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Residues measurement of common insecticides in *Berberis vulgaris* by gas chromatography in Iran

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Abstract

Different pesticides have been used to control pests of barberry (*Berberis vulgaris)* in south Khorasan province, Iran. Due to use barberry in both raw and cooked, identification and quantification of residues of common pesticides (Diazinon, Oxydemeton-methyl and Phosalone) in barberry was monitored by using GC-MS. To achieve a suitable method for extraction and purification, four valid methods such as QuEChERS Extraction, Static extraction, Changing PH and Solid Phase Extraction (SPE) were used. To reduce matrix effects in measurements, the addition standard used and the resulting signal level from GC by using pesticides standards calibration curves were measured. Extraction with acetonitrile solvent and scan mode of GC-MS showed that most of the barberries were contaminated by the pesticides. Four kind of different extraction method were compared with each other in spiked distilled water and then spiked barberry samples. The SPE extraction seemed to give slightly lower recoveries for the sample tested. Although Changing PH and Static extraction were so quick, but had the worst results. The results obtained confirmed that QuEChERS Extraction method may be used to extract pesticide residues from barberry. Other pesticides also found with a very low concentration by GCMS full scan method, such as Dimethoate, Dursban & Acetamyprid, that may has been used in the region and came as drift.

Keywords: Pesticides, Residues, GC-MS, Barberry, extraction

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1. Introduction

Most foods of plant origin are grown using pesticides. Chemical control of weeds; insects, plant diseases and other pests has increased agricultural productivity. However, these economic benefits are not without risk to human health and environmental damage (Calatayud *et al.*, 2016b). Many pesticides and their residues are known to be contributory factors in several diseases such as cancer, heart diseases, Alzeheimer's and Parkinsonism. Feed and fodder offered to animals are often contaminated with pesticide residues and after feeding, these residues assimilated into the body systems of the animals (Peterson, 2003). Frequent spraying and harvesting early after spraying, non-compliance of the currency, submit it to the market and use of these products as raw and fresh seriously threatens the health of consumers (Rumpold & Schlueter, 2013). Less than one percent of pesticides affects the target and more than 99 percent of pesticides reach into the environment. It is necessary to examine the remains of pesticides in the country (Sanchez *et al.*, 2010). More importantly, much of the first part of pesticides find their way into water sources, and directly or indirectly enter the food chain, and may eventually enter the human body and other living organisms (Dirr, 2015). The second most common Business-related diseases, are skin diseases, as 15 to 25% of reports on pesticides, related to skin diseases (Asoudeh, 1999).Water is one of the important components in life cycle. Importance of quality, conservation and development is steadily rising and pesticides are considered one of the main water pollutants. (Jazayeri, 2002)

Barberry (*Berberis vulgaris* L.) owed its popularity to its apparent usefulness. By the seventeenth century, a list of the bushes' reported beneficial medical properties had grown substantially to include use against scurvy, bladder trouble, jaundice, constipation, loss of appetite, and fever. The barberry bush also had proven to be quite a useful plant in many other ways. The juice extracted from the barberry was used for making a "good-tasting, healthful English drink called 'punch'." Water in which the bark was boiled served as a mouthwash. Its red berries were used for making jellies, while the bark and roots, with their characteristic yellow pigment, were used as dye (Peterson, 2003).Although barberries are more known as medical plants, it has been used most often as food additive in Iran. Dried barberries are used for adding to food as flavor. It is used especially with rice (Tehranifar, 2003). Iran is the world's largest producer of *Berberis vulgaris* L. (Ranunculales: Berberidaceae). South Khorasan Province has the 98% of land under cultivation of this product in Iran and has the Production of 98.7% of the country's Barberry (Kafi & Balandari, 2002). The most important pests in barberry gardens of South Khorasan province is as follows: *Syrista parreyssi* Spinola, *Polyphylla adspersa* Motschulsky, *Liosomaphis berberidis* Kalt, *Parlatoria oleae* Colvée; in most cases, these pests control with spraying methods (Moazzen, 1993). No studies exist on measurement of residual pesticides in barberry. In this study we examined four different extraction methods for the detection of three most common pesticides used to control pests of barberry in Iran and introduce the best method.

2. Materials and Methods

We harvested barberries from Haji Abad city in the Zirkouh region (South Khorasan province), X701984, Y3736674, 1400 meters a.s.l.). Obtained Samples placed in plastic bags in cool and dark conditions to transfer to the laboratory.

In the laboratory samples encoded, then extracted, and based on the pesticides found in scanning method by GCMS, that were Diazinon, Oxydemeton methyl and Phosalone different concentrations of pesticides were added, and each sample separately identified and tested by code.

To detect pesticides in spike samples in 0.01,0.1.1 mg/kg to the pesticide-free Barberries, the seeds of the plant barberry isolated and in proper containers and away from light until the time of the test kept in the refrigerator(4°C).

The following table has been prepared, the aqueous extract samples spiked, vortex and keep in suitable containers (Eppendorf Tubes) and kept refrigerated until injected into the Gas chromatograph.

Table 1- manufacturing pesticides Standards and inject to pesticide-free aqueous extract and distilled water

NO.	Pesticide	volume(mg/kg)	No. Of Repeat.
1	Diazinon	0.01 0.1 1	2
2	Oxydemeton methyl	0.01 0.1 1	2
3	Phosalone	0.01 0.1 1	2

Pesticides Standards obtaind from Sigma-Aldrich have been made and obtained by the formula N1V1 = N2V2.

Extraction of Pesticide Residues:

In order to achieve a suitable method for the extraction and purification of it, several methods were used as follows.

- QuEChERS Extraction: QuEChERS (quick, easy, cheap, effective, rugged, and safe)

Weighed 15 g of homogenized (hydrated at least 80%) barberry in 50 ml centrifuge tube, Added 15 ml 1% acetic acid in acetonitrile and shacked briefly, Added 6 g of anhydrous magnesium sulfate and 1.5 g of sodium chloride shacked by hand for 1 minute then Centrifuge at 3700 rpm for 1 minute, Transferred a 1 ml aliquot of supernatant to a 2 ml centrifuge tube containing 150 mg anhydrous magnesium sulfate and 50 mg PSA then shacked for 30 seconds and Concentrated for GC-MS.

- Static extraction: 20 grams of crushed barberry, mixed with 60cc dichlorometan and 60cc aceton in 2 minutes, then leave them for 24hours in room temperature, passed from filter paper, then dried at 30 °C rotary, and 5 cubic centimeters of methanol added again, passed syringe filter, again dried at 30 °C Rotary, by adding a cubic centimeter methanol was ready for analysis.
- Changing PH: with concentrated sulfuric acid solution, at the same time while the color of the solution was changed, the PH measured constantly.
- Solid Phase Extraction (SPE):

1 g of crushed barberry mixed with 50 ml of distilled water. The solution was on the 60-70 °C heater and stear for 30 minutes. Added chloridric Acid Gutty until the PH became below 1. To make the color of the solution lower, smoothed by the filter paper and the Buchner funnel. Then passed the solution through the stationary phase C18 column (SPE). (Supelco) then washed by 1cc methanol, gathered in vial, and analyzed by GCMS.

Before using the column, 10 ml of methanol and 10ml of distilled water was passed through the column. Column type used: Supelco Supelclean LC-SAX 3 ML TUBES LOT NO.SP1274F.

The experiment repeated 3 times to ensure accuracy extraction and recovery steps have been completed and the results have been recorded.

The GC-MS conditions:

Cleantup, before injecting samples into GC-MS: To ensure that the device is clean, all parts of the device specially the injector and detector (interface)increased to the maximum possible temperature for some minutes.

Gas chromatography machine conditions has been set as follows:
Columns: (column of the type: non-polar) Restec-Rxi-1ms
Coloumoven: 120-270© 3 min Hold Rate: 5 Pressure: 80 KPa Injector: 250˚c
Start time: 3 min Coloumn flow: 1 ml/min Carrier gas: helium99.999
Detector settings (MS) as follows:
Interface: 300˚c Ion Source: 200˚c

Analysis:

Schimatzu Gas chromatography was used for analysis, (GC-MS QP 2010 PLUS), the barrel was splitless and the 30 meter Restek column -1ms®Rxi, with External diameter0.25 mm and internal diameter 0.25 µm. The carrier gas was Helium, with 99.9999 purity and One ml per minute flowed, and the Pressure was 80 kPa. The Injector temperature was 250 °C and Detector was 300 °C. The Detector voltage was 70 eV. The Oven Temperature at first 120 °C within 3 minutes, then up to 170 °C with 5 min/c speed. Then raise to 250 °C with 2 min/c speed, then up to 300 °C with 5 min/c speed and 3 minutes stop in this temperature. One micro liter of sample was injected in split less mode; the device tuned in SIM (selected ion monitoring) and by Electron Ionization analyzed (Table3).

Method Validation:

To realize the extraction of pesticides samples from the ones that brought to the laboratory, the pesticides standards in 0.01, 0.1, 1 mg/kg in methanol that have certified references material (CRM) were spikes three times. All the extractions and analytical methods were applied on distilled water and pesticide-free barberry, and the results were compared with the Data Of the collected barberries from cultivated gardens.

For recovery mode, added 0.01, 0.1, 1 mg/kg concentrations from pesticides into distilled water and the remaining amount applied in to the results (Table 4).

In order to reduce matrix effects in measurements, the addition standard used and the resulting signal level from GC by using pesticides standards calibration curves were measured.

To calculate Relative Standard Deviation (RSD), first, the standard deviation and standard error for each sample calculated and then RSD amount in each sample was determined for each pesticide. To determine the detection limit (LOD) and Limit of quantitation (LOQ) from standard solutions of each pesticide, they injected in to the GCMS and the Detection limit and Limit of quantitation for each pesticide achieved (Table 2).

Table 2- Profile samples collected from different areas to determine the residual pesticides

No.	Sampling sites	Cultivation area (ha)	Sampling date (2015)	Sampling date (2016)
1	Zirkooh Qaen	80	22.10.2015	22.10.2016
2	Zirkooh Qaen	80	22.10.2015	22.10.2016
3	Hajiabad Qaen	70	27.10.2015	24.10.2016
4	Hajiabad Qaen	70	27.10.2015	24.10.2016
5	Zohan Qaen	50	27.10.2015	26.10.2016
6	Qaen	100	27.10.2015	02.11.2016

Results

To investigate the probability of the effect of barberry color and other annoying factors on the chromatography, Spike operations and extraction and purification were applied on distilled water and pesticide-free Barberry.

Table 3 shows the wave number which were tracked by GCMS and the Mass spectrum of them.

Table 3- Characteristic Absorbance of pesticides

Element	Wave number(m^{-1})		
Diazinon	179	304	199
Oxydemeton methyl	88	169	60
phosalone	367	182	--

Figures 1-3 show the Chromatograms of pesticides Standards.

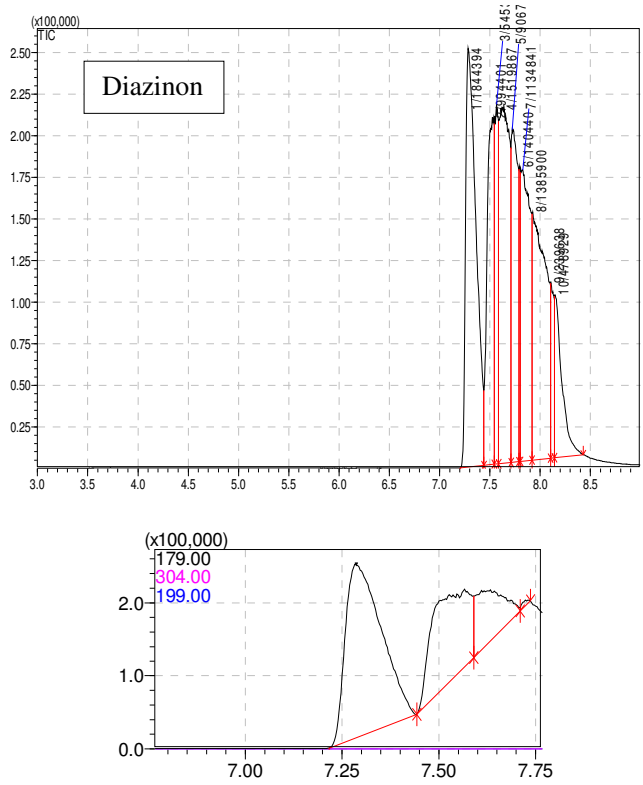


Fig.1– The chromatogram of Diazinon standard

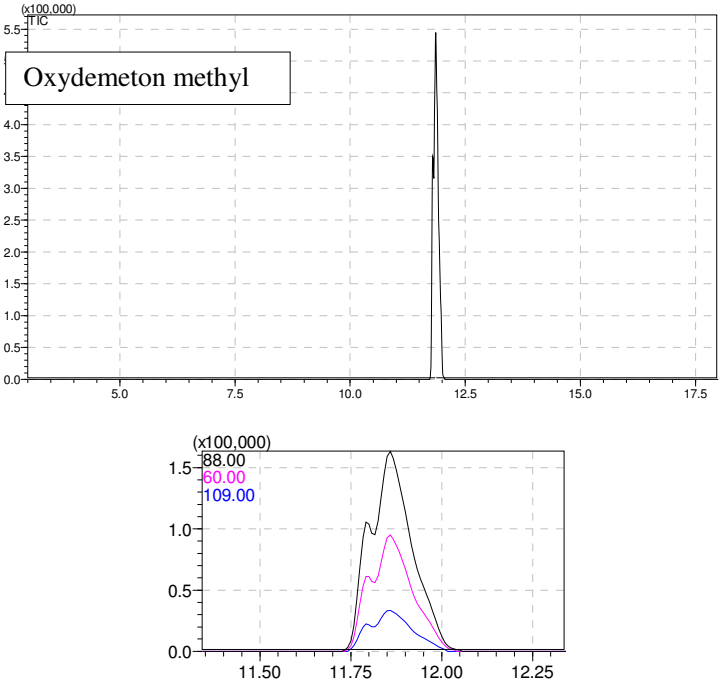


Fig.2– The chromatogram of Oxydemeton methyl standard

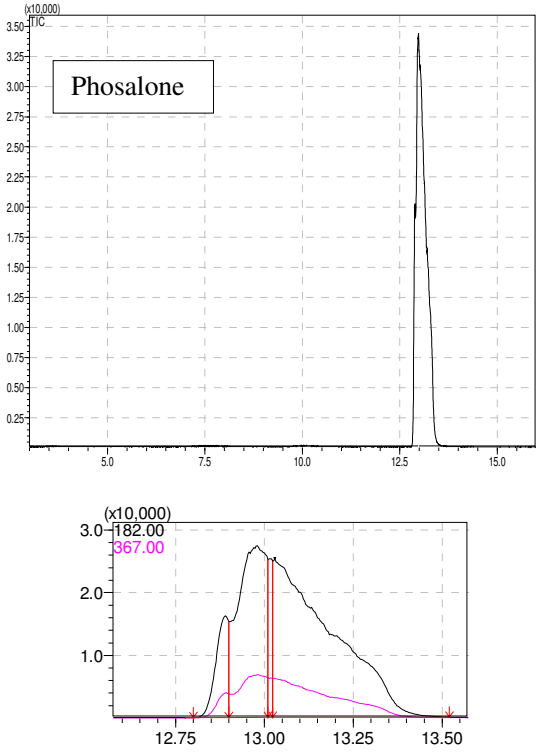
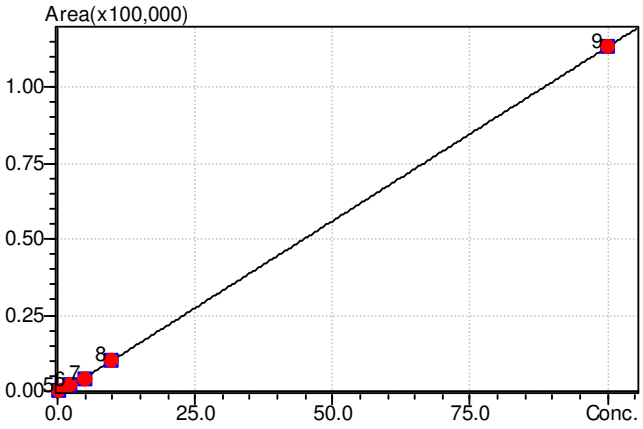


Fig.3– The chromatogram of Phosalone standard

Figures 4-6 show the Calibration curve of each pesticide standards at the concentrations studied.



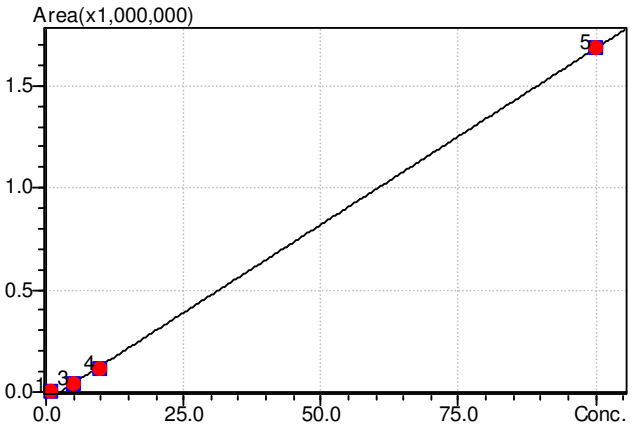
Y = 1146.15X - 1269.737
R^2 = 0.9998804
R = 0.9999402

Diazinon Standard(PPM)
Curve: Linear
Mean RF : 768.442
RF SD : 325.4632

Concentrations in ppm

Level1:	0.001
Level2:	0.005
Level3:	0.01
Level4:	0.1
Level5:	0.5
Level6:	1
Level7:	2
Level8:	5
Level9:	100

Fig.4– Calibration curve of Diazinon standard at the concentrations studied



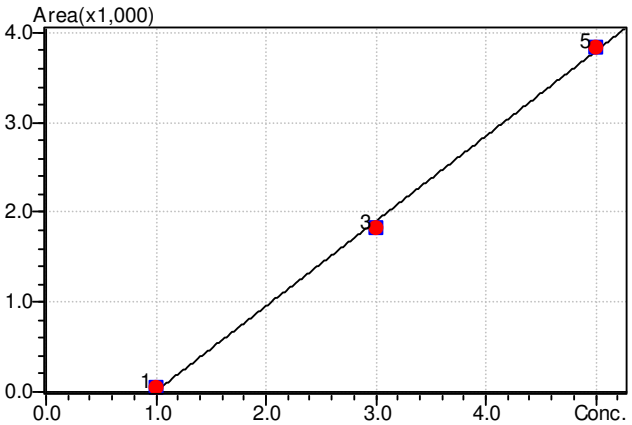
Y = 17255.06X - 42971.37
R^2 = 0.9993801
R = 0.99969

Oxydemeton methyl Standard (PPM)
Curve: Linear
Mean RF : 9050.502
RF SD : 6,444.315
RF %RSD : 71.20395

Concentrations in ppm

Level1:	0.001
Level2:	0.05
Level3:	0.01
Level4:	0.1
Level5:	10

Fig.5– Calibration curve of Oxydemeton methyl standard at the concentrations studied.



Y = 947.25X - 938.4167
R^2 = 0.9986533
R = 0.9993264

Phosalone Standard(PPM)
Curve: Linear
Mean RF : 474.7556
RF SD : 377.2870
RF %RSD : 79.46974

Concentrations in ppm

Level1:	0.001
Level2:	0.05
Level3:	0.01
Level4:	0.1
Level5:	1

Fig.6– Calibration curve of Phosalone standard at the concentrations studied.

Table 4 and 5 show four kind of different extraction method compared with each other in spiked distilled water and then spiked barberry samples. These samples were found in our routine surveillance until analysis. Since the SPE extraction seemed to give slightly lower recoveries for the sample tested, so the precision of QuEChERS is better than SPE extraction in Barberry.

Table 4- Different extraction method on distilled water with different density of pesticides.

No.	Pesticide	Extraction method	Spike Concentration (mg/kg)	Result (3times replication)
1	Diazinon	QuEChERS	0.01	0.098, 0.01, 0.099
			0.1	0.1, 0.099, 0.1
			1	1, 1, 0.99
2		Static extraction	0.01	0.0095, 0.0093, 0.0085
			0.1	0.066, 0.089, 0.094
			1	0.44, 0.9, 0.99
3		Changing PH	0.01	N.D, N.D, N.D
			0.1	0.089, 0.06, 0.044
			1	0.88, 0.90, 0.95
4		SPE	0.01	0.01, 0.01, 0.01
			0.1	0.01, 0.01, 0.01
			1	1, 1, 1
5	Oxydemeton-methyl	QuEChERS	0.01	0.009, 0.01, 0.007
			0.1	0.089, 0.099, 0.095
			1	1, 1, 0.98
		Static extraction	0.01	N.D, 0.009, 0.008
6			0.1	0.09, 0.09, 0.09
			1	0.88, 0.88, 0.89
7		Changing PH	0.01	N.D, N.D, N.D
			0.1	0.04, N.D, 0.06
			1	0.55, 0.90, 0.91
8		SPE	0.01	0.008, 0.009, 0.003
			0.1	0.090, 0.096, 0.099
			1	1, 1, 1
9	Phosalone	QuEChERS	0.01	0.009, 0.009, 0.009
			0.1	0.96, 0.92, 0.99
			1	0.99, 0.99, 0.99
10		Static extraction	0.01	N.D, N.D, N.D
			0.1	0.060, 0.060, 0.075
			1	0.86, 0.82, 0.99
11		Changing PH	0.01	N.D, N.D, N.D
			0.1	0.054, N.D, N.D
			1	0.50, 0.65, 0.70
12		SPE	0.01	0.0095, 0.0093, 0.0089
			0.1	0.092, 0.099, 0.095
			1	1, 0.99, 1

ND: Not detected

Table 5- Comparison of different extraction method on spiked barberry which were pesticide-free (remaining amount in distilled water applied in to the results)

No.	Pesticide	Extraction method	Spike Concentration (mg/kg)	Result (3 replications)	Mean recovery %	RSD %
1	Diazinon	QuEChERS	0.01	0.012, 0.009, 0.01	100.7	2.7
			0.1	0.07, 0.08, 0.099		
			1	0.99, 0.98, 1		
2		Static extraction	0.01	0.0066, 0.0010, 0.022	102.8	12.7
			0.1	0.046, 0.049, 0.048		
			1	0.84, 0.7, 0.85		
3		Changing PH	0.01	N.D, N.D, N.D	36.6	12.7
			0.1	0.012, 0.04, 0.024		
			1	0.62, 0.80, 0.74		
4		SPE	0.01	0.081, 0.075, 0.077	98	5.9
			0.1	0.039, 0.040, 0.082		
			1	0.98, 0.73, 0.89		
5	Oxydemeton-methyl	QuEChERS	0.01	0.009, 0.006, 0.008	90.1	5.0
			0.1	0.043, 0.085, 0.050		
			1	0.92, 0.99, 0.93		
6		Static extraction	0.01	N.D, 0.019, N.D	77	0.5
			0.1	0.022, 0.020, 0.019		
			1	0.80, 0.92, 0.80		
7		Changing PH	0.01	0.001, N.D, N.D	50.5	4.8
			0.1	0.030, 0.011, 0.029		
			1	0.45, 0.17, 0.11		
8		SPE	0.01	0.008, 0.009, 0.023	94.9	3.5
			0.1	0.080, 0.096, 0.049		
			1	0.92, 0.97, 0.86		
9	Phosalone	QuEChERS	0.01	0.008, 0.009, 0.009	96.7	4.9
			0.1	0.86, 0.82, 0.89		
			1	0.99, 0.98, 0.99		
10		Static extraction	0.01	0.002, N.D, N.D	82.9	1.9
			0.1	0.044, 0.040, 0.023		
			1	0.76, 0.82, 0.79		
11		Changing PH	0.01	N.D, N.D, N.D	106.5	10.9
			0.1	0.021, 0.004, 0.01		
			1	0.53, 0.64, 0.75		
12		SPE	0.01	0.0095, 0.0093, 0.0089	49.1	1.9
			0.1	0.09, 0.05, 0.05		
			1	0.92, 0.96, 0.90		

ND: Not detected
RSD: Relative standard deviation

As shown on table 6, in the second year, because of rising temperatures and reduced rainfall, which leads to flooding aphids, aphids were flooding on September, so more chemical pesticides have been used in the region. Although no pesticides were used in the under study area, Also, in addition to tracking pesticides, other toxins has been found with a very low concentration by GCMS full scan method, which has been used in the region and came as drift. such as Dimethoate, Dursban & Acetamiprid.

Table 6- The amount of selected pesticide residues tracking of the barberry Chromatography-Mass Spectrometry

Pesticide	LOQ (mg/kg)	Recovery(%)* ± RSD	Residue in First Year (mg Kg^{-1})					
			1	2	3	4	5	6
Diazinon	0.005	92.3±1.17	0.1	0.1	BDL	BDL	BDL	BDL
Oxydemeton methyl	0.01	89.88±1.1	1.37	BDL	BDL	BDL	BDL	BDL
Phozalone	0.01	79.17±0.1	BDL	BDL	BDL	0.26	BDL	BDL

*: Means of three experiments.
BDL=Below Detection Limit
RSD: Relative standard deviation
LOQ: Limits of quantitation

Pesticide	LOQ (mg/kg)	Recovery(%)* ± RSD	Residue in Second Year (mg Kg^{-1})					
			1	2	3	4	5	6
Diazinon	0.005	92.3±1.17	1.22	1.90	BDL	BDL	0.99	0.01
Oxydemeton methyl	0.01	89.88±1.1	1.45	0.50	0.87	0.011	0.05	BDL
Phozalone	0.01	79.17±0.1	BDL	0.01	0.02	0.11	BDL	BDL

*: Means of three experiments.
BDL=Below Detection Limit
RSD: Relative standard deviation
LOQ: Limits of quantitation

Discussion

In this study, four different methods of extraction and purification were compared on spiked barberry that represents the differences;

Times of extractions in SPE was about 40 minutes and for QuEChERS was about 60 minutes, although the SPE extraction has shorter time, but the results show that QuEChERS was more accurate. Calatayud-Vernich *et al.* (2016a) analyzed 58 pesticides in dead honey bees (Aphis mellifera L.) in very low density by QuEChERS. Blasco *et al.* (2011) tested different extraction procedures of 12 organophosphorus and carbamates insecticides in honey samples and the QuEChERS method recovery's was between 78 and 101% and the SPE method recovery's reported between 72 and 100%.

Changing PH and Static extraction showed the worst results. Although Rumpold & Schlueter (2013) checked 236 nutrient compositions in addition to amino acid spectra and fatty acid compositions in insects as a traditional food and have been used static extraction and found some pesticides such as dimetoate and dorsban.

Changing PH was the cheapest and quickest method but it did not have acceptable results. This study demonstrated that QuEChERS extraction rapidly extracted pesticides from Barberry with good accuracy and precision. QuEChERS could be introduced as a means of determining pesticides residual levels in Barberry. Calatayud-Vernich *et al.* (2016a) proved that the QuEChERS method was the most efficient method for the extraction of the selected pesticides in honey and honeybee. Guan *et al.* (2013) noted that QuEChERS method is suitable for extraction of pesticides in tea, which has different pretreatment conditions such as longer soaking and extraction time to be soaked. Niell *et al.* (2015) extracted pesticides thiacloprid, imidacloprid, methomyl, carbaryl, hexythiazox, azoxystrobin, pyraclostrobin, tebuconazole, and haloxyfop-methyl at 0.0001-0.01 mg/kg levels in beehive by QuEChERS method.

These findings revealed that 45 % of collected *Berberis vulgaris* were contaminated with the pesticides residues. The findings of recent study might help in extending awareness in farmers and local people about pesticides and their hazardous effects on humans.

References

Asoudeh, R. 1999. Barberry dental gel formulation and clinical effects on dental plaque. Basic doctoral dissertation, Ferdowsi University, School of Pharmacy, Mashhad, Iran.

Blasco, C., Vazquez-Roig, P., Onghena, M., Masia, A. and Pico, Y. 2011. Analysis of insecticides in honey by liquid chromatography-ion trap-mass spectrometry: comparison of different extraction procedures. Journal of Chromatography A, 1218: 4892-4901.

Calatayud-Vernich, P., Calatayud, F., Simo, E. and Pico, Y. 2016a. Efficiency of QuEChERS approach for determining 52 pesticide residues in honey and honey bees. MethodsX, 3: 452-458.

Calatayud-Vernich, P., Calatayud, F., Simo, E., Suarez-Varela, M. and Pico, Y. 2016b. Influence of pesticide use in fruit orchards during blooming on honeybee mortality in 4 experimental apiaries. Science of the Total Environment, 541: 33-41.

Dirr, A. M. 2015. Barberries–A new look at old favorites. American Nurseryman, 153: 2-4.

Guan, Y., Tang, H., Chen, D., Xu, T. and Li, L. 2013. Modified QuEChERS method for the analysis of 11 pesticide residues in tea by liquid chromatography-tandem mass spectrometry. Analytical Methods, 5: 3056-3067.

Jazayeri, Gh. 2002. Herbal treatment. Hamgam Publishing, Tehran, Iran. 80pp.

Kafi, M. and Balandari, A. 2002. Barberry processing Technology. Ferdowsi University of Mashhad, Iran. 15pp.

Moazzen, B. 1993. Barberry. Agricultural Extension and Education Publications Research Organization. Iran. 24pp.

Niell, S., Jesus, F., Perez, C., Mendoza, Y., Diaz, R., Franco, J., Cesio, V. and Heinzen, H. 2015. QuChERS adaptability for the analysis of pesticide residue in beehive products seeking the development of an agroecosystem sustainability monitor. Journal of Agricultural and Food Chemistry, 63: 4484-4492.

Peterson, P. 2003. The Common barberry: The past and present situation in Minnesota and the risk of wheat stem rust epidemics. Ph.D. dissertation, North Carolina State University, USA.

Rumpold, B. A. and Schlueter, O. K. 2013. Nutritional composition and safety aspects of edible insects, Molecular Nutrition and Food Research, 57: 802-823.

Sanchez, T. M., Flores-Rojas, K., Guerrero, J., Varo, A. and Marin, D. 2010. Measurement of pesticide residues in peppers by near-infrared reflectance spectroscopy. Pest Management Science, 66: 580-586.

Sonei, A. 2012. The use of C18 as absorbent extracted stationary phase to measure pesticide residues in saffron method using gas chromatography mass spectrometry, The Second National Seminar on food Security, Savadkooh, Iran.

Tehranifar, A. 2003. Barberry growing in Iran. ISHS Acta Horticulture, 620: XXVI International Horticultural Congress: Asian Plants with Unique Horticultural Potential, Genetic Resources, Cultural Practices, and Utilization, 193-195.

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فصلنامه تخصصی تحقیقات حشره‌شناسی
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اندازه گیری باقی‌مانده حشره‌کش‌های رایج در زرشک خوراکی به‌وسیله کروماتوگرافی گازی در ایران

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چکیده

امروزه آفت‌کش‌های مختلفی جهت کنترل آفات زرشک خوراکی در منطقه خراسان جنوبی به‌کار می‌روند. با توجه به مصرف زرشک در دو حالت خام و فرآوری شده، شناسایی و اندازه گیری میزان باقی‌مانده آفت‌کش‌های رایج دیازینون، اکسی دیمتون متیل و فوزالون در زرشک خوراکی به‌وسیله دستگاه کروماتوگرافی گازی مورد بررسی قرار گرفت. جهت به‌دست آوردن روش مناسب استخراج و خالص سازی، چهار روش معتبر از قبیل استخراج کیوچر، استخراج پایا، تغییر میزان اسیدیته و استخراج فاز ساکن مورد بررسی و آزمون قرار گرفت. جهت کاهش خطا، از استاندارد داخلی استفاده شد و نتایج حاصل از دستگاه کروماتوگرافی بوسیله منحنی کالیبراسیون به‌دست آمده از استاندارد سموم مورد اندازه گیری قرار گرفت. استخراج با حلال استونیتریل و اسکن کلی به‌وسیله دستگاه کروماتوگرافی نشان داد که اغلب زرشک ها به آفت‌کش‌ها آلوده هستند. چهار نوع روش مختلف استخراج و خالص سازی در آب مقطر و نیز زرشک‌های سالم اسپایک شده، مورد مقایسه قرار گرفتند. روش استخراج فاز ساکن بازیابی کمتری را در نمونه‌ها نشان داد. اگرچه تغییر میزان اسیدیته و روش استخراج پایا در کمترین زمان انجام گردید، اما نتایج خوبی نداشتند. آفت‌کش‌های دیگری نیز نظیر دی متوآت، دورسبان و استامی پرید به‌وسیله روش اسکن کامل دستگاه کروماتوگرافی گازی با غلظت بسیار اندک در نمونه‌ها نمایانگر شد که احتمالا به دلیل بادبردگی منطقه نمونه‌برداری را آلوده کرده است.

واژه‌های کلیدی: آفت‌کش‌ها، باقیمانده، دستگاه کروماتوگرافی گازی، زرشک خوراکی، استخراج

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