

GAS CHROMATOGRAPHIC DETERMINATION OF 23 ORGANOPHOSPHORUS PESTICIDES RESIDUE IN BOTTLE GUARD MATRIX

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ABSTRACT

Objective: To validate in-house QuEChERS method, which scrutinizes and quantify the residue levels of some most frequently used organophosphorus pesticides and to prove a complete workflow for routine multi-residue pesticide analysis in representative bottle guard matrices and study the persistence of 23 organ phosphorus pesticides.

Methods: QuEChERS (quick, easy, cheap, effective, rugged and safe) extraction method was followed. Method validation and residue screening of bottle guard samples were conducted by GC-FPD (Gas Chromatography-Flame Photometric Detector) were used to analyse the presence of pesticides whereas confirmation of pesticides was done by GC-MS (Gas Chromatographic-Mass Spectrometer).

Results: For bottle guard matrix, LOD (limit of detection) and LOQ (limit of quantification) values are lowest for phorate (0.005 mg/kg and 0.015 mg/kg) and highest for parathion-methyl (0.1 mg/kg and 0.3 mg/kg) respectively. Calibration curve, was plotted between an area of 23 pesticide standard mixture against seven different concentration levels of 0.01, 0.02, 0.05, 0.10, 0.20, 0.50, 1.00 mg/kg with regression coefficient (R^2) are in the range of 0.978-0.994. The mean recovered amount at 0.05 mg/kg spiking concentration are in the range of lowest 0.0413 mg/kg to highest 0.0467 mg/kg and relative standard deviation are in the range of lowest 2.2 to highest 8.571.

Conclusion: For ensuring exposure to contaminants, especially by dietary intake, Robust analytical methods were validated for carrying out both research and monitoring programmes and thus for defining limitations and supporting enforcement of regulations.

Keywords: Bottle guard, Pesticides, LOD, LOQ, MRL, QuEChERS

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INTRODUCTION

The use of pesticides results in the 'green revolution' during the 1960s due to which crop losses reduces, yield improved but results in adverse effects on environmental quality and human health [1-5]. Replacing persistent organochlorines pesticides currently organophosphorus (OP's) Synthetic Pyrethroids (SP's) and Herbicides are most widely used in the world as an alternative pest control [6-9]. Tetraethyl pyrophosphate (TEPP) was the first OP insecticide, which was developed in Germany during World War Two as a by-product of nerve gas development [10]. OPs are derived from phosphoric acid, which are mostly acute, toxic, degrade in the atmospheric conditions [11].

The Association of Official Analytical Chemists (AOAC) adopted a method which is the internationally recognized procedure for MRM. By gas chromatography (GC) or high-performance liquid chromatography (HPLC) with selective detectors, the analyte determination is performed. International organizations: the European Union (EU) and the Codex Alimentarius Commission of the Food and Agriculture Organization (FAO) and the World Health Organization (WHO) controlled pesticide residues in food at permissible levels by the MRLs, which are established by them [12]. The pesticides studied were chosen based on their widespread use for crop protection [13].

To overcome the drawbacks of the traditional approaches modern sample preparation procedures, like accelerated solvent extraction (ASE) [14], supercritical fluid extraction (SFE) [15], microwave-assisted extraction (MAE), [16] solid phase extraction (SPE) [17], solid phase microextraction (SPME), [18] matrix solid-phase dispersion (MSPD), [19] extraction and QuEChERS (quick, easy, cheap, effective, rugged and safe), [20] have been developed.

Analytical methods designed for multiple pesticide residues present in foodstuffs. Analytical methods capable of accurately measuring

extremely small amounts of pesticides are required. QuEChERS (Quick Easy Cheaper Effective Rugged and safe) method in 2003 for pesticide residue analysis, which gives high-quality results in a fast, easy and an inexpensive way. Analytical quality requirements like trueness, precision, sensitivity and selectivity have been met to suit the need for any particular analysis. The QuEChERS method has been validated for hundreds of pesticide residues in many types of foods, and has become Association of Analytical Communities (AOAC) Official Method 2007.01 [21-24].

The present study was undertaken with the objective to validate in-house QuEChERS method which scrutinizes and quantify the residue levels of some most frequently used organophosphorus pesticides on bottle guard. The objective of this research work is to prove a complete workflow solution that can be implemented for routine multi-residue pesticide analysis in representative bottle guard matrices and study the persistence of organophosphorus pesticide in bottle guard.

Gas chromatography (GC equipped Flame Photometric Detector (FPD) or mass spectrometer (MS) has been frequently applied for the analysis of pesticides in food for many years [25-28]. In this paper, we report the laboratory validation and uncertainty measurement of some organophosphorus pesticides in spinach matrix with good selectivity, sensitivity, and cost-effectiveness.

MATERIALS AND METHODS

Chemicals and reagents

Pesticide reference standards (Sigma-Aldrich), ethyl acetate (HPLC grade), n-hexane (HPLC grade), anhy. Na₂SO₄ (Merck), anhy. MgSO₄ (Merck), primary secondary amine (PSA), (Agilent Technologies), and graphitized carbon black sorbent supelco (Bellefonte, PA, USA).

Extraction and standard stock preparation

Bottle guard sample was purchased from vegetable mandi of Satna (M. P.) and transported to the laboratory. QuEChERS (quick, easy, cheap, effective, rugged and safe) method [29] extraction was done. Fifteen-gram sample was weighted homogenized and chopped into a centrifuge tube and ethyl acetate (30 ml) was added for extraction and shaken for 1 min. Na₂SO₄ (10g) was added and shaken for 30 min by rotospin and centrifuged at about 5 min. Cleaning step was followed according to Lehotay (2007) [30]. Cleaning of 6 ml upper layer extract was done adding 0.9 g anhydrous MgSO₄, 0.25 g PSA and 0.25 g activated charcoal and shaken for 1 min into a 15 ml centrifuge tube. The tube was centrifuged and supernatant 4 ml was dried and finally make up to 1 ml for injection in GC-FPD (Gas Chromatography-Flame Photometric Detector) and GC-MS (Gas Chromatographic-Mass Spectrometer).

The stock solution was prepared by Certified Reference Materials (CRM) of individual pesticide weighed approx 2 mg, in 10 ml volumetric flask, which was dissolved in few drops of HPLC grade acetone and filled up to the mark of standard volumetric flask with HPLC grade hexane. Standard stock solution and working standards were stored in deep freezer at -20 °C. 23 organophosphorus commonly used viz anilophos, chlorfenvinfos, chlorpyrifos, chlorpyrifos-methyl, dichlorvos, ethion, malathion, parathion-methyl, monocrotophos, phorate, profenofos, quinolphos, trizophos, fenitrothion, phosalone, paraxon-methyl, fenamiphos, edfinphos, dimetoate, diazinon, fenthion, parathion and phosphamidon, standard solution mixture was prepared at seven different concentration levels of 0.01, 0.02, 0.05, 0.10, 0.20, 0.50, 1.00 mg/kg. All pesticides are sensitive for FPD detector. Seven different concentration levels were prepared for calibration and recovery tests. Fig. 1, shows the standard mix. Chromatogram of 23 organophosphorus pesticides at 0.5 ppm concentration.

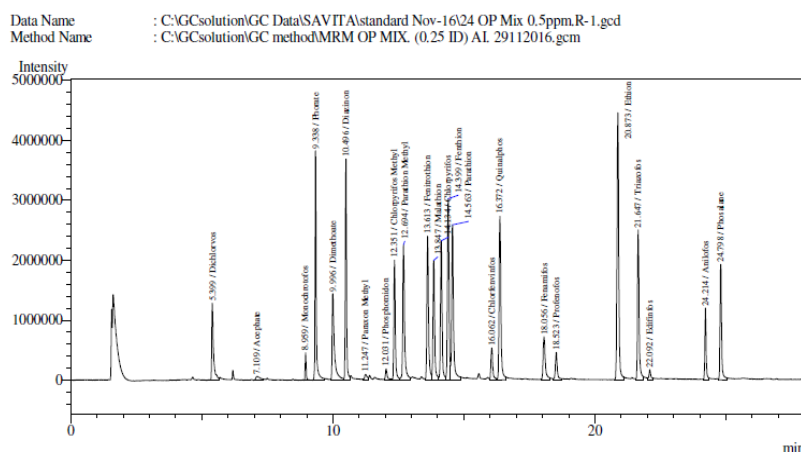


Fig.1: Standard mix chromatogram of 23 organ phosphorus pesticides at 0.5 ppm concentration

Instrument conditions

GC-FPD

Shimadzu makes GC-FPD (GC-QP 2010 model) with the AOC-20S auto sampler. DB-5MS fused silica capillary column was used for analysis. Oven temperature programming of 100 °C as initial temperature for 2 min followed by a ramp rate of 25 °C/min up to 200 °C for 5 min., 4 °C/min up to 230 °C for 2 min and 20 °C/min up to a final temperature of 280 °C with a hold time of 5 min. The injector was set at 250 °C whereas detector at 290 °C. Sampling rate 40 msec, injection volume 1.0 micro litre and 0.5 min equilibrium time. Split Ratio 10:1 was applied. Helium was used as makeup gas and also as carrier gas at a flow rate of 1.23 ml/min. H₂ flow rate 85 ml/min with air flow 110.0 ml/min.

GC-MS

GC Model 7890A (Agilent Technologies) with a mass detector (EI mode). DB-5MS fused silica capillary column was used. Oven programming of initial temperature 50 °C for 2 min followed by a ramp rate of 8 °C/min up to a temperature of 280 °C with a hold time of 10 min, total run time 40.75 min. The injector was set at 280 °C. The interface, ion source and quadrupole temperatures were set at 280 °C and 230 °C and 150 °C respectively. Electron impact (70eV) ionization mode with solvent delay time 3 min. The instrument was operated in scan mode. Sampling rate 40 msec, injection volume 1.0 micro litre and 0.5 min equilibrium time. Concentrated extract 1 microlitre was injected in splitless mode. Helium was used as makeup gas and also as carrier gas at a flow rate of 1 ml/min.

RESULTS AND DISCUSSION

Method validation and residue screening of bottle guard samples were conducted by GC-FPD for the presence of pesticides whereas

concentrated samples were analyzed by GC-MS in full scan mode for further confirmation of pesticides. Matching of retention time (RT) and mass spectrum (MS) data of the sample peak with that of the standard, confirms identification of the pesticides present in the sample. Final quantification was carried out on GC-FPD. Laboratory method validation of the method was carried out on bottle guard matrix for 23 organ phosphorus pesticides.

Pesticides taken for study

23 organ phosphorus pesticides which are commonly used in India viz anilophos, chlorfenvinfos, chlorpyrifos, chlorpyrifos-methyl, dichlorvos, ethion, malathion, parathion-methyl, monocrotophos, phorate, profenofos, quinolphos, trizophos, fenitrothion, phosalone, paraxon-methyl, fenamiphos, edfinphos, dimetoate, diazinon, fenthion, parathion and phosphamidon are considered for the study. The preliminary screening was done to find out the best chromatographic techniques which can be used in terms of peak shape, response and LOD/10Q. All 23 pesticides gave good response for FPD detector which can be used for quantification and confirmation by GC-MS.

Method development and validation

For ensuring a robust method, method development and validation parameters, are-Recovery, Selectivity (Specificity), Calibration, Repeatability, Reproducibility, Limit of Detection (LOD), Limit of Quantitation (LOQ). System performance experiment was performed by injecting six consecutive injections at the beginning [31].

Limit of detection and limit of quantification

LOD and LOQ will be measured by using EPA method as it is simple, easy and practical to implement [32, 33]. Limit of detection (LOD) and Limit of quantification (LOQ) was calculated as the lowest

concentration a pesticide in a selected matrix which gave the signal to noise (S/N) ratio of approximately equals to 3 and 10 respectively. To measure the LOD, peak to peak noise of blank matrix (bottle guard) at or around the retention time of individual pesticides, the chromatogram of the standard mixture is noted and averaged for three replicates.

The concentration of the individual pesticide is calculated (in µg/g) from the matrix spiked chromatogram which could produce the signal equal to three times of blank matrix. LOQ is calculated by multiplying the LOD value by factor 3 round of two decimal place value. Measured method LOD, LOQ and the relevant legislative limits (MRLs) and ADI [34] are listed in table 1. With the present method, all 23 organophosphorus pesticides for the study presented LOD and LOQ were in the range of below MRL. From table 1, it was observed that for bottle guard matrix, LOD value is lowest for phorate (0.005 mg/kg) and highest for parathion-methyl (0.1 mg/kg) whereas LOQ

value is lowest for phorate (0.015 mg/kg) and highest for parathion-methyl (0.3 mg/kg). MRL values of phorate is lowest (0.05 mg/kg) and highest for malathion (3.0 mg/kg) whereas ADI values for diazenon, malathion, phasalone are highest (0.02 mg/kg) and lowest for fenthion, monocrotophos (0.0005 mg/kg). MRL values for some pesticides like chlorpyrifos-methyl, quinalphos, fenamiphos, profenophos, trizophos, edfinphos, anilophos are not available whereas ADI values are not available for monocrotophos, paraxon-methyl, parathion, chlorfenvinphos, quinalphos, fenamiphos, profenophos, ethion, trizophos edifenphos and anilophos.

From LOD and LOQ values it is observed that LOQ is lower than the MRL value as given in PFA (Prevention of Food Adulteration) agency. The method is reliable only if the LOQ value should be always lesser than MRL value of pesticide. Therefore the method can be applicable for the similar type of pesticides and matrix taken for study.

Table 1: Shows class, retention time (R. T), limit of detection (LOD), limit of quantification (LOQ), maximum residues limits (MRL) and acceptable daily intake (ADI) of Organophosphorous pesticide spiked in bottle guard matrix by GC-FPD

S. No.	Organophosphorous pesticides	R. T	LOD (mg/kg)	LOQ (mg/kg)	MRL (mg/kg)	ADI (mg/kg/day)
1	Dichlorvos	5.49	0.007	0.021	0.15	0.004
2	Monocrotophos	9.16	0.03	0.09	0.2	NA
3	Phorate	9.44	0.005	0.015	0.05	0.0005
4	Dimetoate	9.86	0.04	0.12	2	0.002
5	Diazinon	10.43	0.06	0.18	0.50	0.02
6	Paraxon-methyl	11.22	0.05	0.15	0.2	NA
7	Phosphomidon	12.03	0.05	0.15	0.2	0.004
8	Chlorpyrifos-methyl	12.44	0.02	0.06	NA	0.01
9	Parathion methyl	12.67	0.1	0.3	1.00	0.003
10	Fenitrothion	13.61	0.03	0.09	0.30	0.005
11	Malathion	13.88	0.01	0.03	3.00	0.02
12	Chlorpyrifos	14.25	0.02	0.06	0.20	0.01
13	Fenthion	14.25	0.03	0.09	1	0.0005
14	Parathion	14.43	0.01	0.03	0.5	NA
15	Chlorfenvinphos	15.95	0.04	0.12	0.5	NA
16	Quinalphos	16.42	0.01	0.03	NA	NA
17	Fenamiphos	17.86	0.02	0.06	NA	NA
18	Profenofos	18.58	0.03	0.09	NA	NA
19	Ethion	20.93	0.01	0.03	1.00	NA
20	Trizophos	21.60	0.02	0.06	NA	NA
21	Edfinphos	21.99	0.01	0.03	NA	NA
22	Anilophos	24.15	0.05	0.15	NA	NA
23	Phosalone	25.74	0.02	0.06	1	0.02

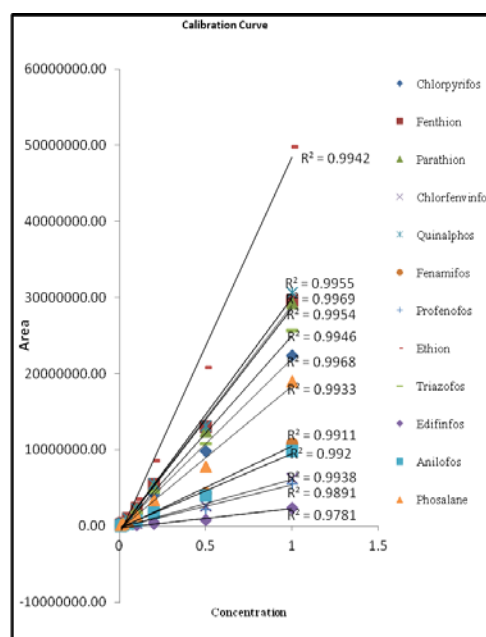
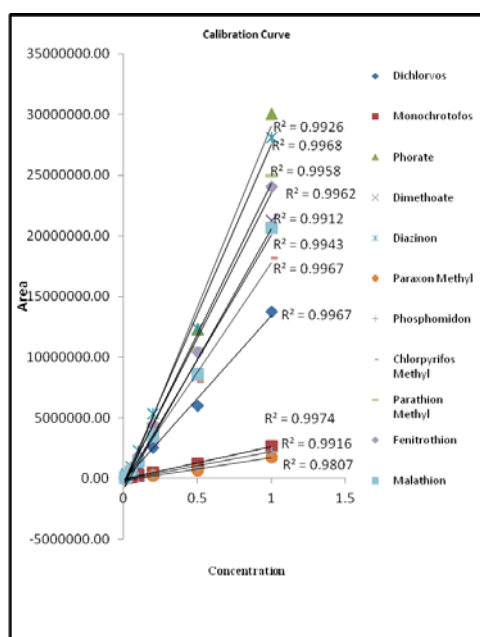


Fig. 2a-b: Calibration curve of 23 organophosphorus pesticides mixture at 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1 ppm conc level

Linearity

The calibration curve of GC-FPD instrument are created at seven levels i. e (matrix match) prepared by spiking 23 pesticides standard mixture solution at different concentration levels and injected in triplicate. Fig. 1 and 2 shows calibration curve, plotted between an area of 23 pesticide standard mixture against seven different concentration levels of 0.01, 0.02, 0.05, 0.10, 0.20, 0.50, 1.00 mg/kg with regression coefficient (R^2) are in the range of 0.978-0.994. edifenphos with lowest (R^2)0.978 and monocrotophos with highest (R^2) 0.9974, rest all pesticides coefficient of regression lies between these value. Fig. 1 and 2 shows calibration curve of 23 organophosphorus pesticides mixture of concentration 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1.0 mg/kg in GC-FPD with coefficient of regression (R^2). Before starting any analysis work in the gas chromatography, it should be sure that the instrument response should be checked. As the coefficient of regression values lies near to one, shows instrument was ready to work.

Recovery

Method trueness was assessed by recovery studies using bottle guard matrix spiked at 0.05 ppm concentration level and injected in three individually prepared replicates. Spiking of samples occurred prior to sample preparation, recovery, standard deviation and relative standard deviation (% RSD) are calculated in table 2 which shows recovery, standard deviation and relative standard deviation for organophosphorus pesticides. From table 1, it is observed that mean recovered amount at 0.05 mg/kg spike concentration of 23 pesticides mixture of organophosphorus in bottle guard matrix are in the range of 0.0413 mg/kg to 0.0467 mg/kg and it is observed that highest recovery percent is for chlorpyrifos (92.67 %) and lowest recovery percent is for ethion (82.67%) rest pesticides recovery percent in between the range whereas relative standard deviation are in the range of 2.2 to 8.571 values lies below codex permissible limit of 15.

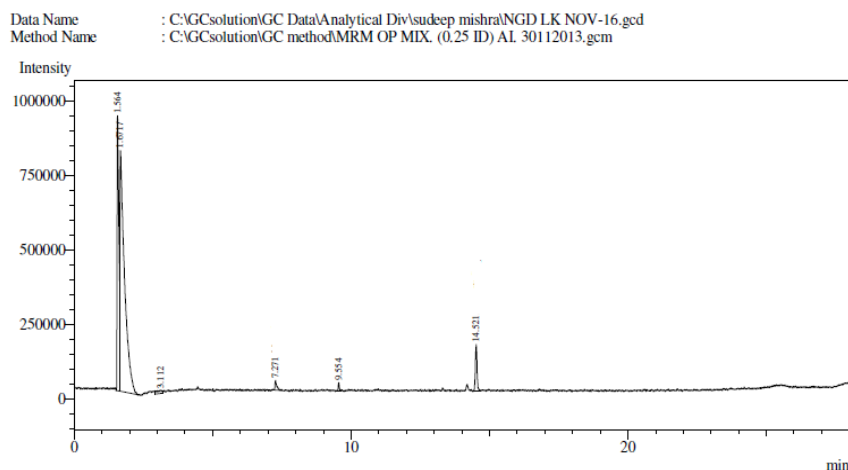


Fig. 3: GC-FPD chromatogram representing a matrix of okra sample

Table 2: Shows recovery, mean recovery, standard deviation (S. D) and relative standard deviation (RSD) of organophosphorus pesticides from spiked bottle guard matrix at 0.05 ppm

S. No.	Pesticide	RT	R1			R2			R3			SD	R. S. D
			Spiking conc.	Amount recovered	Recovery percent	Amount recovered	Recovery percent	Amount recovered	Recovery percent				
1.	Dichlorvos	5.49	0.05	0.043	86	0.045	90	0.04	80	0.0427	85.33	0.0025	5.859
2.	Monocrotophos	9.16	0.05	0.043	86	0.044	88	0.045	90	0.0440	88.00	0.001	2.273
3.	Phorate	9.44	0.05	0.045	90	0.042	84	0.043	86	0.0433	86.67	0.0015	3.462
4.	Dimetoate	9.86	0.05	0.047	94	0.046	92	0.043	86	0.0453	90.67	0.0021	4.632
5.	Diazinon	10.43	0.05	0.043	86	0.048	96	0.046	92	0.0457	91.33	0.0025	5.474
6.	Paraxon-methyl	11.22	0.05	0.043	86	0.04	80	0.043	86	0.0420	84.00	0.0017	4.048
7.	Phosphomidon	12.03	0.05	0.044	88	0.046	92	0.045	90	0.0450	90.00	0.001	2.222
8.	Fenthion	12.25	0.05	0.045	90	0.044	88	0.043	86	0.0440	88.00	0.001	2.273
9.	Chlorpyrifos-methyl	12.44	0.05	0.048	96	0.046	92	0.045	90	0.0463	92.67	0.0015	3.237
10.	Parathion methyl	12.67	0.05	0.04	80	0.046	92	0.043	86	0.0430	86.00	0.003	6.977
11.	Fenitrothion	13.61	0.05	0.04	80	0.047	94	0.043	86	0.0433	86.67	0.0035	8.077
12.	Malathion	13.88	0.05	0.041	82	0.046	92	0.048	96	0.0450	90.00	0.0036	8.000
13.	Chlorpyrifos	14.25	0.05	0.048	96	0.045	90	0.047	94	0.0467	93.33	0.0015	3.214
14.	Parathion	14.43	0.05	0.043	86	0.048	96	0.046	92	0.0457	91.33	0.0025	5.474
15.	Chlorfenvinfos	15.95	0.05	0.045	90	0.046	92	0.041	82	0.0440	88.00	0.0026	5.909
16.	Quinolphos	16.42	0.05	0.043	86	0.046	92	0.046	92	0.0450	90.00	0.0017	3.778
17.	Fenamiphos	17.86	0.05	0.043	86	0.046	92	0.048	96	0.0457	91.33	0.0025	5.474
18.	Profenofos	18.58	0.05	0.046	92	0.042	84	0.044	88	0.0440	88.00	0.002	4.545
19.	Ethion	20.93	0.05	0.04	80	0.043	86	0.041	82	0.0413	82.67	0.0015	3.629
20.	Trizophos	21.6	0.05	0.043	86	0.04	80	0.046	92	0.0430	86.00	0.003	6.977
21.	Edfinphos	21.99	0.05	0.04	80	0.046	92	0.047	94	0.0443	88.67	0.0038	8.571
22.	Anilophos	24.15	0.05	0.04	80	0.044	88	0.043	86	0.0423	84.67	0.0021	4.961
23.	Phosalone	25.74	0.05	0.046	92	0.043	86	0.047	94	0.0453	90.67	0.0021	4.632

According to SANCO, recovery values should come within an acceptable range between 70–120%. It always happens that matrix effect was predominant for some pesticides.

To overcome variable matrix effect, the quantification was done using matrix-matched standards prepared in matrix blank. Recovery table shows that all 23 pesticides give lowest approx 82 to highest approx. 92 percent recovery which is according to SANCO guideline. RSD values are also below 10 as per SANCO.

Therefore method can efficiently be used for analysis of similar type of pesticides and matrix taken for study

Selectivity/Sensitivity

NIST library of Gas chromatography-mass spectrometry was used to confirm the presence of pesticides taken for study. Spectra matched with reference spectra of NIST. Presence of pesticides was confirmed by their fragmentation pattern matches with reference spectra of NIST. Some of the reference spectra of pesticide of the group like chlorpyrifos, profenofos are given in fig. 2 and fig. 3 respectively. Table 3 shows mass fragmentation pattern of each pesticide for confirmation in GC-MS. Matrix blank sample of bottle guard (fig. 3), was inspected in GC-FPD for the presence of interfering peaks in close to target retention times observed for all pesticides taken for study.

Table 3: Fragmentation pattern of 23 organophosphorus pesticides taken for study

S. No.	Organophosphorus pesticides	Qualifier ions (m/z)			S. No.	Organophosphorus pesticides	Qualifier ions (m/z)		
1.	Anilofos	226	125	-	2.	Trizophos	257	161	-
3.	Chlorfenvinfos	323	269	267	4.	Fenitrothion	277	260	109
5.	Chlorpyrifos	314	286	197	6.	Phosalone	367	182	-
7.	Chlorpyrifos-methyl	286	125	-	8.	Paraxon-methyl	125	109	263
9.	Dichlorvos	220	109	185	10.	Fenamifos	303	154	80
11.	Ethion	231	153	-	12.	Edfinfos	310	173	-
13.	Malathion	173	158	-	14.	Dimetoate	125	87	-
15.	Parathion methyl	263	233	125	16.	Diazinone	304	179	152
17.	Monocrotophos	192	164	127	18.	Fenthion	278	125	93
19.	Phorate	260	231	75	20.	Parathion	194	109	67
21.	Profenofos	339	139	97	22.	Phosphomidon	264	127	72
23.	Quinolphos	157	146	118					

Repeatability and reproducibility

The method was analysed for recovery studies which were repeated for three times are R1, R2 and R3. Their Mean (M), Standard Deviation (SD) and Relative Standard Deviation (RSD) of each pesticide were calculated as shown in table 2. Similarly from table 4. three repeatable injections for 23 organophosphorus pesticide at seven concentration level i. e. 0.01, 0.02, 0.1, 0.2, 0.5, 1 ppm shows that area of three injections of each pesticides mixture are repeatable at all concentration with relative standard deviation lies from 0.4 for parathion-methyl and chlorpyrifos to 4.2 for paraxon methyl and anilofos at 0.2 ppm concentration. It is observed that all the three injections are repeatable at different conc. with Relative Standard Deviations lies ≤ 5 . Instrument injection precision was tested for both retention time and peak area for all target

compounds by subsequent injections (n=3) of low concentration level (0.01) standard solution mixture for GC-FPD. Instrument injection precision for retention time was below 0.5% for all compounds. According to SANCO requirements, $<20\%$ was set as acceptance criteria for the target compounds and matrices. Therefore the method adopted for analysis of pesticide residue is reliable for a similar type of pesticides and matrices.

As the method is well suitable for the qualitative and quantitative determination of pesticides in bottle guard matrix. A similar type of result can also be seen in bottle guard like other matrices. Many studies have been carried out for monitoring pesticides in various matrices. A study conducted in tomato, banana and wheat matrices, for the analysis of 25 pesticides of various groups, by a single method, single run and single ECD instrument [35-37].

Table 4: Shows repeatability, standard deviation (SD), relative standard deviation (RSD) of organophosphorus pesticides at 0.5 ppm concentration

Pesticide OP Mix	RT	Area1	Area2	Area3	Average area	SD	RSD
Conc-0.5 ppm							
Dichlorvos	5.40	6011349.3	6006880.1	6040900.1	6019709.8	18486.9	0.3
Monochrotofos	8.96	1182863.4	1180992.6	1183551.6	1182469.2	1324.3	0.1
Phorate	9.34	12260776.2	12268967.9	12275272.0	12268338.7	7268.4	0.1
Dimethoate	10.00	8435229.7	8410649.8	8578248.7	8474709.4	90506.0	1.1
Diazinon	10.50	12243666.7	12430952.5	12368525.2	12347714.8	95361.4	0.8
Paraxon Methyl	11.24	634866.8	625034.9	638437.3	632779.7	6940.7	1.1
Phosphomidon	12.03	852218.5	889402.3	938065.1	893228.6	43051.0	4.8
Chlorpyrifos Methyl	12.35	7878194.2	7953549.2	8064050.8	7965264.7	93480.5	1.2
Parathion Methyl	12.69	10609289.7	10686466.0	10833056.1	10709603.9	113663.4	1.1
Fenitrothion	13.62	10343503.5	10446194.7	10544864.9	10444854.4	100687.4	1.0
Malathion	13.85	8561172.8	8607688.2	8702414.5	8623758.5	71979.1	0.8
Chlorpyrifos	14.14	9724404.9	9870959.6	9910863.7	9835409.4	98181.4	1.0
Fenthion	14.40	12873700.5	13070325.3	13061142.0	13001722.6	110965.4	0.9
Parathion	14.57	12416931.0	12569078.4	12538812.3	12508273.9	80539.8	0.6
Chlorfenvinfos	16.06	2558500.4	2576435.6	2657833.8	2597589.9	52937.8	2.0
Quinalphos	16.37	13035836.8	13188248.6	13098370.6	13107485.3	76613.6	0.6
Fenamifos	18.06	4304599.7	4431810.2	4259665.8	4332025.2	89289.1	2.1
Profenofos	18.52	2117843.4	2156272.4	2239560.6	2171225.5	62221.1	2.9
Ethion	20.87	20771770.0	20905654.9	20842785.3	20840070.1	66983.7	0.3
Triazofos	21.65	10775845.0	10852581.4	10734936.7	10787787.7	59724.7	0.6
Edifinfos	22.09	777446.1	766196.0	833851.3	792497.8	36252.2	4.6
Anilofos	24.21	3877620.4	3979122.0	4148026.1	4001589.5	136595.8	3.4
Phosalane	24.80	7766225.6	7752598.1	7973108.7	7830644.1	123565.9	1.6

CONCLUSION

Determination of pesticide residues in vegetables is necessary for ensuring human exposure to contaminants, especially by dietary intake, does not exceed acceptable levels for health. Robust analytical methods were validated for carrying out both research and monitoring programmes and thus for defining limitations and supporting enforcement of regulations. Reproducible analytical methods are required to allow the effective separation, selective identification, and accurate quantification of pesticide analytes at low levels in food-stuffs including foods of plant and animal origin.

The qualitative and quantitative determination of pesticide residue in vegetables provide following informations.

Vegetables are commonly consumed throughout the region, therefore it is important to know the toxicity pertains in it. Food and Agriculture Organization of the World Health Organization (FAO/WHO) and the European Union (EU), requires an adequate methodology for enforcement of maximum residue limits (MRL) and acceptable daily intake (ADI) values.

The advantage of this approach would be rapidity and ease of analysis, higher recoveries, good sensitivity, low limit of detection and less reagent requirement. Commonly used pesticides such as organochlorine, organophosphorus, synthetic pyrethroids and herbicides will be monitored in matrices. Different extraction techniques will be followed by a selection of most precise techniques with higher recovery percentage.

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Author name Sudeep Mishra is a Ph. D. student under Dr. Smt. Neelam Richhariya. The work relates to Ph. D work of author under the guidance of Dr Neelam Richhariya.

CONFLICT OF INTERESTS

Declared none

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