراوح بين 77.2-103.6% وانحراف معياري نسبي يتراوح بين 6.33-11.84%. هذا وقد أوضحت النتائج أن اختفاء مبيد الدايكوفول من ثمار التمر يتبع النموذج ثنائي الوجه وأن تركيز مبيد الدايكوفول عند زمن صفر والذي يساوي 11.22 جزء في المليون قد تناقص ليصبح 1.90جزء في المليون بعد 7 أيام والتي تعتبر أقل من القيمة المسموح بتواجدها على الفواكه والخضروات (2 جزء في المليون). أيضاً وجد أن معدل اختفاء الدايكوفول كان سريعاً في الطور الأول مقارنة بالطور الثاني حيث أن فترات نصف عمر المبيد ($t_{1/2}$) على أشجار النخيل تساوي 1.35 و 38.52 يوم على التوالي. وتمتاز الطريقة المستخدمة في هذه الدراسة بسرعة الأداء والحساسية العالية وارتفاع نسبة الاسترجاع مع إمكانية تكرار النتائج.

ABSTRACT: The fate of pre-harvest-sprayed dicofol (DCF) on date fruits has been investigated. Date palm trees, variety Sukkari, were sprayed with DCF (18.5%, EC) at the rate of 200 ml/100 L. DCF residues in date fruits were determined at different time intervals using high performance liquid chromatography (HPLC) with UV detection at 220 nm. The method was based on extraction with ethyl acetate. The results showed that the HPLC response was linear ($r^2 > 0.98$) for DCF in the range of 0.0 to 2 mμg. The limits of detection and quantification were 0.24 and 0.80 ppm, respectively. The method was developed by using spiked date fruits at levels of 0.25, 0.50 and 1.0 ppm. Recovery percentage was satisfactory with a range of 77.2 to 103.6% and an RSD ranging from 6.33 to 11.84%. A biphasic model was assumed in order to carry out the statistical study of the loss of DCF from date fruits. The results showed that the initial deposit of DCF on date fruits was 11.22 ppm, while the residue value decreased to 1.90 ppm seven days after spraying, which is below the tolerance limit for fruits and vegetables (2 ppm). Also, the data indicate that there was a faster rate of DCF loss in the first phase than the second one. This is clearly reflected in the $t_{1/2}$ values, where the half-lives of DCF were 1.35 and 38.52 days, for the first and second phase model, respectively. The described method is rapid and sensitive, with satisfactory recoveries and reproducibility.

Keywords: Date fruits, fate, dicofol, residue, limit of detection, half-life

Introduction

Pesticides are widely used in agriculture to control a variety of pernicious organisms that spoil crops. More than 600 kinds of agrochemicals are used around the world (Miyake *et al.*, 1999). They provide unquestionable benefit for agricultural production, even though, as a consequence, low amounts of some residues may persist in the food supply and could constitute a significant exposure pathway for humans. Exposure to food residues has created uncertainty for potential chronic toxicity and, in some cases, acute toxicity (Saunders and Harper, 1994; Ekström *et al.*, 1996; Osman and Al-Rehiayani, 2003).

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The use of chlorinated hydrocarbon insecticides has been sharply curtailed or banned, but they are still the active ingredients of some pest control products (Moore, 1986). For example, dicofol (DCF) is used world-wide as a pre-harvest miticide on cotton, citrus, vegetable, nuts and other crops (Rohm and Hass, 1984; Mourer *et al.*, 1990; Fernǎndez *et al.*, 2001; Kitajama *et al.*, 2003). DCF is structurally similar to DDT, which is used as the starting material for synthesis for DCF (Wiemeyer *et al.*, 2001). The US-EPA became concerned about the continued use products containing DCF because they also contained DDT and related compounds (Moore, 1986). DCF products marketed after May 1986 could contain no more than 2.5% DDT and related compounds and those marketed after 1988 could have no more than 0.1% DDT and related compounds. Following reassessment of dietary risk, the EPA has updated U.S. tolerances for DCF residues on a variety of crop commodities and described harmonization with Codex MRLs (U.S.-EPA, 1998).

Al-Qassem region is one of the largest agricultural areas in the Kingdom of Saudi Arabia and much of its arable and fertile land is under date palms (*Phoenix dactylifera* L.). Al-Qassem community produces the majority of date fruits consumed locally and much of those exported outside the country. During the 2002 season, date fruit production was greater than 130,000 tons, of which approximately 1,092 tons were destined for export (Saudi Ministry of Agriculture and Water, 2003). Expansion in date fruit production has brought about increasing pest problems. Pest control has been achieved by heavy reliance on injudicious and indiscriminate use of broad-spectrum pesticides. Consequently, unacceptable concentrations of pesticide residues have been found in date fruits (Al-Rehiayani and Osman, 2003).

DCF is often applied to date palms in Saudi Arabia at a rate of 200 ml/100L to control mites. Unfortunately, no data are available on DCF residues and its loss on date fruits, although it has been reported in cucumber (Nazer and Masoud, 1986), fish (Barbera, *et al*., 1986), meat (Yossef *et al.*, 2003), avian eggs (Krynitsky *et al.,* 1988) and citrus (Fernǎndez *et al*., 2001; Torres *et al*., 1996; Saitta *et al*., 2000). Because fruits of the date palm are vital components of the diet in Saudi Arabia, consumers are increasingly concerned about DCF residues in date fruits and their carry-over to processed products.

Quantitative methods for DCF determination in agricultural products have been reported using colorimetry (Rosenthal *et al.*, 1957), ultra-violet spectrophotometry (Gunther and Blinn, 1957), gas liquid chromatography using an electron capture detector, (Morgan, 1967; Ribeiro *et al.*, 2000) or mass selective detection (Soleas *et al.*, 2000) and high performance liquid chromatography (Fernǎndez *et al.*, 2001) The difficult and crucial step in pesticide residue analysis of complex media, such as foods, is the clean-up or separation of chemicals of interest. In this study, clean-up, separation and determination of the optimum conditions for p,p'-DCF analysis in date fruits were developed. Also, the present study was carried out to determine the residue levels of DCF at different time intervals following field application, as well as the half-life ($t_{1/2}$) on date fruits.

Materials and Methods

Dicofol (DCF), [2,2,2-trichloro-1,1-bis-(4-chloro-phenyl) ethanol], (18.5% EC) was purchased from Hockly International Company, UK, while technical DCF was provided by Environmental Protection Agency (EPA, USA) with a purity of 99%. Certified HPLC-grade ethyl acetate, methanol, acetonitrile (ACN) and granular AR anhydrous sodium sulfate were purchased from BDH Company, while Florisil (60-100 mesh) was purchased from Riedel-de Haën (Germany). Deionized water of 15 MWΩ.cm resistivity was obtained from a water purification system (PURELAB Option-R, ELGA, UK). All solvents and solutions were passed, before used, through a 0.45 mμm sterile disposable nylon filter (AcroCp, Gelman Sciences).

Sukkari, the most widely grown date variety in Al-Qassem region, was selected for this study. Date palm trees cultivated in El-Soltan Farm, north of El-Meledia District, Al-Qassem area, Saudi Arabia, were arranged in a randomized design and sprayed at the end of May 2002 with DCF (18.5% EC) at the rate of 200 ml/100 L (37 g active ingredient, a.i./100 L) using a motorized sprayer that was calibrated to deliver 10 L/tree. Treatments were done in triplicate with eight trees assigned to each replicate. Untreated trees were sprayed only with water and used as analytical controls for background residues and for fortification recovery studies. Date fruits were collected 0, 2 and 4 hrs and 1, 2, 4, 7, 14, 21, 28, 42 and 56 days after DCF application to determine residue concentration. Samples were immediately transported to the laboratory and kept at -15ºC until residue analysis.

Three 50-g aliquots of date fruits from each time interval were chopped and placed in a 250-ml conical flask and blended with 100 ml ethyl acetate for 2 min. The homogenate was then filtered under vacuum through Whatman No. 2 filter paper. The solids were re-extracted with 100 ml ethyl acetate for another 2 min. The combined, filtered extract was passed through a funnel containing anhydrous sodium sulfate, evaporated using a rotary evaporator (38°C) to ca. 2 ml and then made up to 5 ml with ethyl acetate. The concentrated extract was passed through a glass chromatographic column (20 cm x 10 mm i.d.) packed in sequence from bottom to top, with glass wool, 10 g of activated Florisil and topped with 2 g anhydrous sodium sulfate. The column was pre-washed with ethyl acetate. The extract was added to the column and eluted with 50 ml of ethyl acetate. The eluent was evaporated to dryness using a gentle stream of nitrogen and redissolved in 10 ml methanol. The final solution was filtered through a 0.45 mµm sterile disposable nylon filter prior to HPLC analysi***s.***

The HPLC system was standardized on the same day as the samples were analyzed by injecting 20 µl of eight solutions of freshly prepared DCF in methanol with concentrations ranging from 0.0 to 10 ppm from a stock solution of 1000 ppm. Areas under the peak (uV.sec) versus concentrations (µg) were plotted and simple linear regression used to obtain an equation for the standard curve. The amount of DCF in each sample was thus calculated based on the slope of the standard curve.

Three 50-g aliquots of the untreated control were treated as individual replicates for a DCF fortification recovery study. Spiking was done at the levels of 0.25, 0.50 and 1.0 µg/g (ppm). In addition to the spiked samples, one date fruit blank and one solvent blank were run concurrently with the extraction set. Recoveries for DCF and relative standard deviation (RSD) were calculated after HPLC analysis.

Extracts were chromatographed on a Perkin Elmer HPLC system model 200 equipped with a degasser, quaternary LC pump model 2000Q/410, 20 µl loop, Spheri-5 RP-18 column (25 cm x 4.6 mm i.d., 5 µm, Perkin Elmer), oven column, a LC200 UV detector. The Turbochrom Workstation Software package was used for instrument control, data acquisition, and data analysis. The column temperature was kept at 25 °C. DCF was eluted using an isocratic gradient of 78:22 v/v ACN:H2O. DCF was detected by scanning the eluent at wavelengths (lλ) from 200 to 260 nm at a flow rate of 1.1 ml/min. The retention time for DCF was 10.4 min.

The detection (LOD) and quantification (LOQ) limits with this procedure were defined as the concentration of the DCF in date fruits (expressed as ppm) that gave signals of 3 and 10 times the noise, respectively, within its retention time (tR) window (Falqui-Cao e*t al*., 2001). Corresponding results were compared with the maximum residue limit.

The data were calculated as mean ±± S.D and analyzed using analysis of variance technique (ANOVA). Probability of 0.05 or less was considered significant. All statistical analysis was done with Costat Program (Version 2, CoHort Software, 1986).

Results and Discussion

The HPLC response was linear ($r^2 > 0.98$) for DCF in the range of 0.0 to 2 microgram (µg). The LOD of DCF in date fruits was 0.24 ppm or about 8.3 times lower than the maximum residue limit (MRL) of 2 ppm. In a study of DCF residues on oranges, the LOD by GC-ECD analysis was also reported to be 0.24 ppm (Fernăndez *et al.*, 2001). In a multi-residue study of matrix solid phase dispersion extraction of oranges followed by detection with GC-ECD, the LOD for DCF was 0.010 ppm (Torres *et al.,* 1996). The LOQ determined from the method validation was 0.80 ppm. Interferences from co-extractives raise the LOQ of a method by masking the detector response to the residue or by preventing injection of the specified sample matrix without undesirable damage to the system (Falqui-Cao *et al.*, 2001). Additional procedures to clean the sample extract prior to determination may improve the LOQ by removing these interferences. Column cleanup in this study was needed to remove coextractives from date extracts prior to HPLC. The Florisil elution system used in the present study efficiently isolated DCF from the coextractives, where a few unknown peaks appeared in HPLC chromatograms due to coextractives, but did not interfere with DCF peak.

Results of the recoveries of spiked samples are summarized in Table 1. Recovery percentage was satisfactory with a range of 77.2 to 103.6% and an

Table 1. Mean recoveries of DCF from spiked date fruits.

Amount Spiked (ppm)	No. of Determi-nations	Recovery (%)	RSD (%)
0.25	3	103.6	6.33
0.50	5	77.2	11.48
1.00	3	89.6	8.98

RSD ranging from 6.33 to 11.84%. The results are in agreement with the extraction efficiencies reported by other authors who found that the mean recoveries for DCF ranged from 77.2 to 93.8% in egg yolk (Mourer *et al.*, 1990), 78 to 104% in tea (Zhu and Wang, 2000) and 87 to 95% in oranges (Ribeiro *et al.*, 2000). The percentage recoveries for the present study can be considered satisfactory according to guidelines which state that any method with a proven recovery of 70% or more could be eligible for future official adoption (Schuller *et al.*, 1976). Also, the low RSDs indicated a high level of repeatability for the tested method.

Data in Table 2 illustrate the levels of DCF in date fruits over a period of 56 days following one application of DCF at rate of 37 g a.i./100 L. Initial DCF residues were 11.22 ppm but decreased by 78, 66, 60, 46 and 18% of the initial deposit after 2, 4, 24, 48 and 96 hrs, respectively, following application. From day 4 on, the decrease was slow with non significant differences between the residue levels of DCF.

Although MRLs have been established for registered pesticides on agricultural commodities to guarantee their proper use and adequate control (Council Directive, 1990), no maximal limit of DCF residues has been established on date fruits. The residue level 7 days after DCF spraying was 1.90 ppm which is less than the maximum residue limits for fruits and vegetables of 2 ppm (Fernăndez *et al.*, 2001). Also, data in Table 2 illustrate that less than 9% of the initial deposit was detected during the period of 4-8 weeks after DCF application. The present results are consistent with another investigation, where DCF residues on cucumber fruits were below the tolerance level of 2 ppm and no residue could be detected 8 days after DCF application under either plastic house or plastic tunnels (Nazer and Masoud, 1986).

The rate of loss of DCF residue from date fruits is presented in Figure 1. A biphasic model was assumed in order to carry out the statistical study of the loss of DCF according to equation (1).

Table 2. DCF residues in pre-harvest-sprayed date fruits after different time intervals.

Time Interval (days)	DCF Level (ppm) Mean ±S.D*	Initial Deposit (%)	Relative Standard Deviation (RSD) (%)
0	11.22 ± 1.53[d]	100.00	13.64
2/24	8.71 ± 0.72[c]	77.66	8.27
4/24	7.38 ± 0.42b[c]	65.78	5.69
1	6.69 ± 0.97[bc]	59.63	14.50
2	5.19 ± 0.75[b]	46.26	14.45
4	2.07 ± 0.14[a]	18.45	6.76
7	1.90 ± 0.14[a]	16.93	7.37
14	1.76 ± 0.14[a]	15.69	7.95
21	1.25 ± 0.07[a]	11.15	5.60
28	0.99 ± 0.01[a]	8.82	1.01
42	0.93 ± 0.09[a]	8.29	9.73
56	0.67 ± 0.09[a]	5.97	13.43

* Results are expressed as means ± standard deviations (S.D). Means in a column with different coefficients (a-d) are significantly different ($p \leq 0.05$ and $LSD_{0.05}$ is 1.79).

$$R=A_0e^{-a\alpha t} + B_0e^{-b\beta t} \qquad (1)$$

where R is residue level at t days, A_0 and B_0 are the residue concentrations at t=0 and aα and bβ are the loss rate constants for the first and second phase model,

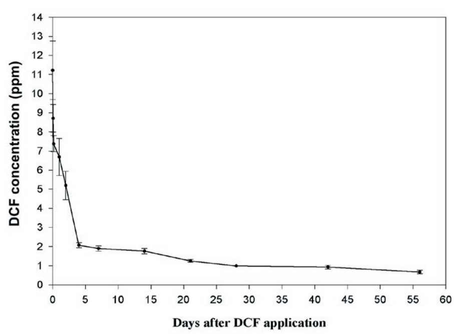

Figure 1. Dissipation curve for DCF. Data are expressed as means ±± S.D (n=3).

Table 3. Statistical parameters of DCF dissipation from treated date fruits.

Statistical Parameters	Value
A_0 (ppm)	7.71
B_0 (ppm)	1.85
aα (day^{s}-1	0.51
bβ (day^{s}-1	0.02
$T_{1/2a}$ α (days	1.35
$T_{1/2b}$ β (days	38.52
Regression coefficient	0.98

respectively. The half-life ($t_{1/2}$) of the exponential decay was calculated according to equation (2).

$$t_{1/2} = (2.303 \log 2)/\text{rate constant} \qquad (2)$$

The data indicate that there was a faster rate of DCF loss in the first phase than the second one (Table 3). This is clearly reflected in the $t_{1/2}$ values, where the half-lives of DCF in date fruits were estimated to be 1.35 and 38.52 days, for the first and second phase models, respectively.

Conclusion

Although dicofol disappears rapidly from date fruits where the residue level 7 days after DCF spraying was 1.90 ppm, less than the maximum residue limits for fruits and vegetables of 2 ppm, pesticide residue monitoring programs should nevertheless be implemented to ensure minimal residue levels in date fruits and to check compliance with the existing regulations. Also, the described procedure is simple and less labor intensive and troublesome emulsions, such as those frequently observed in liquid-liquid partitioning, did not occur. Further, these were satisfactory recoveries. The sensitivity of detection of DCF in the present study was 0.24 ppm, which is equivalent to that obtained by Fernăndez *et al.* (2001) when DCF was analyzed by GC-ECD.

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