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Determination of chlorpyrifos 20% EC (Dursban 20 EC) in scented rose and its products

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Abstract

The method for determination of chlorpyrifos is validated and dissipation behaviour of residue in scented rose and percent transfer in different products is described. GC–electron-capture detection with a HP-1, 30 m × 0.53 mm, 3.0 μm capillary column and nitrogen at 1 ml/min was used in the study. Plant matrices studied were: leaves, flowers, soil, rose water, absolute and concrete. Detector response linearity and sensitivity, limit of detection and determination, percent recovery were determined based on area response (mm^2) of the standard. Analytical field and laboratory samples (rose water by hydro-distillation of the flowers, concrete and absolute by hexane extraction and condensation) were analysed for evaluation of the method. Samples were extracted with acetone, partitioned with water, saturated sodium chloride solution and dichloromethane. The organic layer was rotary-evaporated to 2 ml for cleanup with silica–carbon column. The column was eluted with dichloromethane–toluene–acetone (10:2:2, v/v/v) and the derived solution was rotary-evaporated to 5 ml for end analysis. Matrix enhancement effect was observed for leaf and soil samples for which corrective approach was followed to compensate for overestimation of the residue. Limit of detection for chlorpyrifos standard was 0.05 mg/l with good linearity of detector response (R^2 = 0.99). Percent recovery ranged from 78 to 117% in different plant matrices (fortification level 1, 4 and 8 mg/l). Dissipation behaviour showed that chlorpyrifos was below detection limit by the 12th day of application on the scented rose with half life of 3.40 days on leaves and 3.10 days on flowers at 0.1% dosage. Percent transfer studies showed that 5.71, 46.91 and 38.80% of the residue from flowers was transferred to rose water, concrete and absolute, respectively.

Keywords: Chlorpyrifos; Dursban; Scented rose; Residue

1. Introduction

Scented rose (*Rosa damascena* Mill.) is one among the important commercial aromatic and medicinal plants whose commercial cultivation in India dates back to Mughal times (16th Century). The plant produces flowers for 25–35 days once in a year during early summer, which yield valuable products like rose oil, rose water, concrete, absolute, dry petals and are also used in traditional medicines and teas [1,2]. All these rose products have world wide domestic and industrial acceptance. Over the centuries, damask rose oil has been used in high class perfumes and cosmetics. Rose water finds use in dermal and ophthalmic diseases. In India, scented rose is cultivated in the states of Jammu and Kashmir, Himachal Pradesh, Uttar Pradesh, Rajasthan and Bihar producing about 150 kg rose oil besides making major quantity of rose water and a small quantity of blended rose oil called ‘attars’. In the world scenario, Turkey and Bulgaria are the leading countries in the production of rose oil while Morocco produces mainly rose water. During the past 15 years, Turkey has become an important producer of rose oil and concrete [3]. Egypt, China, France, New Zealand and Russia are among other countries, which also produce rose products [4,5].

Scented rose is attacked by several insect pests causing economical losses. Aphids, thrips, rose midge, chaffers, beetles, red scale, mites, termites, caterpillars, leaf hoppers,

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rose leaf rollers, borers and saw flies are common insects of scented rose [6,7]. Management of the pests by all possible means becomes an obligatory need to have profits from this crop. Pesticides invariably are important tools in the pest management programs, but their application results in the deposition of the residues, which are detrimental to human health. Rising awareness for the ever increasing residue problem has been strictly addressed by several agencies like Codex Alimentarius Commission of the Food and Agriculture Organization of the United Nations (FAO), World Health Organization (WHO), European Community (EC), advocating strict maximum residue limits (MRLs) [8–11]. Work on the residue aspect of pesticides used on scented rose and its products is scanty, however, in Germany residues of methiocarb were measured by high-performance liquid chromatography [12].

Several multiresidue analytical methods have been reported and used for the determination of pesticides in different matrices world over [13–15] while detailed manuals on multiresidue and single pesticide for several compounds covering almost all the existing group of pesticides in different crops (harvested commodities) have been documented [16,17]. However, little information is available on analytical methods (single/multiresidue) or validation and evaluation of different methods for pesticide residue in medicinal and aromatic plants. Therefore, present study was conducted to validate the multiresidue method for chlorpyrifos [16,17] with the new matrices as such or with slight modifications and then its application in the determination of dissipation behaviour of chlorpyrifos residue in scented rose. The study was also aimed to determine percent transfer of the residue in different products when manufactured in the laboratory from the residue laden flowers of rose.

2. Experimental

2.1. Chemicals and solvents

Formulated product, Chlorpyrifos 20% (w/w) EC (Dursban 20 EC, De-Nocil Crop Protection, Mumbai, India) was procured from the local market. Reference standard of chlorpyrifos ($C_9H_{11}Cl_3NO_3PS$; *O*,*O*-diethyl-*O*-3,5,6-tichloro-2-pyridyl phosphorothioate) for residue analysis was obtained from Dr. Ehrenstorfer (Augsburg, Germany) with certified purity of 99.0%. Acetone (analytical reagent grade), Dichloromethane (GR grade), *n*-hexane (analytical reagent grade), toluene (analytical reagent grade), activated carbon (Merck India, Mumbai, India), sodium chloride (analytical reagent grade), sodium sulphate anhydrous (analytical reagent grade) (s.d. fine-Chem, Mumbai) while silica (60–120 mesh) for column chromatography was purchased from Qualigens Fine Chemicals, Mumbai, India.

Saturated solution of sodium chloride in distilled water and eluting mixture: dichloromethane–toluene–acetone (10:2:2, v/v/v) were prepared. Stock solution of chlorpyrifos was prepared at 100 mg/l in acetone in a volumetric flask and stored at −4 °C. Working solutions of 1–10 mg/l were prepared from the stock solution for the preparation of standard curve to estimate the linearity and sensitivity of response. The limit of detection (LOD) was determined by serial dilution of 1 mg/l standard solution.

2.2. Apparatus and equipments

Orbital Shaker, distillation apparatus consisting of 2 l flask with Fridrich's condenser and stopper, reflux assembly consisting of 500 ml round bottom flask and Leibig condenser (500 mm effective length), extraction columns (50 cm × 30 mm i.d. with sintered disc and screw type PTFE stop cock, rotary vacuum film evaporator (Perfit, India), gas chromatograph (Hewlett Packard, 5890 Series II Gas Chromatograph, Avondale, PA, USA) equipped with a ^{63}Ni electron capture detection (ECD) system, HP-7673 injector and controller, controlled by HP-3365 Series II Chemstation software were used in the study.

The GC system was equipped with a split–splitless injector operated in a split mode (split ratio = 50:1), a nonpolar capillary column, HP-1, 30 m × 0.53 mm, 3.0 μm film thickness, coated with 100% dimethylpolysiloxane gum (Hewlett-Packard) and nitrogen (99.99% purity) was used as carrier gas (1 ml/min column flow). Analysis was carried out with the injection port set at 250 °C, detector 300 °C and the oven temperature programmed at 180 °C for 2 min, then ramped at 2 °C/min to 204 °C and finally maintained for 1 min (total run time: 15 min). Column equilibration time of 10 min was set at 250 °C for complete elusion of the impurities if any from the column before the next injection.

2.3. Experimental site and sampling

Field experiment was conducted at Chandpur scented rose farm-IHBT, Palampur (HP), India during May 2003. Location, weather conditions and main physico-chemical properties of soil (pH, organic matter and texture) are presented in Table 1.

Chlorpyrifos 20% EC was sprayed with a calibrated knapsack sprayer at 0.1% dosage (recommended) and 0.2% dosage (double the recommended). Samples were collected in the early morning hours, 06:30 AM IST (just before sunrise) at 0 (1 h after spraying), 1, 3, 5, 7, 11, 14, 21 days after treatment. Because fresh flowers have higher concentration of oil whose loss occurs due to sunlight. For quality products and routine farm practices, flowers have to be collected before sunrise. Leaf, flower and soil (0–10 cm of the plough layer) were collected and brought to the laboratory for chlorpyrifos residue analysis.

2.4. Calibration and LODs determination

Standard solutions (1 μl) were injected into the GC and graphical representation of the response (mm^2)/concentration (mg/l) curve was plotted for the

Table 1
Experimental site characteristics (May, 2003)

Field	
Latitude	76°33′29″ East
Longitude	32°6′20″ North
Elevation	1356 m amsl
Temperature	30.22 ± 0.40 °C (maximum), 18.67 ± 0.47 °C (minimum)
Relative humidity	37.98 ± 2.79% (maximum), 15.08 ± 1.96% (minimum)
Rainfall	41.41 mm
Sunshine	1737.18 ± 75.96 umols/m^2
Soil (mean ± S.E.M.)	
Temperature	31.34 ± 0.41 °C (maximum), 23.31 ± 0.61 °C (minimum)
pH	5.21 ± 0.13
Organic matter content	2.32 ± 0.30
Soil texture	Clay loam silt: 23.03%, clay: 29.14%, sand: 47.83%

areas obtained. Linearity regression analysis and sensitivity was determined from the results obtained. Similarly, serially diluted standard solutions of 1 mg/l stock were injected and the LOD was determined based on the lowest level of standard concentration detected.

2.5. Quantification of residue

For recovery experiments (% recovery) three concentrations (1, 4 and 8 mg/l) were prepared from the stock solution. Recovery was done from the spiked samples of residue free plant material, viz. leaves (20 g), flowers (20 g) with 2 ml of the spiking concentrations, soil (100 g), rose water (100 ml), absolute (100 ml) with 10 ml of the spiking concentrations and concrete (10 g) with 1 ml of the spiking concentrations. Spiked samples were allowed to stand for 4 h before analysis. Percent transfer of the pesticide was studied by spiking rose petals with the formulated product and further residue deposit on rose petals and products after processing were quantified. The experiment was replicated thrice.

To determine LOD, percent recovery and percent relative standard deviation (R.S.D.), fortified samples (1 μl) were injected into the GC and the response (mm^2) obtained for the pesticide identified based on retention time of the standard was recorded (Fig. 1). The residue '*R*' expressed in mg/kg of an identified compound was calculated from the following equation:

$$R = X\frac{F_A V_{end} W_{St}}{F_{St} V_i G}$$

where F_A is the peak area obtained from V_i (in mm^2), V_{end} the terminal volume of sample solution from 2.8 (in ml), W_{St} the amount of compound injected with standard solution (in ng), F_{St} the peak area obtained from W_{st} (in mm^2), V_i the portion of volume V_{end} injected into gas chromatograph (in μl), G the sample weight (in g) and X is the portion of filtered extract taken (1/5 = 5).

2.6. Preparation of different scented rose products

2.6.1. Rose water

Rose water was prepared by hydro-distillation of the rose flowers. Flowers (100 g) were put in 2 l round bottom flask with 1 l distilled water. Friedrich's condenser was attached and the unit was heated on a heating mantle with temperature control arrangement. One hundred millilitres of the condensate was collected per distillation. The condensate was cooled to room temperature and further processed for residue determination.

2.6.2. Rose absolute and concrete

Rose absolute and concrete (wax) produced from the scented rose is the yield of a single extraction process. Initially rose flowers (500 g) were coarsely chopped and were placed in glass container and *n*-hexane was poured into it to a level where the material was fully immersed in it. The container was left for 1 h with intermittent stirring and then hexane was filtered and collected. The material was re-extracted twice and the combined extract was then rotary evaporated to near dryness (to approximately 5 ml) at 40 °C at high vacuum and then to complete dryness at low vacuum. The semisolid material obtained was then redissolved in 100 ml ethanol at 55 °C and allowed to stand in a beaker for wax formation. Concrete was collected and weighed (on an average 8 g concrete was obtained per 500 g petals). Filtered alcoholic portion (100 ml) was taken in a 500 ml flask attached to a Leibig condenser (reflux assembly) and refluxed at 70 °C for 1 h to prepare absolute. The absolute obtained was cooled to room temperature and further analysed for residue determination.

2.7. Extraction

Leaf and flower materials collected from field were finely chopped and each plant material was mixed thoroughly and then 20 g samples were drawn for analysis. One hundred millilitres for rose water, absolute and 8 g for concrete were taken as samples for extraction. For extraction of residue from concrete (semisolid material), the sample was ground with silica gel (concrete:silica 1:5 g) to increase the surface area in contact with the solvent for good recovery of the pesticide.

The analytical samples were taken in 500 ml conical flasks with 200 ml acetone and homogenized for about 30 min on an orbital shaker. Homogenates were suction filtered through a moistened filter paper in Buchner funnel followed by rinsing with 50 ml of acetone. One-fifth of the filtrate was then partitioned with 250 ml water, 25 ml saturated sodium chloride solution and 50 ml dichloromethane in a 1 l separatory funnel. The dichloromethane layer was separated and the left aqueous phase was re-partitioned with two 50 ml portions of dichloromethane. Vigorous shaking for at least 2 min during partitioning is necessary for better recoveries. The combined dichloromethane phases were then dried on 30 g sodium sulphate for 15 min with intermittent stirring, filtered the extract through a fluted filter paper and rinsed the flask and filter

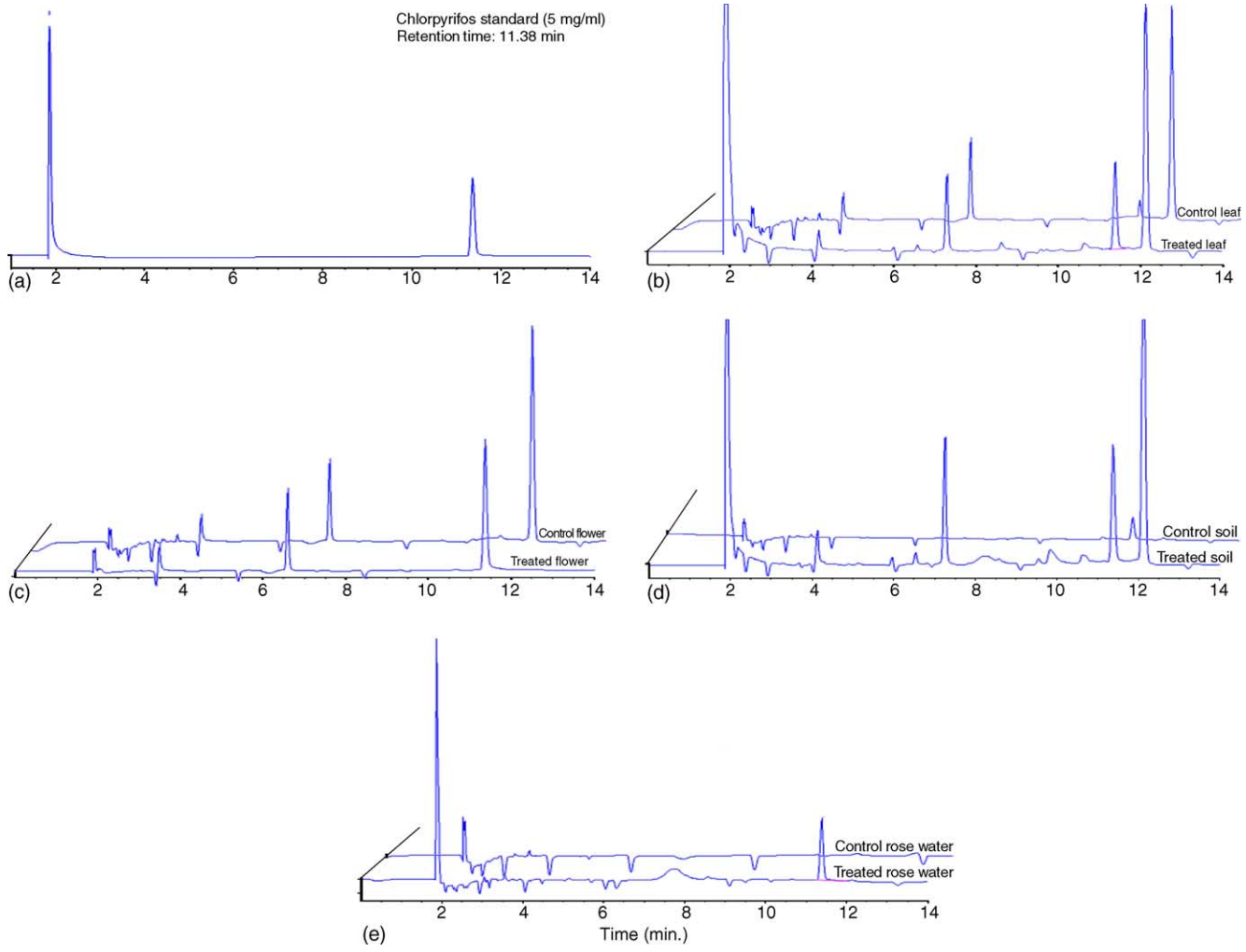

Fig. 1. Residue analysis of chlorpyrifos on 0 day at 0.1% dosage in scented rose: (a) chlorpyrifos standard, (b) leaf, (c) flower, (d) soil and (e) rose water.

paper with 30 ml dichloromethane applied in three portions. The filtrate was rotary-evaporated to about 2 ml and removed the last traces of solvent by swirling the flask manually. Finally, the residue was redissolved in 10 ml dichloromethane for cleanup.

2.8. Cleanup

Cleanup was done with silica–carbon column. Chromatographic column was first filled with 1 cm layer of eluting mixture and then silica gel slurry (5 g silica in 15 ml eluting mixture) was poured onto it and allowed to settle. Supernatant was drained off to the level of silica gel. Then 15 g silica and 1 g activated carbon were mixed in a 50 ml beaker to form flowable slurry. Poured the carbon–silica gel mixture onto the silica gel layer in the column, at first slowly and then in a gush. After settling, the eluting mixture was drained to a level 2 cm above the packing and topped with 5 g sodium sulphate and the column was pre-washed with 50 ml eluting mixture.

The dichloromethane solution obtained from extraction step was transferred to the column, completing the transfer with a total of 5 ml dichloromethane. The column was eluted with 140 ml of eluting mixture, which was collected in a 250 ml round-bottomed flask. Rotary evaporated the eluate to about 30 ml and transferred it to a 50 ml round-bottomed flask and again rotary evaporated it to about 2 ml (solution should not be taken to complete dryness under vacuum). Transferred the derived solution to a 5 ml volumetric flask to make up the final volume with acetone (V_{end}). Injected an aliqout of this solution (V_i) for analysis into the gas chromatograph.

3. Results and discussion

3.1. Method validation

Chlorpyrifos was identified on the basis of retention time (11.38 min) obtained from the working solutions of concentrations (1–10 mg/l) when injected into the gas chromatograph (Fig. 1). LOD was obtained from the area response of serial dilutions of 1 mg/l standard solution. Closely lying LODs (n = 5) were grouped into a class, i.e. 0.05 mg/l [18].

Table 2
Percent recovery (mean ± S.E.M. (%)), relative standard deviation (%), and limit of determination (LOD in mg/l) for the fortified samples (n = 3)

Plant matrix (mg/l)	Recovery (%)	R.S.D. (%)	LOD (mg/l)
Leaf			
1	117.00 ± 5.68	9.85	0.5
4	108.00 ± 5.29	9.16	
8	101.67 ± 3.48	6.03	
Flower			
1	105.67 ± 4.63	8.02	0.25
4	96.23 ± 2.60	4.51	
8	98.62 ± 3.48	6.03	
Rose water			
1	96.48 ± 1.29	2.24	0.07
4	97.58 ± 0.52	0.90	
8	98.08 ± 0.10	0.17	
Concrete			
1	78.46 ± 4.67	8.08	1.20
4	83.23 ± 5.49	9.50	
8	85.33 ± 4.26	7.37	
Absolute			
1	96.33 ± 1.76	3.05	0.68
4	98.93 ± 0.58	1.01	
8	98.68 ± 0.50	0.87	
Soil			
1	116.33 ± 9.56	16.56	0.5
4	108.00 ± 7.00	12.12	
8	99.67 ± 6.69	11.59	

The values show good linearity (0.99) and LOD (0.05 mg/l) thus, making GC–ECD a sensitive instrument for analytical study of chlorpyrifos. Lower LOD (0.05 mg/l) suggests that chlorpyrifos can be detected in trace quantities with good precision provided the extraction procedure adopted gives good recoveries.

Good recoveries were obtained by the fortification at three concentration levels of different scented rose matrices. It is evident from the percent data that chlorpyrifos showed good recoveries in all the type of matrices. However, in the case of leaf and soil samples, recoveries above 100% were observed which are attributed to a matrix enhancement effect (Table 2, Fig. 1). Matrix induced chromatographic response enhancement is a phenomenon that causes high recovery results for some pesticides in food [19]. This effect is more pronounced for polar pesticides as reported by Luke et al. in 1981 [20] and is influenced by many factors such as pesticide character, matrix type, state of GC system and analyte/matrix concentration [21].

As a common practice, analytical procedures commonly used in food analysis can be employed without any fundamental modifications for examination of various matrices of plants/animal origin. On that account the same strategies to prevent/minimize potential matrix effects should be applied when analysing organic contaminants in various environmental or other biotic samples [21]. Chromatographic response enhancements were discussed in depth for the first time by Erney et al. [22]. Similar matrix enhancement effect was observed in leaf and soil samples with recoveries ranging from 101.67 ± 3.48 to 117.00 ± 5.68% and 99.67 ± 6.69 to 116.33 ± 9.56%, respectively. Higher recoveries at lower fortification levels can be attributed to lower analyte concentration in relation to matrix concentration [14]. In case of concrete, the recoveries ranged from 78.46 ± 4.67 to 85.33 ± 4.26% implying that some percentage might have been retained when the waxy semi-solid material is dispersed in silica gel keeping some of it unexposed to the extracting solvents (Table 2).

Matrix-induced peak enhancement remains a problem due to the co-extractives competing for the active sites [23–26]. Higher recoveries (145–247%) had been reported earlier in many organic matrices including apricot and wine samples [14,27–29]. Several authors have proposed a variety of solution for this problem but practically could not eliminate it. Standards prepared in blank matrix induced effects for quantitation [24,25,29–31] or using clean up procedures alone or in combination with the techniques (more rigorous clean up or use of GC columns with fewer active sites) [26], daily column-cutting (after a set of 10–12 samples) [32] are cited. A different approach, proposed by Egea González et al. [33] applied correction factors to obtain the data. Calibration curves obtained by using external standards (i) pure solvents and (ii) matrix method standards were used for comparison. In our validation study and determination of residue in scented rose leaf and soil, this approach was followed with the correction factors obtained by statistical validation of the data set obtained from residue free plant matrix. The correction factor approach reduced both wet and time of analysis [22]. Schenck and Lehotay [23] had shown that even extensive clean up of crude extracts obtained from various vegetables, fruits and cereals, carried out by contamination of several type of SPE cartridges (sorbents: graphitized carbon, primary/secondary amines and strong anion exchanger) could not assure that no recovery exceed above 100% for any organophosphorous pesticides/matrix combination, although reduced matrix enhancement effects may be obtained. Rose water showed the lowest limit of determination (0.07 mg/l) while rose concrete was the highest (1.20 mg/l) whereas, in leaf, flower, soil and absolute were 0.5, 0.25, 0.5 and 0.68 mg/l, respectively (Table 2).

3.2. Chlorpyrifos residue in scented rose and its products

Field experiment at Chandpur rose farm was conducted to study the dissipation of chlorpyrifos and its transfer to different products with the application of this method. Mainly rose water was evaluated to study the residue dynamics as it is the prime product of commerce. The data is presented in Table 3. Initial deposit on leaves at 0.1% and 0.2% dosages were 10.74 ± 0.15 and 19.38 ± 0.77 mg/kg, which dissipated to 1.04 ± 0.23 and 1.09 ± 0.34 mg/kg, respectively, by the 11th day. The residue was below detection level (BDL) on the 14th day. The rate of degradation when fitted to a classical first-order rate equation calculated from the regression

Table 3
Chlorpyrifos residue in scented rose and its products (mg/kg) at 0.1% and 0.2% dosages (n = 3)

Plant matrix	Concentration (%)	Mean residue ± S.E.M. (mg/kg) at different time intervals							
		0	1	3	5	7	9	11	14
Leaf	0.1	10.74 ± 0.15	8.31 ± 0.52	7.11 ± 0.30	5.20 ± 0.11	3.14 ± 0.19	1.91 ± 0.08	1.04 ± 0.23	BDL
	0.2	19.38 ± 0.77	16.46 ± 0.69	14.54 ± 0.18	9.24 ± 0.75	4.00 ± 0.12	2.07 ± 0.10	1.09 ± 0.34	BDL
Flower	0.1	13.58 ± 0.46	7.44 ± 0.19	6.18 ± 0.51	4.84 ± 0.09	1.66 ± 0.14	BDL	BDL	BDL
	0.2	23.54 ± 0.80	8.94 ± 0.52	8.32 ± 0.07	6.14 ± 0.05	2.52 ± 0.13	1.74 ± 0.41	BDL	BDL
Rose water	0.1	0.63 ± 0.03	0.47 ± 0.02	0.40 ± 0.01	0.26 ± 0.01	BDL	BDL	BDL	BDL
	0.2	0.95 ± 0.05	0.51 ± 0.02	0.46 ± 0.01	0.32 ± 0.01	0.17 ± 0.02	BDL	BDL	BDL
Soil*	0.1	1.65 ± 0.09							0.14 ± 0.02
	0.2	2.15 ± 0.14							0.11 ± 0.04

* No specific pattern observed, therefore data not shown for Day 1–11.

analysis of natural logarithmic transformation (ln of residue in mg/kg) versus time interval (days) from the data of residue dissipation on leaves at 0.1% and 0.2% dosages yielded good linearities (0.97 and 0.95) and sensitivities (−0.20 and −0.27) ($y = -0.20x + 2.47$ and $y = -0.27x + 3.21$), respectively. Both the concentrations showed similar dissipation rate with the residue decreasing to zero around 12th day after treatment as evident from the extrapolation of the lines of best fit. Half $life_{(0.1\%)}$ was observed to be 3.40 days on the treated leaves and 3.10 days on flowers. When flowers from the treated plots were analysed, initial deposit was little higher, i.e. 13.58 ± 0.46 and 23.54 ± 0.80 mg/kg, respectively. Higher residue deposit was due to the trapping of spray liquid in the folds of petals of partially/fully opened flowers. After plucking of 0 day flowers of the whole field, flowers analysed thereafter as per scheduled time showed lesser residue deposit in comparison to the leaves largely due to crop dilution in freshly opened flowers. The residue was detected upto the 9th day only, on flowers (Table 3).

When rose water was prepared from the field collected flowers subjected to hydro distillation in the laboratory, some of the residue was transferred to the product. The data showed that 0.63 ± 0.03 and 0.95 ± 0.05 mg/kg of the residue in rose water was transferred during distillation from 13.58 ± 0.46 and 23.54 ± 0.80 mg/kg deposit of flowers. By the 7th day, the amount of residue transfer reached below detection level at 0.1% and 0.17 ± 0.02 mg/kg at 0.2% dosage (Table 3). Thereafter, it was not detected in rose water. Only 5.71 ± 0.43% of the residue was transferred in the process of hydro distillation (Table 4). Thus, the observations suggest that a waiting period of at least 7 days is necessary to have rose water with low/safe level of residue contamination.

Rose concrete and absolute showed good percent transfer rate of the pesticide from the base material to the final products. High percent transfer in concrete (46.91 ± 1.94%) and absolute (38.80 ± 1.03%) are due to direct solvent extraction process which leaches out most of the residue from the flowers and further divides them into both the products (85.71%) (Table 4). Thus, these products should not be processed from the treated crop before a waiting period of at least 10 days.

Table 4
Percent transfer of chlorpyrifos to different products (n = 3)

Plant matrix	Mean transfer (%)	S.E.M. (%)	R.S.D. (%)
Rose water[a]	5.71	0.43	0.86
Concrete	46.91	1.94	3.37
Absolute	38.80	1.03	1.79

[a] Percent transfer calculated from residue data at 0.1% concentration.

Pesticide residue in soil showed no specific pattern as the deposition was uneven on the undersurface of the scented rose bushes. The spray which finally reached the surface was nonuniform due to leaf shading and dripping of the spray from the leaves differently under the bushes. Therefore, dissipation pattern was not studied and residue was detected just for the presence of pesticide till it degraded to nearly zero. The obtained data showed that chlorpyrifos persisted for longer time in soil which extended over to the 14th day after treatment but could not be detected by the 21st day (Table 3).

4. Conclusion

Residue analysis in medicinal and aromatic plants in these days is gaining ground with the rising concern of environmental pollution due to pesticides. The method described for residue analysis of chlorpyrifos is suitable for determination of residue in scented rose which can be extended to other medicinal and aromatic plants. It is also suitable for analysis of residue in the products directly or with suitable modifications depending on the nature of the product. In general, the method can be suitably applied to organophosphate compounds, but needs to be validated for the respective plant matrices due to matrix interferences, a commonly encountered problem with this group. Overlooking this aspect would result in overestimation of the residue than the actual amount present and thus, rejection of some products which are probably residue free or with residue below the permissible limits.

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