

Introduction:

Analysis of imidacloprid residue in soil and water samples is becoming increasingly important due to the health hazards. The purpose of analytical study in such cases is to obtain information about substances and analytes present in the soil and water sample. Analytical process involves several steps: sampling, sample preparation, separation, quantification and data analysis. Sample preparation is a very important step and indeed the bottleneck of analytical methodologies, in the analysis of soil and water for the presence of imidacloprid.

This chapter reports method for extraction of imidacloprid from soil and water and its quantitation using HPLC method. The extraction procedure and instrument parameters were validated in soil and water. The soil was collected from different places in Gujarat and characterized. The procedure for extraction of imidacloprid in different types of soil was optimized and validated. Pesticides degradation in the soil environment depend upon their physico-chemical properties of soil like, soil organic matter and soil minerals, soil *pH*, moisture. Soil characterization is play important role for dissipation of imidacloprid. We characterized the soil collected from different places of Gujarat.

This Chapter is divided in to two Sections

Section -1 Soil Characterization

Section -2 Method Validation

Soil Characterization

This study was performed to determine the physico-chemical properties of soil viz., *pH*, organic carbon, water holding capacity (WHC), sand content (particle size distribution), moisture content, oven dry weight. The soil was collected from Valvada, Bardoli, Umarsadi and Baroda, Gujarat, India. The characterization methods followed were as per Walkley and Black, (1934) and Baruah and Barthakur, (1997). The tests were performed at laboratory ambient temperature.

Collection and Preparation of Soil

The test soil was collected from a pasture land at a depth of 20 to 25 cm. The field was free from any chemical and biological contamination. A quantity of 5 kg well aerated

moist soil was collected from the different spots of the land at pre monsoon period. The soil was transferred to the laboratory on the collection day, after manual removal of stones and raw plant materials. The soil was sieved through 2.0 mm sieve and the moisture content was maintained between 40 to 60% of the maximum water holding capacity, using distilled water. The soil was stored at 4.0 ± 2.0 °C in refrigerator under aerobic condition.

Materials and Methods

Determination of Soil pH

Preparation of 0.01M CaCl₂ Solution

A quantity of 1.47 g calcium chloride dihydrate was weighed into a volumetric flask of 1.0 L capacity, dissolved in 400 mL distilled water and the volume was made upto the mark with distilled water.

Procedure

Ten gram air-dried soil was suspended in 25 mL 0.01M CaCl₂ solution and 25 mL distilled water, separately and kept overnight for equilibration. After equilibration period, the soil suspension was disturbed once and the pH was measured using a calibrated pH analyzer.

Determination of Organic Carbon

Preparation of Reagents

Potassium dichromate solution (1N): A quantity 24.52 g K₂Cr₂O₇ was dissolved in 500 mL distilled water.

Ferrous ammonium sulphate solution (0.5N) : A quantity of 98.00 g Fe(NH₄)₂(SO₄)₂.6H₂O was dissolved in 200 mL distilled water containing 3.8 mL concentrated H₂SO₄ into a 500 mL volumetric flask and the volume was made upto the mark with distilled water.

Concentrated H₂SO₄ with 1.25% Ag₂SO₄: A quantity of 1.25g Ag₂SO₄ was dissolved in 100 mL conc. H₂SO₄.

Orthophosphoric acid : 88 - 93%

Diphenylamine indicator: A quantity of 0.5 g diphenylamine was dissolved in 20 mL distilled water and 100 mL concentrated H₂SO₄ was added.

Procedure

One gram soil was weighed and transferred into a 500 mL conical flask in two replicates. Ten mL 1N K₂Cr₂O₇ solution was added with thorough mixing followed by 20 mL concentrated H₂SO₄. The flask was swirled 2-3 times and allowed to stand for 30 minutes for the reaction to complete. The mixture was diluted with 200 mL distilled water followed by addition of 10 mL orthophosphoric acid. The mixture was titrated with 0.5N ferrous ammonium sulphate solution using 1.0 mL diphenylamine indicator until the colour flashed from violet through blue to bright green. Blank titration (without soil) was also carried out in the similar manner.

Calculation

Volume of 1N K₂Cr₂O₇ used for oxidation of C = $0.5 \times (B-S)$ mL

[1 mL of 1N K₂Cr₂O₇ (=1 meq) = 3 (=12/4) mg of organic C = 0.003 g of organic C]

Walkley and Black (1934) averaged a 77% recovery of organic C by this method. Thus, the correction factor is $100/77 = 1.3$

% organic C in soil (uncorrected) = $0.5 \times (B - S) \times N \times 0.003 \times (100/W) \times 1.3$

Therefore, % organic C in soil (corrected) = $0.5 \times (B - S) \times N \times 0.003 \times (100/W) \times 1.3$

where,

W = Weight (g) of soil taken

B = Volume (mL) of 0.5N Fe (NH₄)₂(SO₄)₂ solution used for blank titration

S = Volume (mL) of 0.5N Fe (NH₄)₂(SO₄)₂ solution used for sample titration

N = Normality of K₂Cr₂O₇

Particle Size Analysis

Preparation of Reagents

Hydrogen peroxide (30%)

HCl (2N) : A quantity of 87.40 mL concentrated HCl was mixed with 400 mL distilled water into a 500 mL volumetric flask and volume was made upto the mark with distilled water.

AgNO₃ (0.1N) : A quantity of 1.70 g silver nitrate was dissolved in 80.0 mL distilled water into a 100 mL volumetric flask and volume was made upto the mark with distilled water.

NaOH (0.1N) : A quantity of 4.0 g sodium hydroxide was dissolved in 800 mL distilled water into a 1000 mL volumetric flask and volume was made upto the mark with distilled water.

Phenolphthalein indicator.

Procedure

Treatment with Hydrogen Peroxide

Twenty gram air dried soil was weighed and transferred into a 500 mL beaker followed by addition of 15 mL H₂O₂ in two replicates. The beaker was swirled and allowed to stand for 10 minutes to complete the reaction. The beaker was then placed on a water bath to continue the digestion with intermittent stirring until the reaction completely subsided. As the frothing persisted, the procedure was repeated. The beaker was then cooled and the walls were rinsed with distilled water.

Treatment with Hydrochloric Acid and Filtration

To remove CaCO₃ present in the soil, 25 mL 2N HCl was added to the same beaker and contents were stirred. The content was diluted to 250 mL with distilled water and allowed to react for one hour with intermittent shaking. The content was then filtered using Whatman filter paper No 1. The soil was washed with distilled water until the filtrate was free from chloride (AgNO₃ solution test).

Dispersion and Separation of Coarse Sand

The soil sample was transferred from the filter paper to a 500 mL polypropylene bottle with a jet of distilled water and the volume was made upto 500 mL with distilled water. Few drops of phenolphthalein indicator were added to the mixture followed by addition of 0.1NaOH until the whole suspension turned to pink colour. The content of the bottle was then stirred for dispersion. After dispersion, content was transferred to a 70 mesh sieve (ASTM) and the coarse sand was separated out. The content on the sieve was then washed with a jet of distilled water until no more clay and silt remained over the sieve. The coarse sand was dried at 105 °C in oven to a constant weight and the weight was recorded.

Determination of Silt + Clay

After separation of coarse sand, the suspension was transferred to a 1000 mL measuring cylinder and volume was made upto 1000 mL with distilled water. The content was mixed thoroughly and kept in constant temperature chamber (25 °C) to ensure minimum variation of temperature between the two samplings. Ten mL sample was drawn at a particular time depending on the temperature of the suspension and the size of the desired particle (silt + clay and clay) to be determined. The samples withdrawn at different time points were transferred to pre-weighed dishes and dried at 105 °C to a constant weight and the weight was recorded.

Determination of Fine Sand

To separate the fine sand, the bulk of the suspension was decanted and sediment was transferred to a 500 mL beaker with a jet of distilled water. The sediment was washed with distilled water and turbid solution was decanted until the liquid above the sediment was no longer turbid. The sediment was dried at 105 °C to a constant weight and the weight was recorded. The moisture content of the test soil was determined simultaneously.

Observations and Calculation

- a. Weight of the air dried soil taken = X g
- b. Moisture content of the soil = M % (on dry weight basis)
Therefore, oven dry wt. of the soil (g) = $(100 \times X) / (100 + M) = W$
- c. Weight of the dish (g) = W_1

- d. Weight of the dish with coarse sand (g) = W_2
Percent coarse sand, $P = (W_2 - W_1) / W \times 100$
- e. Temperature of the suspension = $T\text{ }^{\circ}\text{C}$
- f. Sediment commencement time = t_0
- g. Time of the sampling (silt + clay) = $t_0 + t_{\text{SIC}}$
- h. Time of the sampling (clay) = $t_0 + t_c$
At, $t_0 + t_{\text{SIC}}$
- i. Weight of the dish (g) = W_3
- j. Volume of the suspension taken for analysis = 10 mL
- k. Oven dry weight of dish and silt + clay (g) = W_4
- l. Weight of silt + clay (g) = $(W_4 - W_3)$

$$\text{Percent silt + clay} = \frac{W_4 - W_3}{W} \times \frac{1000}{10} \times 100$$

- m. Weight of the dish (g) = W_5
- n. Volume of the suspension taken for analysis = 10 mL
- o. Oven dry weight of dish + clay (g) = W_6
- p. Weight of clay (g) = $(W_6 - W_5)$

$$\text{Percent clay} = \frac{W_6 - W_5}{W} \times \frac{1000}{10} \times 100$$

$$\text{Per cent silt, } P_{\text{SI}} = P_{\text{SIC}} - P_{\text{C}}$$

- q. Weight of the dish (g) = W_7
- r. Oven dry Weight of dish + fine sand (g) = W_8

$$\text{Percent fine sand} = \frac{W_8 - W_7}{W} \times 100$$

Water Holding Capacity (WHC)

Procedure

A quantity of 50 g air-dried soil was weighed in two replicates and transferred into a funnel with a Whatman filter paper No 1 fitted inside a funnel and clamped on a stand. The water was added into the funnel to moist the soil upto saturation, which was judged by dropping excess water from the funnel. The wet filter paper with wet soil was transferred into a porcelain crucible when the water dropping from the funnel had stopped. Wet and dry filter papers along with porcelain crucible were weighed. Wet soil with filter paper and crucible was weighed. The samples were placed in the oven for drying at the temperature of 105 °C until constant weight was observed. The samples were taken out from the oven and weighed for dry soil weight with filter paper and crucible.

Calculation

The water holding capacity of soil (mass basis) as the percentage of dry soil was calculated using the following formula:

$$\text{Water holding capacity (\% of the soil)} = [(A - B) / (B)] \times 100$$

where,

$$\begin{aligned} \text{Weight (g) of wet soil (A)} &= (\text{Weight of wet soil with filter paper plus crucible}) \\ &- (\text{Weight of wet filter paper with crucible}) \end{aligned}$$

$$\begin{aligned} \text{Weight (g) of dry soil (B)} &= (\text{Weight of dry soil with filter paper plus crucible}) \\ &- (\text{Weight of dry filter paper with crucible}) \end{aligned}$$

Results and Discussion

This study was performed to determine physico-chemical properties of soil viz., pH, organic carbon content, and water holding capacity (WHC), moisture content, oven dry weight, sand content (particle size distribution) for soil collected from Valvada, Bardoli, Umarsadi and Baroda, Gujarat, India. The per cent organic carbon, pH and water holding capacity were found to be 0.84%, 7.00 and 47.51%, 1.37%, 6.60 and 67.06, for Baroda soil and Bardoli, respectively. The corresponding values for Umarsadi and Valvada soil were 0.42%, 4.32 and 65.74%, 0.86%, 7.15 and 60.06, respectively. The results are depicted in **Table 2**.

Section -2

Analytical Method Development

Analytical method to determine the residue of imidacloprid by using HPLC was developed by selecting suitable instrument parameters and solvent to get clear resolution and separation of compound of interest.

Materials and Methods

Analytical reference standards of imidacloprid (98.5% purity) were obtained from Dr. Ehrenstorfer, Germany. All the other chemicals and solvents used were analytical and HPLC grade. HPLC A Shimadzu LC-2010 AHT equipped with UV detector, Phenomenex C-18 column (250 mm length \times 4.6 mm i.d. and 0.5 μ m particle size) and LC-solution software was used. Mobile phase A: 0.01% (v/v) acetic acid in water (60). Add 0.1 mL acetic acid and dilute to 1 litre with water. Mobile phase B: acetonitrile (40). The mobile phase was delivered to mode of low pressure gradient system at 1mL flow rate and detector set a 252 nm λ_{max} was used for analysis. Imidacloprid standard showed sharp peak at 4.93 minute under the described HPLC conditions. Fig. 2, 3 and 4 depicts typical chromatograms of the separation of imidacloprid reference standard and recovery in soil and water samples.

The soil characterization was performed to determine different physico-chemical properties viz., pH, organic carbon, water holding capacity (WHC) and clay content (particle size distribution) of soils collected from different parts of Gujarat, India.

The test soils were collected from different parts of Gujarat, India; viz. (a) Baroda (b) Bardoli, (c) Umarsadi, (d) Vikram Farm, Valvada and were coded as Soil-1 (sandy loam soil), Soil- 2 (clay soil), Soil-3 (red soil), and Soil-4 (black soil), respectively. Based on organic carbon (%), pH and clay content, the soils i.e. Soil-1, Soil- 2, Soil-3, and Soil-4, were classified (OECD No 106, 2006). The soil characteristics data is shown in **Table 2**.

Validation of the Analytical Method

The analytical method for the determination of imidacloprid residue was validated by analysis of imidacloprid in soil and water samples. The validation focused on the

following aspects: (i) specificity, (ii) linear dynamic range (LDR), (iii) limit of detection (LOD), (iv) limit of quantitation (LOQ), (v) precision (% RSD) [repeatability and reproducibility] and (vi) accuracy (% Recovery).

Specificity

The solvent (used for standard solution and sample solution preparation), mobile phase, standard solution and control soil samples and water sample were injected into a High Performance Liquid Chromatograph (HPLC) using selected instrument parameters. The interference (if any), in the determination of residues of imidacloprid was reported. Interference should not be more than 3% to the total peak area measured for target active ingredient.

Linear Dynamic Range (LDR)

Five different concentrations of imidacloprid reference standard were prepared and injected into HPLC in duplicate. The linear calibration curve was established by plotting the mean peak area against concentration (mg/L). The correlation coefficient (r), intercept (a) and slope (b) were calculated.

Limit of Detection (LOD)

The minimum quantity of imidacloprid, which could be detected by the HPLC with signal to noise ratio of 3 ± 0.5 , was calculated as limit of detection (LOD).

Limit of Quantitation (LOQ)

The minimum quantity of imidacloprid, which could be quantified by the HPLC with signal to noise ratio between 5 and 10, was calculated as limit of quantitation (LOQ).

Precision (% RSD)

Determination of Repeatability

Five replicate injections of fortified and extracted soil and water sample solutions of imidacloprid along with sequential injections of standard solution were injected into HPLC using the optimized instrument parameters. The residue of imidacloprid residue % RSD was calculated.

Calculation of Precision (% RSD)

Precision was defined by the relative standard deviation (% RSD). The precision (% RSD) was calculated as follows.

$$\text{Precision (\%RSD)} = \frac{\text{Standard deviation}}{\text{Arithmetic mean (a.i. content)}} \times 100$$

Accuracy (% Recovery)

Accuracy (% recovery) of the analytical method was determined soil and water samples fortified and extracted with solvent. The samples was fortify at LOQ and $10 \times \text{LOQ}$ levels for soil and water pH 4, 7, and 9.

Calculation of % Recovery (Accuracy)

% Recovery for active ingredient was calculated as follows:

The linear calibration curve was established by plotting mean peak area of standard of imidacloprid solutions against concentrations (mg/L). The regression constant viz. slope (a), intercept (b) and correlation co-efficient (r) were calculated. The a.i. content of imidacloprid was calculated using the following formula:

$$\text{Imidacloprid Concentration (mg/L)} = \frac{(Y - a)}{b}$$

Calculation of % Recovery (Accuracy)

$$\% \text{ Recovery} = \frac{\text{Quantity recovered}}{\text{Quantity fortified}} \times 100$$

The specificity of the analytical method was studied by injecting solvent, reference standard solution, control soil and water sample different pH extracts injected onto

HPLC. There was no interference of the components with each other. The linearity was established by injecting five different concentrations, viz. 0.02 mg/L to 5.00 mg/L, and determining the response of the compound; these were fitted by linear regression to assess the linearity. Detection Limit (signal-to-noise ratio = $3 \pm 0.5:1$) was established. The linear dynamic range of imidacloprid is shown in Figure 1.

The precision (% RSD) of the analytical method was determined by five replications in duplicate injection of fortified substrate soil and water extracts at LOQ level. The accuracy (% recovery) of the method was determined by five replications in duplicate injection of fortified substrate soil and water extracts at LOQ and 10 times LOQ levels. Precision (% RSD) should not exceed 20 %.

Sample fortification

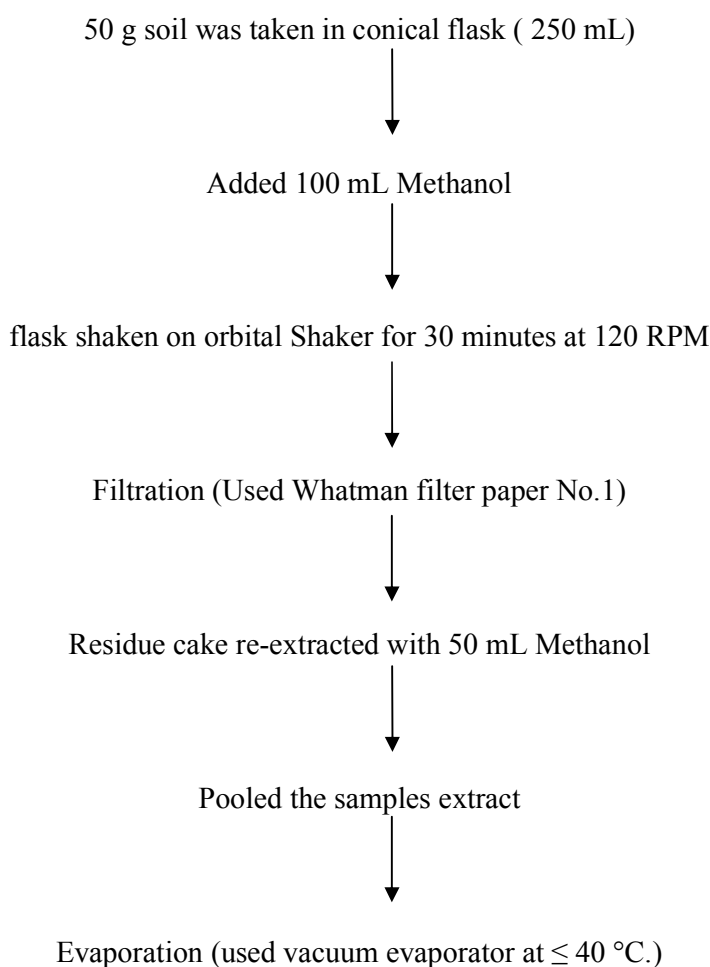
A representative sample (50g) of a particular soil (black, red, sandy loam or clay) was transferred to 250mL conical flask. The soil sample was fortified with imidacloprid at two different fortification levels: LOQ and $10 \times$ LOQ, separately. A volume of 0.5 and 5.00 mL imidacloprid solution was transferred to each conical flask for 0.02 and 0.20 mg/L fortification levels. In case of water, samples at pH 4, 7 and 9 (25 mL) were transferred in to volumetric flask 50 mL capacity and fortified with imidacloprid at LOQ and 10 times LOQ levels separately. A volume of 0.25 and 2.5 mL imidacloprid was transferred to each volumetric flask for 0.02 and 0.20 mg/L fortification levels. The control samples were processed similarly where in 0.25 and 2.5 mL acetonitrile was added.

Extraction Procedure from Soil

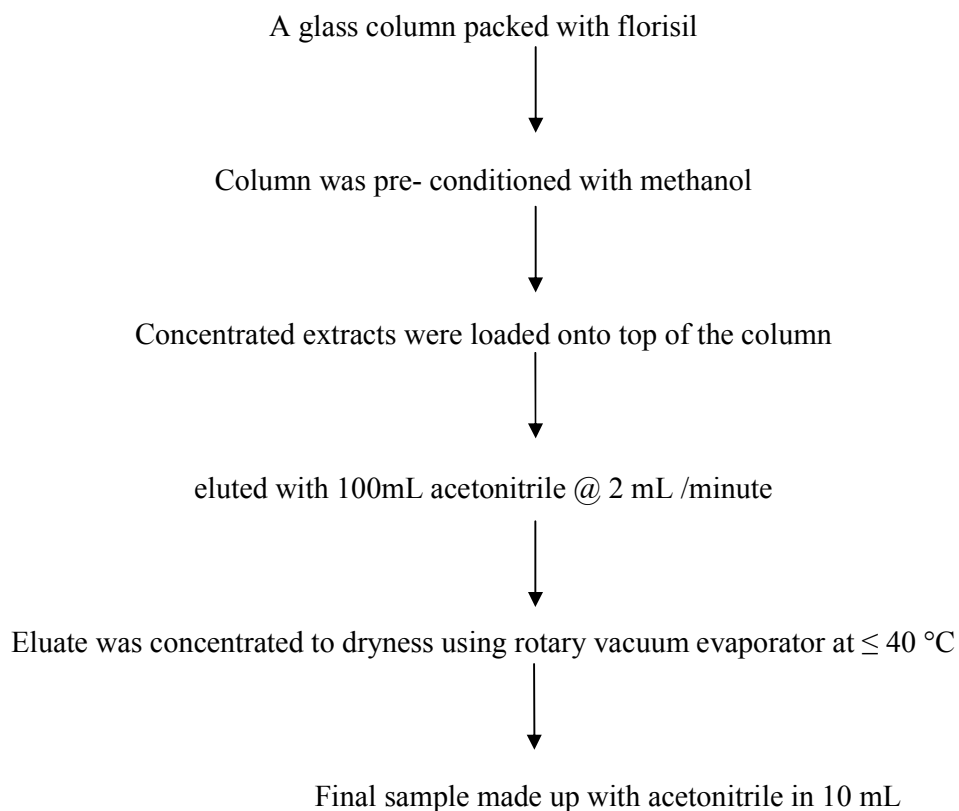
A volume of 100 mL methanol was transferred into the conical flask containing (50g) fortified soil sample and allowed to stand for 2 hour. The conical flask was placed onto orbital shaker for 30 minutes. After shaking, the solutions were filtered into a round bottom flask of 500 mL capacity through Whatman filter paper No.1 bearing a bed of anhydrous sodium sulphate. Solvent was removed using vacuum evaporator. The residual cake was re-extracted twice with additional volume of 50 mL methanol. The methanol extracts were collected, pooled and concentrated to smaller volume (5 to 10 mL) using vacuum evaporator at ≤ 40 °C. The concentrated extract was subjected to further clean up by column chromatography. A glass column packed with florisil as

adsorbent placed in between two layer of anhydrous sodium sulphate was employed. The column was pre- conditioned with methanol and concentrated extracts were loaded onto the top of the column and eluted with 100mL acetonitrile @ 2 mL /minute. Eluate was concentrated to dryness using rotary vacuum evaporator at $\leq 40^{\circ}\text{C}$ and residue re-dissolved in 5mL acetonitrile. The samples were transferred into volumetric flask of 10 mL capacity using Whatman No. 1 filter paper and final volume was made up to the mark with acetonitrile. The control soil and water samples procedure followed same and injected onto HPLC.

Flow Chart for Extraction Procedure of Imidacloprid from Soil



Sample Cleanup by Column Chromatography

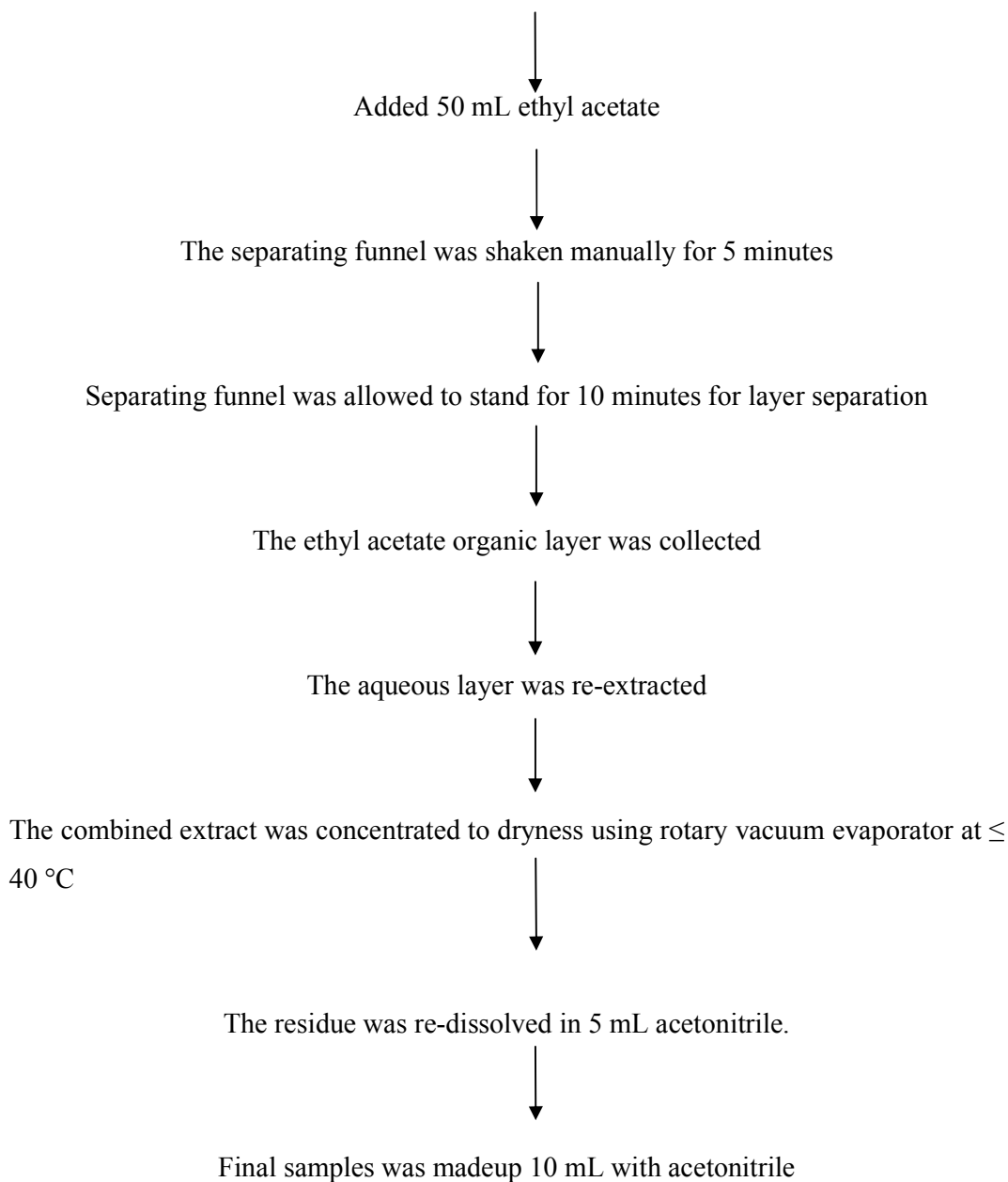


Extraction procedure for water samples

The fortified water samples (25 mL) at different pH viz. 4.0, 7.0 and 9.0, were transferred separately into a separating funnel of 250 mL capacity and a volume of 50 mL ethyl acetate was added into it. The separating funnel was shaken manually for 5 minutes with frequent vent. The contents of the separating funnel were allowed to stand for 10 minutes for layer separation. The ethyl acetate organic layer was collected into a round bottom flask of 500 mL capacity. The aqueous layer was re-extracted twice with additional volume of 50 mL ethyl acetate and collected in the same round bottom flask. The combined extract was concentrated to dryness using rotary vacuum evaporator at $\leq 40^{\circ}\text{C}$ temperature. The residue was re-dissolved in 5 mL acetonitrile. The samples were transferred into volumetric flask of 10 mL capacity through Whatman No. 1 filter paper and final volume was made up to the mark with acetonitrile.

Flow Chart for Extraction Procedure of Imidacloprid from Water

A volume of 25 mL water samples was taken in separating funnel (250 mL capacity)



The quantitative analysis of imidacloprid in soil and water extracts was conducted by reverse phase HPLC technique.

Results and Discussion

Soil characterization: Table 2 shows that the mean pH (0.01M $CaCl_2$ suspension) of the different soils as determined (soil solution of (1:2.5) for Soil-1, Soil- 2, Soil-3, and Soil-4 were 7.15, 6.60, 4.32, and 7.00, respectively. The corresponding mean pH (distilled water suspension) of the different soils determined (soil solution 1:2.5) were 7.47, 6.91, 4.82 and 7.25 respectively. The percent organic carbon for soils i.e. Soil-1, Soil- 2, Soil-3, and Soil-4 were 0.86, 1.37, 0.72 and 0.84, respectively. The coarse sand content of the test soils i.e. Soil-1, Soil- 2, Soil-3, and Soil-4 were 3.64, 2.42, 10.13 and 1.63%, respectively. The fine sand content was 10.64, 9.18, 7.47 and 59.96% respectively. The silt content of the test soils were 19.01, 10.73, 7.98 and 5.06%, respectively. The percent clay content of the test soils were 38.00, 53.68, 42.55 and 15.20, respectively. The water holding capacity of the test soils i.e. Soil-1, Soil- 2, Soil-3, and Soil-4 were 60.06, 67.06, 65.74 and 47.51%, respectively. The collected soil was analysed and soil characterised accordance with the above data. If soil was contain above 40% sand called sandy loam soil.

HPLC method: The linearity of the detector response was tested for imidacloprid, in solvent and in matrix (soil) over the concentration range of 0.02 to 5.00 mg/kg. A very precise linear relation between the injected amount and the resulting peak area was observed over the entire concentration range with correlation coefficient value of 0.999 Table 3. Ishii-Y, *et. al.* 1994 have also reported an HPLC method for determination of imidacloprid residue in 9 kinds of crops and soil. The method consisted of extraction with acetonitrile /water (80:20 v/v), pre-washing of the concentrated extracts with cyclohexane and alkaline solution, silica gel column chromatography, and finally reversed-phase HPLC. The recoveries of imidacloprid were 75-109%. The limits of detection were 0.005, 0.01 and 0.02 mg/kg for crops, rice straw and soil, respectively. We developed the analytical method for imidacloprid analysis in soil and water LOD and LOQ is lower than reported method and recovery is better than other method.

We have developed and validated the analytical method for the determination of low amounts of imidacloprid in/on different soils viz. sandy loam soil, clay soil, red soil and black soil and in water at different pH values, viz. 4, 7 and 9 using HPLC. The accuracy and precision of the method was evaluated on the basis of the recoveries obtained for fortified soil and water samples. The limit of quantitation (LOQ) was

found to be 0.02 mg/kg for imidacloprid in soil and 0.02 mg/L in water. The limit of detection (LOD) was 0.01 mg/kg for imidacloprid in soil and 0.01 mg/L in water. Recoveries for imidacloprid were 95.18, 94.66, 95.27 and 94.78 % in black soil red soil sandy loam and clay soil, respectively. The recoveries for imidacloprid were 96.86, 96.14 and 92.34 % in water at pH 4.0, 7.0 and 9.0 respectively. The accuracy (% recovery) data in soil and water is depicted in **Table 1**. The % RSD and recovery range from 1.21 to 3.37 and 94.66 to 95.27% in soil. The % RSD was the resulting mean recovery ranged from 92.34 to 96.86% in water with relative standard deviations between 1.66 and 3.23%. These data demonstrate the excellent sensitivity, selectivity and precision of the method.

Conclusion

We have developed and validated a rapid, simple, sensitive and specific method for the determination of imidacloprid residues in/on different soil viz. sandy loam, clay, red and black soil and waters viz. pH 4, 7 and 9 through HPLC. A simple clean-up procedure using column chromatography was found to yield sufficiently clean samples.

Table 1 Precision (%RSD and Accuracy (% Recovery) of Imidacloprid in Soil and Water

Substrates	Fortification of LOQ and 10X LOQ levels in mg/kg	%Recovery	Mean %recovery	SD	%RSD
Black soil	0.02	97.20	95.18	1.18	1.21
	0.20	93.16		2.24	2.40
Red soil	0.02	97.25	94.66	1.86	1.91
	0.20	92.07		3.10	3.37
Sandy loam soil	0.02	97.00	95.27	1.97	2.03
	0.20	93.54		3.01	3.22
Clay soil	0.02	96.35	94.78	1.31	1.36
	0.20	94.60		2.23	2.36
Water pH 4	0.02	97.60	96.86	1.63	1.67
	0.20	96.11		2.21	2.30
Water pH 7	0.02	96.30	96.14	1.60	1.66
	0.20	95.98		3.10	3.23
Water pH 9	0.02	95.00	92.34	1.25	1.31
	0.20	89.67		2.34	2.61

Table 2 Characteristics of soil

Soil Sampling (Location)	Characteristics								Soil Type
	Organic Carbon (%)	pH (0.01M CaCl ₂)	pH Distilled Water	Coarse sand (%)	Fine sand (%)	Silt (%)	Clay (%)	Water Holding Capacity	
Valvada	0.86	7.15	7.47	3.64	10.64	19.01	38.00	60.06	Black soil
Bardoli	1.37	6.60	6.91	2.42	9.18	10.73	53.68	67.06	Clay Soil
Umarsadi	0.42	4.32	4.82	10.13	7.47	7.98	42.55	65.74	Red Soil
Vadu Baroda	0.84	7.00	7.25	1.67	59.96	5.06	15.20	47.51	Sandy loam Soil

Table 3 Data for Linearity Determination of Imidacloprid in Solvent

Concentration (mg/L)	Mean Peak Area	% Variation
0.02	647.00	0.62
0.05	1628.50	0.06
0.25	8172.00	0.05
1.00	32523.00	0.11
5.00	165401.50	0.11

Regression parameters Imidacloprid standard in solvent

Slop: (b) = 33096.66

Y-axis intercept: (a) = -159.78

Correlation coefficient: (r) = 0.999

Figure 1 Linearity for Imidacloprid in Solvent

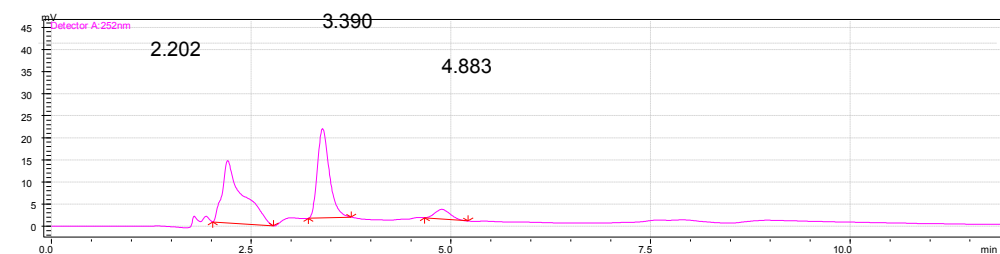
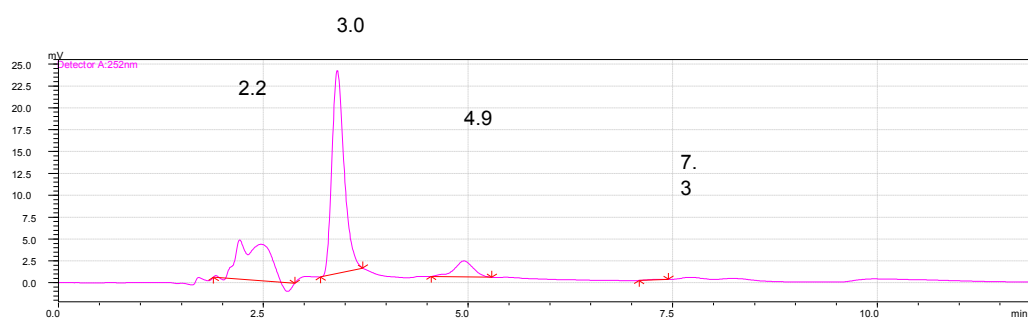
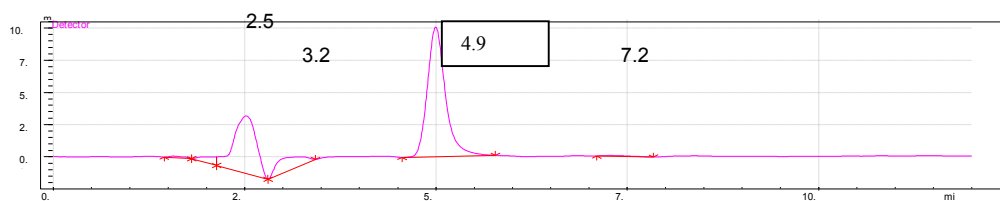
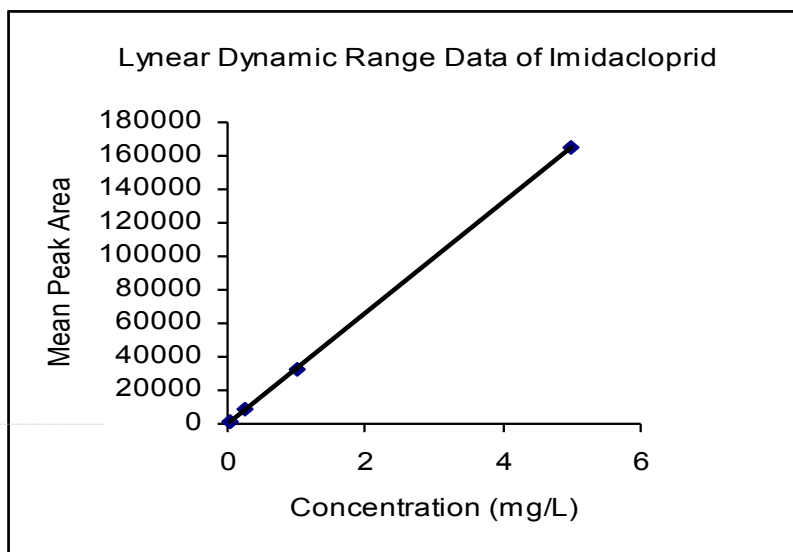


Figure. [2] Chromatograms of reference standard of imidacloprid 5.00 mg/L, [3] and [4], chromatogram of soil and water samples for recovery in sandy loam soil and water *pH* 4.

Figure 2- Recovery % of Imidacloprid in Soil

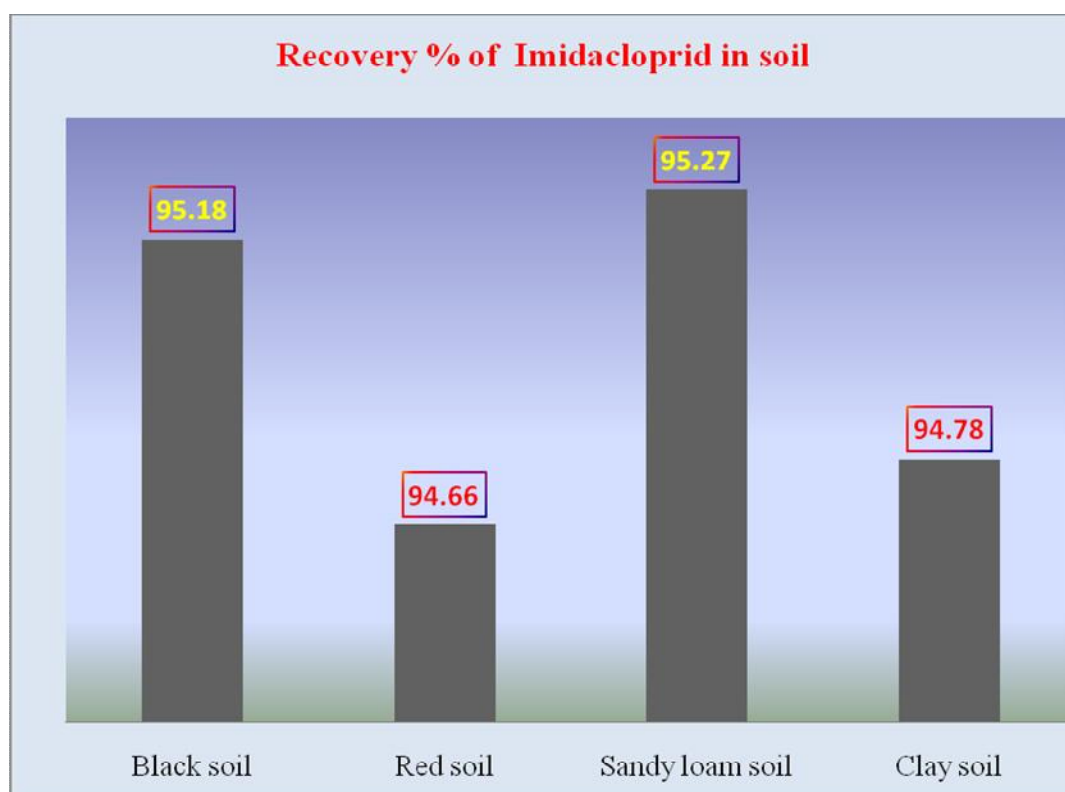
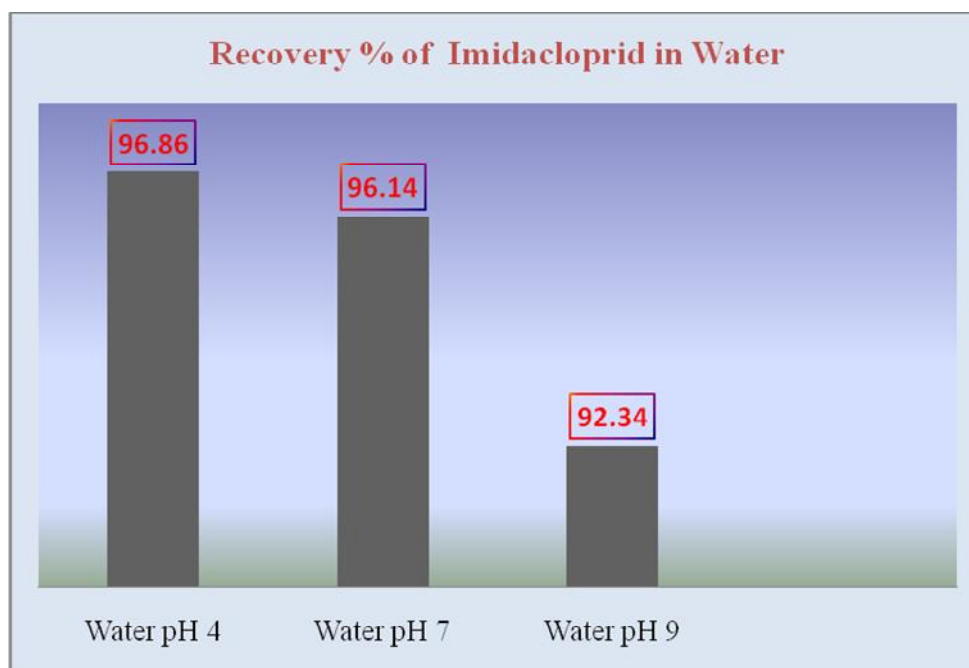


Figure 3- Recovery % of Imidacloprid in Water



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