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A Fast, Sensitive and Validated Analytical Method for Quantitation of Imidacloprid Pesticide Residual in *Mangifera indica* Matrix by LC-Tandem Mass Spectrometry (LC-ESI-MS/MS)

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An accurate and precise analytical method was performed for the quantitation of imidacloprid pesticide residue in the matrix of *Mangifera indica* (alphonso), specific species of Indian mango of Valsad District, Gujarat. The validation of the analytical technique was carried out using UPLC-ESI-MS/MS and was done in accordance with the regulatory recommendations. The projected technique is based on QuEChERS Method with a mixture of diluents used followed by clean up with the PSA. Three concentration levels (0.50, 2.55 and 5.10 µg/kg) were used to validate the approach and the outcomes ranged between 70 to 120%, with relative standard deviations (% RSD) of ≤ 20%. The detection and quantification limits were derived at the concentration of 0.010 and 0.50 µg/kg, respectively with a significant %ME of 9.90%. The quantitation of imidacloprid pesticide residue in the mango matrix can be done at a lower quantification level by UPLC-ESI-MS/MS.

Keywords: Imidacloprid, *Mangifera indica*, UPLC-ESI-MS/MS, Matrix matched calibration, Residual plot.

INTRODUCTION

Pesticides are used extensively in the Indian agriculture field. Pesticides can kill or control pests as well as bacteria, fungi, insects, weeds and rodents if it is used in a proper way and its effective application. In the current period, pesticide usage is giving more production and raising the economy in India [1]. Besides that scenario, pesticides have negative consequences on both human health and the environment [2]. The primary benefits are the consequences of the pesticide effects like the indirect gains expected from their use. Nowadays, the accesses amount of pesticides are using to improve crop yields and improve business prospectus [3].

Mango is a highly consumable and common tropical fruit of India. In year 2018-2019, India has exported 46510.27 MT mangoes in the international market and made a turnover of US $ 60.26 million [4]. International standards defined by the Joint Food and Agriculture Organization of the United Nations and the World Health Organization (FAO/WHO) Codex Alimentarius Commission necessitate food safety activities and monitoring. As a result, despite efforts under the auspices of the World Trade Organization (WTO) and the Codex Alimentarius to unify maximum residue levels worldwide, MRLs (Maximum Residual Levels) still differ from one geographical region to the next. MRLs for a certain animal product may vary by nation, based on local food safety regulatory bodies and medicine consumption habits [5]. After the Registration Committee approves a pesticide, the Food Safety and Standard Authority of India (FSSAI), which is part of the Ministry of Health and Family Welfare, uses GAP data to determine the MRL, taking into account dietary exposure and risk assessment [6]. The collection and sample processing in pesticide residue analysis of food and soil are becoming more critical for achieving precise and reliable results. Examine the quality of their sample processing approach and the frequently overlooked but critical element of sample collecting and processing for pesticide residue analysis [7].

In this research work, we focussed on the determination of pesticide residual level of imidacloprid in the alphonso mangoes (*Mangifera indica*) of Valsad District, India. The analytical technique for determining specified pesticide residues was developed and validated with the help of sophisticated techniques of chromatograpy and tandem mass-spectrometry [8]. The positive growth has been observed highest in Andaman

and Nicobar Islands, Jammu & Kashmir and Tripura states of India. Maharashtra, Uttar Pradesh, Haryana, Punjab and Andhra Pradesh are the states that accounted for 70% of total pesticide consumption [9].

The sample extraction procedure quick, easy, cheap, effective, rugged and safe (QuEChERS) was optimized and validated for the residual analysis was performed for pesticide residual analysis and organophosphate pesticides in mango results were achieved within the acceptance range [10]. The optimized methodology is based on liquid-liquid extraction by using acetonitrile and primary secondary amine. The developed and validated method provides the quantitation of pesticides accurately onto LC-MS [11]. The QuEChERS extraction technique was used to prepare the sample, which was then analyzed using GC-MS/MS (gas chromatography tandem with mass spectroscopy) and UPLC-ESI-MS/MS for the measurement of several pesticides in various stages of alphonso mangoes (*Mangifera indica*). Purification sorbents and various solvent compositions were tuned for better results using the analytical procedures of GC-MS/MS and UPLC-ESI-MS/MS [12]. The multi-residue technique, based on the improved quick easy cheap effective rugged and safe (QuEChERS) sample preparation process and LC-ESI-MS/MS, was used to analyze 14 pesticides in 145 vegetable samples from an intensive agricultural plain in Italy, tilled using integrated pest management practises and destined for the Italian fresh-cut commercial market [13]. In a short period, analytical methods have been developed and optimized to determine imidacloprid residual levels in various matrices [14-21]. Modern measurable methods for identifying imidacloprid are predominantly based on high-performance liquid chromatography (HPLC) [22-26] and liquid chromatography with tandem mass spectrometry (UPLC-ESI-MS/MS) [27-31].

Dimethyl thiophosphate, an organophosphate metabolite, was found in 49.1% of the samples and measured at a median value of 344 ng/g. These findings show that prenatal exposure to pesticides such as organophosphates, pyrethroids and carbamates is high [32]. Then, the optimized method was validated by following the SANCO required parameters for the respective matrix [33].

EXPERIMENTAL

The LC with tandem MS (UPLC-ESI-MS/MS) was used to optimize and validate the analytical method using LC separation of mixtures with multiple components and mass spectrometry (MS) to provide the structural identity of the individual component with high molecular specificity and lowest detection sensitivity. Precursor and substance ion scanning, as well as compound optimization using a mass spectrometer, were used to tune the compound. The HPLC column was chosen to optimize chemical retention, high peak response and increased chromatography optimization efficiency. By selecting an appropriate solvent mixture for the mobile phase composition, the repeatability of sensitivity and consistent ionization of the repeated response for the target analyte were maximized.

A pesticide reference material with a purity of 99.90% was purchased from Sigma-Aldrich, USA. The solvent acetonitrile (LC-MS grade) was bought from J.T. Baker, USA. The PSA (primary secondary amine, Source: Agilent Technologies, USA), magnesium sulfate anhydrous (AR grade) from TCI, USA and sodium chloride (AR grade) from FINAR, India were acquired. A water purification system was used to purify the Milli-Q water of Merck, USA. Before UPLC-ESI-MS/MS Analysis, the polyvinylidene fluoride (PVDF) filters (pore size: 0.45 µm) were used for filtered (Source: Millipore Co.).

Liquid chromatography-mass spectrometry- UPLC-ESI-MS/MS (Model: API 6500 Q TRAP, Make: AB Sciex) coupled with high-performance liquid chromatography (HPLC) (Model: Nexera X2, Shimadzu) was used. The analytical balance (Model: GR-202, Adair and Dutt), Refrigerator (Model: Eon, Godrej), Micropipette (Eppendorf) and Centrifuge (Model: Sorvall Legend X1/X1R, Thermo Fisher Scientific) were used.

Standard solution: Pure imidacloprid (10.25 mg) as reference standard was accurately weighed and transferred to a 10 mL capacity of volumetric flask. The volume was then topped up with acetonitrile and well mixed. Standard working solution with imidacloprid concentration of 1023975.00 µg/L was achieved.

QuEChERS extraction procedure: The homogenized mango sample (10.0 g) was added to a 10.0 mL acetonitrile and rapidly agitated for 5 min using a vortex mixer, according to the QuEChERS technique. The samples were shaken briskly for 2 min after adding 3 g NaCl, then centrifuged for 5 min at 3800 rpm at 4 ºC. A volume 1.0 mL supernatant aliquot was transferred to 2.0 mL centrifuge tubes containing 150 mg $MgSO_4$ and 50 mg PSA. The tubes were then centrifuged for 5 min at 10,000 rpm at 4 ºC. Finally, a volume 0.5 mL of purified supernatant was filtered through a syringr 0.45 µm filter membrane and 10 times diluted with diluent (acetonitrile: milli-Q water (70:30, v/v) and then, injected onto the UPLC-ESI-MS/MS for analysis [34-37].

Chromatographic (LC) conditions: Using the analytical procedure parameter of LC conditions, different aliquots of standard and sample solutions were conducted. The analytical UPLC is a system that includes binary pumps, an autosampler injector (50 µL loop) and a 10 ºC cooler temperature. In the validation of analytical techniques, a column (Make: Atlantis® T3 [150 mm × 4.6 mm (i.d.), 3.0 µm particle size]) with an elution mode mobile phase (mobile phase:acetonitrile (70): 10 mM ammonium bicarbonate Milli-Q water (30), v/v) and a flow rate of 0.80 mL/min was shown to be adequate. The experiment was carried out at 40 ºC in a column oven with a 5 µL injection volume of the appropriate sample solutions. The analysis took 4.0 min to complete and the retention durations for the analytes were around 2.44 min.

Mass spectrometry conditions: The instrument (Model: API 6500 Q-TRAP triple quadrupole mass spectrometer, AB-Sciex) in positive electrospray ionization mode was used to design and evaluate the analytical technique. The 1/X weighted linear regression was used to calculate the concentrations in the matrix. Analyst® software version 1.6.3 was used for sample collecting and quantification. In order to achieve optimal sensitivity, mass transitions were calculated by injecting standards as stated in Table-1. Curtain gas (CUR) 35 psi, entrance potential

TABLE-1
OPTIMIZED OPERATIONAL CONDITIONS FOR UPLC-ESI-MS/MS OF TARGET ANALYTES

Parameter	Imidacloprid	
MRM transitions	256.0/209.0	256.0/175.0
Collision energy (eV)	21	25
Collision cell exit potential (V)	14	12
Declustering potential (DP) V	60	

(EP) 10 V, ion spray voltage 5500 V and temperature 400 ºC, GS1: 50 psi and GS2: 60 psi pressure were the optimum MS variables.

Analytical method parameters: Serial dilutions of the standard solution combination were used to create calibration curves. According to the suggested extraction approach and analytical method parameters, the reference standard working solutions were processed in the blank extract of alphonso mango. The reference standard was then evaluated using UPLC-ESI-MS/MS, with the peak area shown *versus* concentrations (µg/L). Table-2 shows the correlation coefficient (r), slope (b) and intercept (a) values. The lowest concentration at which adequate recovery with acceptable (% RSD) was attained was reported as the LOQ.

TABLE-2
DEMONSTRATION OF LINEAR CALIBRATION FOR DESIGNING OF A RESIDUAL PLOT

Regression equation:	y = 4.91e + 005x + 6.79e + 003
Intercept:	6790
Slope:	491000

ID	Conc. (µg/L)	Area (y_i)	Estimated peak area (y_{yi})	Residual (d_i)
L1	0.025	21289	19065	2224
L2	0.050	27780	31340	-3560
L3	0.100	53685	55890	-2205
L4	0.210	108458	109900	-1442
L5	0.410	212663	208100	4563
L6	0.820	411802	409410	2392
L7	1.640	811120	812030	-910

At LOQ level test concentrations, precision and accuracy were established at 0.5 µg/kg and 10 times LOQ level at 5.10 µg/kg along with the control. The five different replications were prepared individually for LOQ levels and 10 times LOQ levels. For the preparation of fortified samples, the reference standard was fortified in the blank matrix of alphonso mango at three different levels (0.50, 2.55 and 5.10 µg/kg) and the uniformity at each level, fortified samples were obtained from three layers. The samples were extracted using the suggested extraction method, then diluted further before being filtered through a 0.45 m PVF syringe filter and analyzed using UPLC-ESI-MS/MS. The mean of the analyte concentration, standard deviation (SD) and percent relative standard deviation (RSD) were computed and reported for each duplicate.

RESULTS AND DISCUSSION

By injecting solvent (acetonitrile), diluent, blank matrix, reference standard solution and standard fortified sample solution, the specificity of the technique for determining imidacloprid content in fortified samples of mango matrix (*Mangifera indica*) was investigated. Since there was no interference between the analyte, solvent and diluent peaks for imidacloprid. The technique was tailored to the required analyte of a certain mass. The matrix effect (ME) is estimated as:

$$ME\ (\%) = \left(\frac{\text{Peak response of analyte matrix}}{\text{Peak response of analyte in solvent}} - 1 \right) \times 100$$

The matrix effect can be negative or positive and classified as follows: soft matrix effect (–20% < ME < 20%) [38,39]. The derived ME (%) was 9.90 as shown in Figs. 1 and 2. The linearity for precision, accuracy and fortified samples (at the lower, middle and higher dose levels) was established in the matrix by injecting seven different concentrations of working standard solutions (linearity range: 0.025-1.64 µg/L) and the peak area was plotted *versus* concentration (g/L). analyst® software 1.6.3 calculated the intercept with the Y-axis (a), slope (b) and regression coefficient (r). The correlation co-efficient (r > 0.99) was calculated as shown in Fig. 3.

According to the presented formula, the regression residual (d_i) determines the vertical distance of observed values from the regression curve:

$$d_i = y_i - yy_i$$

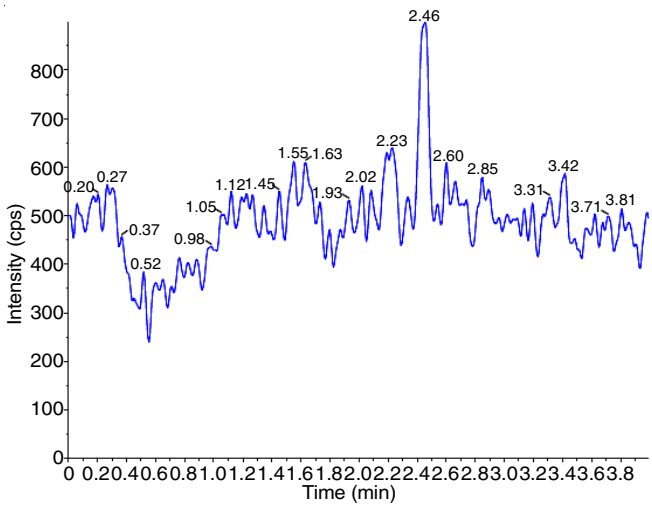

Fig. 1. Chromatogram of the blank extract of mango matrix – control

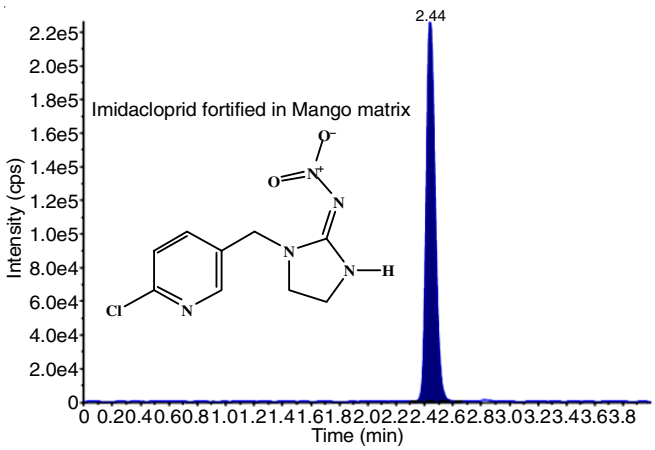

Fig. 2. Chromatogram of reference standard fortified in the matrix

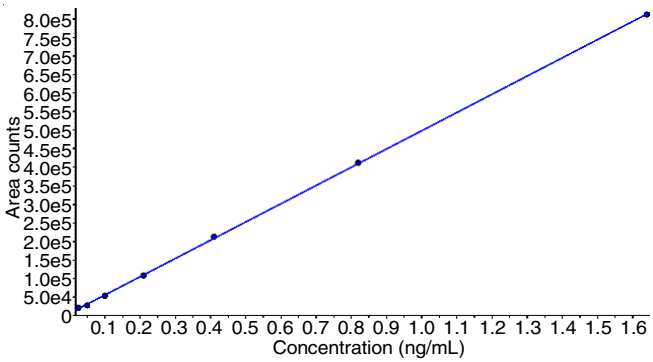

Fig. 3. Matrix matched calibration curve of imidacloprid

where, y_i = measured value, yy_i = estimated value that corresponds to y_i and is derived from the calibration function.

A residual plot was used to display the regression residual, as illustrated in Fig. 4. Visual examination was used to determine whether d_i were randomly distributed and therefore linear calibration was demonstrated. There was no discernible trend in the residuals, indicating that the calibration model was adequate [40].

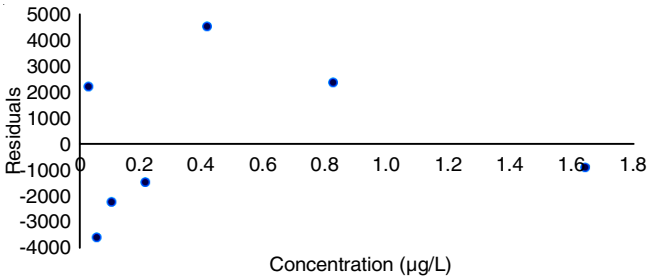

Fig. 4. Residual plot for the matrix-matched (% ME) linearity curve

As illustrated in Fig. 5, the LOQ was calculated by injecting different concentrations of imidacloprid solution. The precision (% RSD), accuracy (% recovery) and fortified reference standard in the matrix at the lower (0.50 µg/kg), middle (2.55 µg/kg) and higher (5.10 µg/kg) dose level as shown in Table-3. The % recovery was determined to be within an acceptable range of 70-110%. The results of method validation were concluding that the determination of pesticide residue of imidacloprid is possible through the validated analytical method.

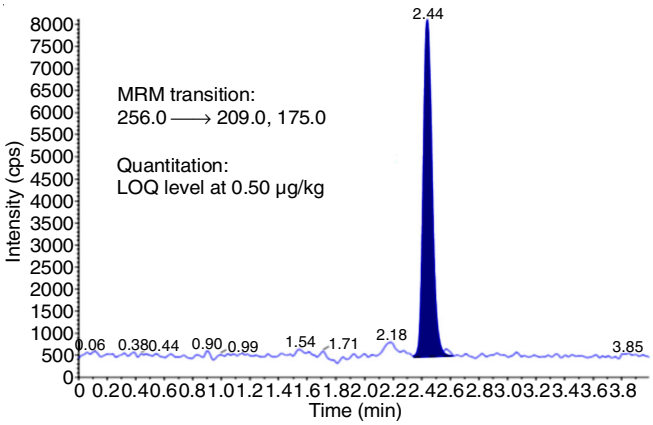

Fig. 5. Chromatogram of precision (% RSD) and accuracy (% recovery) – LOQ level

TABLE-3
SUMMARY RESULTS OF ANALYTICAL METHOD VALIDATION FOR IMIDACLOPRID PESTICIDE RESIDUE IN MANGO MATRIX

Parameter		Result
Test matrix		Alphonso mango (*Mangifera indica*)
Specificity (non-analyte interference)		No interference
Linearity	Concentration range (µg/L)	0.025 – 1.640
	Intercept (a)	6.79e + 003
	Slope of the line (b)	4.91e + 005
	Correlation coefficient (r)	0.999
	Equation: Y = bX + a	Y = 4.91e + 005x + 6.79e + 003
Limit of detection (LOD) (µg/kg)		0.50
Limit of quantification (LOQ) (µg/kg)		5.10
Precision (% RSD)	LOQ Level (0.50*)	2.19
	10 × LOQ level (5.10*)	1.41
Accuracy (% Recovery)	LOQ Level (0.50*)	99.92
	10 × LOQ level (5.10*)	107.01
Overall mean recovery (%)		103.47
Overall % RSD		4.00
(% Recovery) for fortified samples	Lower level (0.50*)	101.93
	Middle level (2.55*)	107.49
	Higher level (5.10*)	105.86

* = µg/kg

In mass spectrometry, two imidacloprid transitions were quantified in order to get a lower analyte response, since the first fragment, at *m/z* = 209, is due to NO_2 loss, while the second fragment, at *m/z* = 175, is due to both NO_2 and ^{35}Cl loss.

Conclusion

The experiment phase was performed for the imidacloprid residue method validation on Indian mango for the specific matrix of genus-species: *Mangifera indica L.* alphonso mango for the achieving of lower dose level concentration by using LC coupled with mass spectrometry (UPLC-ESI-MS/MS) with the most sensitive instrument. In this research work, the value-drawn linearity curve achieved the lower concentration calibration curve with the range of 0.025 to 1.640 µg/L with the % ME (matrix effects) of 9.92 for the quantitation level of 0.50 µg/kg LOQ level for imidacloprid. The analytical technique validation indicated that the approach is sensitive, precise and accurate for determining imidacloprid in the particular matrix of *Mangifera indica* from the Valsad District, India.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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