RASĀYAN *J. Chem.*
Vol. 19 | No. 3 | 1374-1381 | July-September | 2026
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

ASSESSMENT OF THE PERSISTENCE STUDIES OF PRETILACHLOR HERBICIDE RESIDUES IN GROUND WATER DURING THE INDIAN WINTER CLIMATIC SESSION EMPLOYING A VALIDATED GC-FID METHOD

K. Kiranmai[1], V. Sridhar[2], Tentu Nageswara Rao[3], and Y. Prashanthi[2,✉]
[1]Department of Chemistry, Andhra Mahila Sabha Arts and Science College for Women, Hyderabad-500 007, India
[2]Department of Chemistry, Mahatma Gandhi University, Nalgonda-508254, TS, India
[3]Department of Analytical Chemistry, Isosphere Biosciences Pvt Ltd., Hyderabad, TS, India
✉Corresponding author:dryprasanthi@gmail.com

ABSTRACT
A study was conducted to evaluate the persistent behaviour of pretilachlor in groundwater by applying uniform concentrations: T0 – Untreated Control, T1 – Pretilachlor 37% EW (Emulsifiable in Water) at 0.5 µg/mL, and T2 – Pretilachlor 37% EW (Emulsifiable in Water) at 1.0 µg/mL. The spiked samples were exposed to sunlight. Water samples were collected at various intervals (0.2, 1, 2, 5, 10, 20, 30, 40, and 50 days). All samples were analysed until the residues dropped below the detectable level. The quantification of Pretilachlor residues was conducted using a validated gas chromatography with a flame ionisation detector (GC-FID), with key parameters including a DB-1MS column (30 m × 0.25 mm ID, 0.25 µm film thickness), an injector temperature of 250° C, a detector temperature of 260° C, and a column oven temperature of 240° C. The analysis involved a 1.0 µL injection volume with a split ratio of 10 and utilised nitrogen as the carrier gas at a flow rate of 2.0 mL/min. The total run time was 6.0 minutes, with a retention time for Pretilachlor of approximately 3.2 minutes. The method's limit of detection was 0.01 µg/mL, and the limit of quantification (LOQ) was 0.03 µg/mL based on recovery. The residues of Pretilachlor in the aqueous solution decreased to below the detectable level by the 50th day. The DT50 (Half-Life) of Pretilachlor was determined through regression analysis of the dissipation data, resulting in a DT50 value of 14.37 days for the T1 dose and 14.42 days for the T2 dose.
Keywords: Pretilachlor, Persistence, DT50, Residues, Ground Water, GC-FID
RASĀYAN *J. Chem.*, Vol. 19, No. 3, 2026

INTRODUCTION

Pesticide persistence studies evaluate how long pesticides remain present in soil, water, and ecosystems, concentrating on their degradation, mobility, and environmental impacts.[1] These studies are crucial for assessing environmental risks, ensuring human health safety, and guiding regulatory decisions. Significant factors that affect pesticide persistence include chemical characteristics such as solubility and soil adsorption, along with environmental conditions like soil type and climate.[2-5] Monitoring involves laboratory degradation tests and field sampling to detect residues.[6] Current trends highlight sustainable agriculture, microbial bioremediation, and the application of advanced materials for remediation. In[7] summary, persistence studies offer vital information for managing long-term pesticide risks and formulating effective environmental strategies.[8] The mechanisms of pesticide persistence elucidate the extended presence of specific pesticides in the environment, influenced by factors such as chemical stability, low biodegradability, soil adsorption, and environmental conditions.[9] Notable mechanisms encompass the chemical stability of compounds like DDT, which resists degradation due to robust bonds; inadequate microbial degradation of complex molecules; strong binding to soil particles that limits mobility; and hydrophobicity, which facilitates bioaccumulation in organisms.[10] Environmental factors such as temperature, sunlight, and moisture further affect degradation rates. The ecological implications include bioaccumulation within food chains, non-target toxicity to beneficial species, and long-term contamination issues.[11] Research indicates that bioremediation techniques, photocatalysis, and biopesticides may serve as potential solutions, in addition to policy measures aimed at limiting persistent

http://doi.org/10.31788/RJC.2026.1939752

pesticides.[12] Important persistence data concerning pesticides signifies measurable information that helps scientists and regulators grasp the environmental lifespan and risks tied to pesticides. Crucial persistence data includes half-life (DT_{50}), DT_{90}, sorption coefficient (Koc), water solubility and partitioning (log Kow), degradation pathways, and field dissipation studies. These indicators facilitate risk assessment, regulatory decisions, remediation design, and agricultural planning. Pesticide residues in water can be detectable for different lengths of time, which are affected by both chemical properties and environmental factors.[13] Emerging remediation techniques include photocatalysis, bio-based adsorbents, and bioremediation utilising engineered microbes. In conclusion, effective monitoring and remediation strategies are critical due to the substantial environmental and health risks associated with pesticide residues in water.[14] Pretilachlor serves as a selective, pre-emergence herbicide, predominantly employed in rice agriculture to combat numerous annual weeds, including grasses and sedges. Pretilachlor is a synthetic chloroacetanilide herbicide primarily employed in rice cultivation as a pre-emergence treatment. Its toxicity is characterised by neurological and gastrointestinal symptoms that mimic organophosphate poisoning, although it does not act as an acetylcholinesterase inhibitor. Concerns regarding the environment include its moderate persistence and acute toxicity to aquatic life, resulting in its classification as a moderately hazardous pesticide under FAO/WHO guidelines.[15]

EXPERIMENTAL

Materials

The reference analytical standard for pretilachlor, with a purity of 97.60 % (g/g), was sourced from Ehrenstorfer. The test item, Pretilachlor 37% EW (Emulsifiable in Water), was acquired from the local market in Hyderabad. Acetone of GC grade was obtained from Merck India Limited. Groundwater samples were collected from Charlapalli in Telangana.

Standard Stock Solution

Exactly 10.25 mg of the pretilachlor reference standard, exhibiting a purity of 97.60%, was accurately weighed into a 10 mL volumetric flask. The compound was dissolved in 5 mL of acetone, subjected to sonication, and then the volume was adjusted to the mark with the same solvent. This procedure yielded a solution concentration of 1000.40 µg/mL, which was later stored in a freezer at -18°C. The stock standard solutions remained stable for a duration of up to 3 months. Working standards of appropriate concentrations were prepared from the stock solutions by diluting with acetone immediately prior to sample preparation.

Sample Stock Solution

An exact quantity of 25.29 mg of the test substance (purity 37.12%) of pretilachlor was placed into a 10 mL volumetric flask. The substance was dissolved in 5 mL of acetone, treated with sonication, and then adjusted to the mark with further acetone. This yielded a concentration of 938.76 µg/mL solution. The stock sample solution was utilized for preparing dose samples (T1 and T2) in groundwater.

Application Data

Three replication doses were developed in groundwater utilizing a pretilachlor sample stock solution. The first dose is the untreated control, identified as T0. The second dose, T1, possesses a concentration of 1 µg/mL, which was realised by fortifying 0.938 mL of the test item stock solution in 1000 mL of groundwater. The third dose, T2, has a concentration of 2 µg/mL, which was achieved by fortifying 1.876 mL of the test item stock solution in 1000 mL of groundwater.

Sampling Data

The samples underwent exposure to direct sunlight, and they were prepared by thoroughly mixing and subsequently subsampling 20ml with a pipette. Water samples were collected on specified days: 0, 1, 2, 5, 10, 20, 30, and 40 days. The laboratory environment was regulated to maintain a temperature range of 20 °C to 25 °C.

Instrument Conditions

The conditions for Gas Chromatography (GC-FID) were specified as follows: The Agilent Gas Chromatograph model 6890N, utilising EZchrom software, was employed. The column used was DB-

1MS, which is 30 m long and has an internal diameter of 0.25 mm, with a film thickness of 0.25 µm. An injection volume of 1.0 µL was applied, with the injector temperature set at 250°C. Nitrogen was the carrier gas, flowing at a rate of 2.0 mL/min. The injection mode was split, with a split ratio of 10. Acetone was utilised as both the solvent for needle rinsing and the diluent. The column oven temperature was maintained at 240°C, and the total analysis time was 6.0 minutes. The FID temperature was set to 260°C, with nitrogen as the makeup gas flowing at 30.0 mL/min, and hydrogen and air flowing at 30.0 mL/min and 300.0 mL/min, respectively. The retention time for Pretilachlor was approximately 3.2 minutes.

Method Validation

Validation of methods is essential for guaranteeing the credibility of analyses. This study focused on the parameters of linearity, recovery, repeatability, and the limits of detection (LOD) and quantification (LOQ). The accuracy of the method was evaluated through recovery tests, using samples spiked at concentration levels of 0.03, 0.3, and 3.0 µg/mL. Linearity was assessed by utilising various known concentrations (0.01, 0.03, 0.5, 1.0, 10.0, and 50.0 µg/mL), which were prepared by diluting the stock solution. The limit of detection (LOD, µg/mL) was defined as the lowest concentration that generated a response of 3.3 times the baseline noise, as determined by the analysis of a control sample. The limit of quantification (LOQ, µg/mL) was established based on recovery tests.

Dissipation Kinetics Studies

To explore the dissipation kinetics of pretilachlor residues in water, we utilised concentrations of 1.0 µg/mL (T1) and 2.0 µg/mL (T2) with triplicate replications, alongside control samples for comparative analysis. The samples were exposed to direct sunlight. Aliquots were collected at specified intervals. Throughout the study, water samples were gathered, with temperatures ranging from 25 to 34°C. A 0.45 mm PTFE membrane filter was used to filter the samples obtained at various sampling times. The resulting filtrates were then placed into amber-colored vials. Before conducting GC analysis, all samples were stored at 5°C in a dark environment. Samples were collected on multiple occasions until the concentrations of T1 and T2 decreased below the limit of quantification (LOQ). After completing the analysis based on the occasions and concentrations, we calculated the half-life (DT50) of pretilachlor using the provided formula.

The first-order kinetics form is:

$$-\ln(C_t/C_0) = k$$

$$t_{1/2} = DT50 = \ln 2/k = 0.6931/k$$

Where,

C_0 - Herbicide concentration at time zero

C_t - herbicide concentration at time t,

k - The rate constant

Sample Process

The representative gathered 5 ml from each concentration of water samples, concentrating them to dryness through a vacuum rotary evaporator, and afterwards diluted with acetone to the required mark. The prepared solution was then injected into the GC.

RESULTS AND DISCUSSION

Specificity

The specificity of the method was determined by injecting the diluent solvent, the reference solution (Linearity solution-1), sample solution (P1) into the GC-FID. From the analysis, no interference peak was observed at the retention time of analyte between them, with each other. For details, refer to Table-1. The specificity representative chromatograms are shown in Fig.-1.

Table-1: Specificity of Pretilachlor

Name of Compounds	Pretilachlor	
	RT (minutes)	Area
Solvent (Acetone)	Nil	Nil
Sample	3.206	9052
Standard	3.207	426287

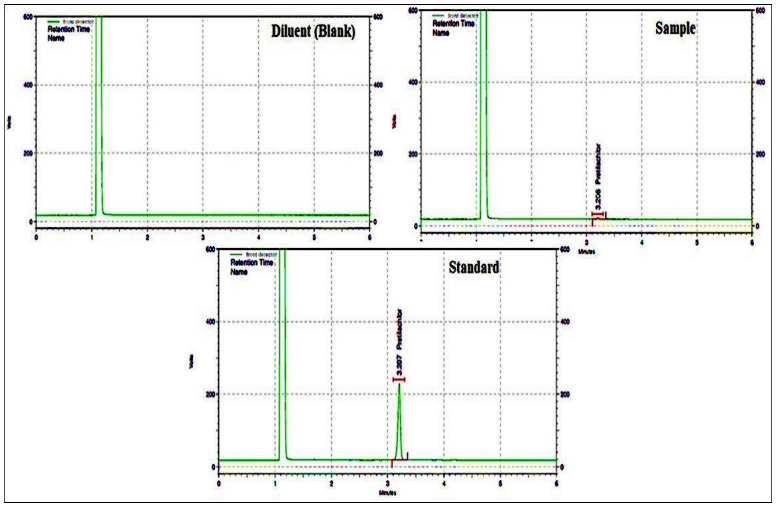

Fig.-1: Representative Specificity Chromatograms of Pretilachlor

Linearity

Different known concentrations of fungicides (0.01, 0.03, 0.5, 1.0, 10.0, and 50.0 µg/mL) were prepared in distinct 10 mL volumetric flasks by diluting the stock solution. The details of the serial dilution and responses were provided in Table-2. These standard solutions were injected directly into a GC. The representative chromatograms are shown in Fig.-2. A calibration curve was plotted to show the relationship between the concentrations of the injected standards and the observed areas, and the linearity of the method was evaluated by analysing six standard solutions. The peak areas obtained from the different concentrations of standards were used to calculate the linear regression equation, which is Y=18968.75X + 28.15, with a correlation coefficient of 1.0000. A calibration curve is shown in Fig.-3.

Table-2: Serial Dilutions for Linearity Standard Solutions and Their Response

Standard Stock (µg/mL)	Volume taken (mL)	Final volume (mL)	Final Concentration (µg/mL)	Std. Area
1.00	0.100	10	0.01	1022
1.00	0.300	10	0.03	2852
10.00	0.500	10	0.5	9042
10.00	1.000	10	1.00	19025
1000.40	0.100	10	10.00	186442
1000.40	0.500	10	50.02	949502

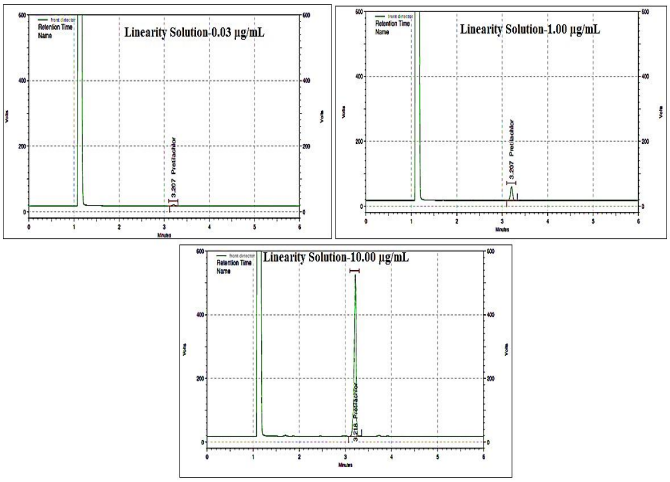

Fig.-2: Representative Linearity Chromatograms of Pretilachlor

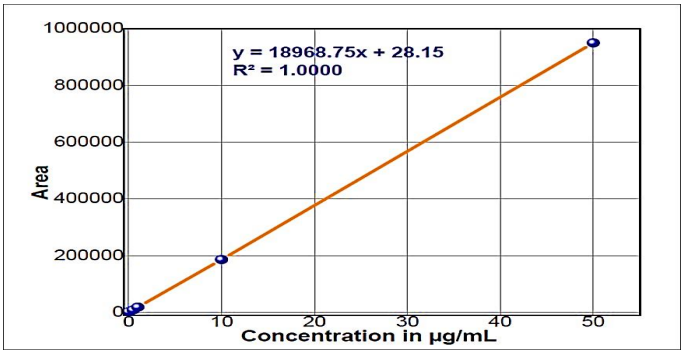

Fig.-3: Calibration Curve of Pretilachlor Standard

Recovery and Repeatability

The validation of the analytical method was conducted to ensure the recovery of the test item at two different concentration levels in acidic, neutral, and basic water.

Preparation of Test Item Stock Solution

An accurate measurement of 6.74 mg of the test item (purity 37.12%) of Pretilachlor was transferred into a 100 mL volumetric flask. The contents were dissolved in 20 mL of acetone, sonicated, and then made up to the mark with the same solvent. This produced a concentration of 25.02 µg/mL solution.

Preparation of 0.03 µg/mL Fortification Level

A 30 µL sample of the test item stock solution, which has a concentration of 25.02 µg/mL, was introduced into 25 mL of groundwater. This was performed in six repetitions.

Preparation of 0.3 µg/mL Fortification Level

A 300 µL aliquot of 25.02 µg/mL test item stock solution was fortified into 25 mL of groundwater. A 300 µL aliquot of a 25.02 µg/mL stock solution of the test item was added to 25 mL of groundwater. This procedure was performed in six replicates. The samples were evaluated for accuracy and repeatability using GC. Accuracy was determined as % recovery, while repeatability was assessed as % RSD, with the results presented in Table-3.

Table-3: Recovery and Repeatability of Pretilachlor 37.12 % EC in Ground Water

Replication	% of Recovery at 0.03 µg/mL Fortification Level	% of Recovery at 0.3 µg/mL Fortification Level
R1	83.26	92.26
R2	85.17	93.18
R3	82.69	95.29
R4	84.15	91.96
R5	83.41	92.66
R6	81.52	94.58
Mean	83.37	93.32
STDEV	1.24	1.33
% of RSD	1.49	1.43

Detection and Quantification Limits

The quantification limit has been established at 0.03 µg/mL. This limit is characterised as the minimum fortification level evaluated that produces acceptable average recoveries (82-85%, RSD<2%). Furthermore, this quantification threshold indicates the fortification level at which an analyte peak is reliably detected at roughly 10 times the baseline noise in the chromatogram. The detection limit was determined to be 0.01 µg/mL, which is equivalent to a level of about three times the background of the control injection at the retention time of the peak of interest.

Dissipation Details of Pretilachlor in Groundwater

On day 0, the initial concentration of Pretilachlor in acidic water was 0.4980 µg/mL for T1 and 0.9970 µg/mL for T2. As shown in the representative chromatograms in Fig.-4, by day 1, the concentrations had

reduced to 0.4074 µg/mL for T1 and 0.8062 µg/mL for T2. The samples from day 2 revealed residue levels of 0.3029 µg/mL for T1 and 0.6105 µg/mL for T2. On day 5, the concentrations were recorded at 0.2171 µg/mL for T1 and 0.4218 µg/mL for T2. By day 10, the samples indicated concentrations of 0.1758 µg/mL for T1 and 0.3485 µg/mL for T2. The day 20 samples showed levels of 0.1113 µg/mL for T1 and 0.2202 µg/mL for T2. On day 30, the concentrations were 0.0852 µg/mL for T1 and 0.1712 µg/mL for T2. The day 40 samples indicated 0.0605 µg/mL for T1 and 0.1209 µg/mL for T2. A complete dissipation of residues to below the detectable level (BDL) was noted on day 50 for both dosages (T1 and T2). The representative chromatograms are shown in Fig.-5.

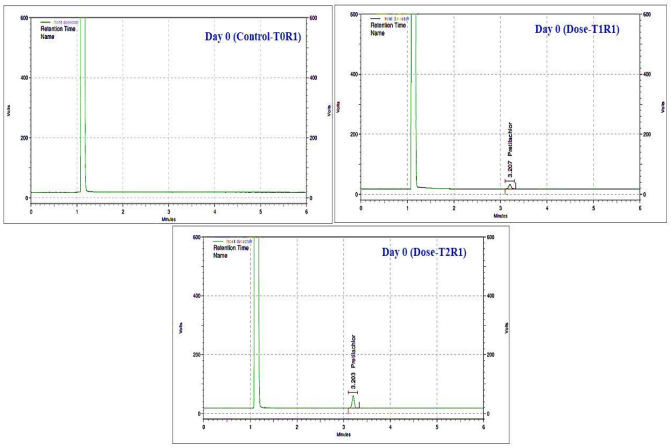

Fig.-4: Representative Day 0 Ccasion Chromatograms of Pretilachlor in Ground Water

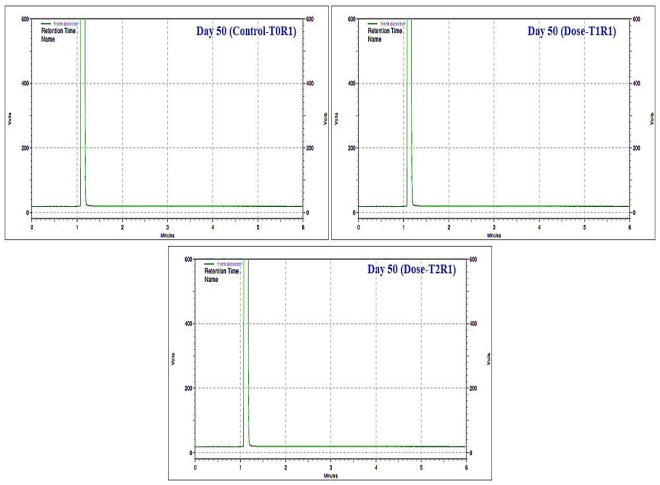

Fig.-5: Representative Day 50 Occasion Chromatograms of Pretilachlor in Ground Water

The dissipation curve, which is plotted to show the correlation between the concentration of the analyte and the sampling occasions, is illustrated in Fig.-6. The DT50 was computed using the following formula:

$$DT50 = \ln 2 / (k)$$

Where 'k' denotes the slope of the curve obtained from the dissipation data. The DT50 values, which represent the time required to degrade 50% of the residues, are detailed in Table-3. The rate constant was calculated using a linear regression equation derived from the first-order rate equation.

$$K = \ln a / (a - x) / dt$$

In this equation, dt refers to the time interval between t1 and t2, while a and x represent the concentrations of pesticides at times t1 and t2, respectively. A plot of the concentration of residues against the rate, with an R2 value, indicates that first-order kinetics govern the dissipation of both fungicides. The DT50 (Half-Life) for Pretilachlor was determined through regression analysis of the dissipation data, with results presented in Table-4.

Table-4: Regression Analysis of Pretilachlor in Ground Water

Occasion (Days)	T1 Concentration (µg/mL)	T2 Concentration (µg/mL)	Log T1 Concentration	Log T2 Concentration
0	0.4980	0.9970	-0.3028	-0.0013
1	0.4074	0.8062	-0.3900	-0.0936
2	0.3029	0.6105	-0.5187	-0.2143
5	0.2171	0.4218	-0.6633	-0.3749
10	0.1758	0.3485	-0.7550	-0.4578
20	0.1113	0.2202	-0.9535	-0.6572
30	0.0852	0.1712	-1.0696	-0.7665
40	0.0605	0.1209	-1.2182	-0.9176
50	0.0000	0.0000	0.0000	0.0000
Intercept			-0.4512	-0.1538
CC			-0.9579	-0.9553
slope			-0.0209	-0.0209
Rate Constant (k)			0.0482	0.0480
t1/2 (DT50) in Days			14.37	14.42

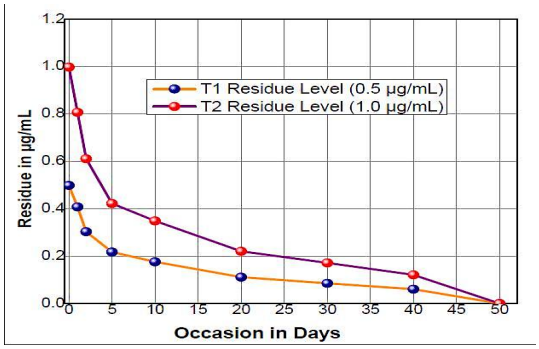

Fig.-6: Dissipation Curve of Pretilachlor in Ground Water

CONCLUSION

This research details a fast and efficient analytical method that employs GC-FID to quantify Pretilachlor residues in groundwater. By using nitrogen as the carrier gas, the method achieved excellent separation and resolution, with a very short analysis time of about 6 minutes for each chromatographic run. Validation parameters, including linearity, recovery, precision, and LOQ, along with DT 50 values, were satisfactorily established by following the guidelines from the Directorate-General for Health and Consumers (DG SANCO) and the Environmental Protection Agency (EPA). As a result, the proposed analytical method and dissipation data could be advantageous for routine monitoring, residue laboratories, and researchers evaluating Pretilachlor residues in various products such as crops, water, and soil samples. The study also investigates the dissipation kinetics of the Pretilachlor herbicide in groundwater, assessing its environmental impact and persistence in aquatic ecosystems. The findings indicate that the DT50 (Half-Life) of Pretilachlor is 14.37 days for the T1 dose and 14.42 days for the T2 dose.

AKNOWLEDGEMENTS

The authors are grateful to the Department of Chemistry at Mahatma Gandhi University, Nalgonda, Telangana - 508 254, for its significant contribution to the successful completion of this research work.

CONFLICT OF INTERESTS

All authors acknowledge that there are no conflicts of interest or financial interests linked to this research work.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-14: Life Below Water*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

K. Kiranmai https://orcid.org/0009-0009-1327-2383
V. Sridhar https://orcid.org/0000-0001-6089-2336
Tentu Nageswara Rao http://orchid.org/0000-0001-7623-3343
Y. Prashanthi https://orcid.org/0009-0004-5397-050X

Cite this article as: K. Kiranmai *et al.*, *Rasayan J. Chem.*, 19(3), 1374-1381(2026), https://doi.org/10.31788/RJC.2026.1939752

REFERENCES

1. A. Alaoui, F. Christ, N. Abrantes, V. Silva, N. Gonz, D. Torre, *Environmental Pollution,* **1**, 363(2024), https://doi.org/10.1016/j.envpol.2024.125056
2. V.M.Pathak, V.K.Verma, B.S. Rawat, B. Kaur, N. Babu, *A Comprehensive Review,* **2**, 1(2022), https://doi.org/10.3389/fmicb.2022.962619
3. I. Meftaul, K.Venkateswarlu, *Environmental Geochemistry and Health*, **45**, 1599(2023), https://doi.org/10.1007/s10653-022-01278-w
4. M. Tudi, H.D. Ruan, L.Wang, J. Lyu, R. Sadler, D. Connell, *International Journal of Environmental Research and Public Health*, **18,** 124(2021), https://doi.org/10.3390/ijerph18031112
5. G. Araya, A.P. Bolaño, M. Sandoval, A. Araneda, R.O. Barra, *A Historic Review and Critical Analysis* **43,** 2478(2024), https://doi.org/10.1002/etc.5869
6. A. Schaeffer, C. Wijntjes, *Eco-Environment & Health,* **1**, 23(2022), https://doi.org/10.1016/j.eehl.2022.02.002
7. W. Zhou, M. Li, V. A. Achal, *Emerging Contaminants*, **11**, 100410(2025), https://doi.org/10.1016/j.emcon.2024.100410
8. M.M. Banu, N. Reehana, M.M. Imran, *A Comprehensive review of Mechanisms , Factors and Biodiversity*, **4,** 65(2024), https://doi.org/10.37394/232023.2024.4.8
9. F. Ahmad, F. Ali, A.A. Alsayegh, A.M. Alshahrani, K. Muzammil, A. Ali , *Heliyon*, **10,** e29128 (2024), https://doi.org/10.1016/j.heliyon.2024.e29128
10. S. Shekhar, *A paradigm,* **8**, 6569(2021), https://doi.org/10.1039/d1ma00329a.
11. S. Mahalle, R.S. Bhende, P. Bokade, A. Bajaj, *Total Environment Microbiology*, **1,**100026(2025), https://doi.org/10.1016/j.temicr.2025.100026
12. M. Aljabri, *Environmental Pollutants and Bioavailability,* **37,** 103(2025), https://doi.org/10.1080/26395940.2025.2554173
13. W. Aktar, D. Sengupta, A. Chowdhury, *Interdisciplinary Toxicology*, **2**, 1(2009), https://doi.org/10.2478/v10102-009-0001-7
14. B.S. Chauhan, S. Tran, T. Ngoc, D. Duong, P. Ngoc Le, *Plant Production Science* **17 (4)**, 315(2014), https://doi.org/10.1626/pps.17.315 2014;17:315–20
15. M. Dilipkumar, M.A. Adzemi, T..S Chuah, *Weed Science*, **2**, 126(2012), https://doi.org/10.1614/WS-D-11-00075.1

[RJC-9752/2026]