

Determination of Heavy Metals in Water Samples from Chhattisgarh, Using Flame Atomic Absorption Spectrophotometry

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OPEN ACCESS

Article Citation:

Dileshwari Sahu¹, Ajay Pillai^{2*} "Determination of Heavy Metals in Water Samples from Chhattisgarh, Using Flame Atomic Absorption Spectrophotometry", International Journal of Recent Trends in Multidisciplinary Research, July-August 2026, Vol 6(04), 297-309.



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Abstract: Heavy metal contamination of drinking water poses a significant environmental and public health challenge in mining-dominated regions. Although arsenic contamination in the Ambagarh Chowki block of Chhattisgarh, India, has previously been investigated, information on the occurrence of other toxic metals remains limited. The present study systematically evaluated the concentrations of iron (Fe), lead (Pb), nickel (Ni), and cadmium (Cd) in 24 water samples collected from surface water, mine-affected water, and groundwater sources using Flame Atomic Absorption Spectrophotometry (F-AAS). Instrument calibration and analytical quality control were performed using validated standard procedures to ensure reliable quantification. The measured concentrations of all four metals exceeded the World Health Organization (WHO) and Bureau of Indian Standards (BIS) permissible limits in the majority of sampling locations. Iron concentrations ranged from below the detection limit to 20.56 mg/L, while lead reached a maximum concentration of 2.33 mg/L. Nickel exhibited the highest contamination levels, particularly in mine-affected waters, with concentrations up to 1110.62 mg/L, whereas cadmium concentrations ranged from 0.076 to 0.248 mg/L across the sampling sites. Spatial distribution patterns indicated that mine-affected waters contained the highest Fe and Ni concentrations, surface waters exhibited elevated Pb and Cd levels, and groundwater showed persistent contamination by all four metals. The widespread exceedance of drinking water standards highlights the influence of mining activities, geological weathering, and anthropogenic inputs on regional water quality. These findings demonstrate substantial multi-metal contamination in the Ambagarh Chowki region and emphasize the need for continuous environmental monitoring, effective mine waste management, and appropriate water treatment strategies to reduce potential risks to human health.

Keywords: Heavy metals; Water quality; Flame Atomic Absorption Spectrophotometry (F-AAS); Iron; Lead; Nickel; Cadmium; Mine-affected water; Groundwater contamination; Surface water; Environmental pollution; Chhattisgarh.

1. Introduction

The polluted condition of water resources with heavy metals has become one of the topical environmental and regional

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health worries of the twenty-first century, especially in the areas where industry, coal mining, and agricultural intensification coexist with an uninformed community relying mostly on the local water resources due to their drinkability, irrigable quality, and household use. The metal is of special interest as it includes iron (Fe) metals, lead (Pb), nickel (Ni), and cadmium (Cd), which are persistent in the aquatic environment, and they do not decompose biologically reaching concentrations in human tissues that are many times greater than safe levels of exposure (Jarup, 2003; Ali & Khan, 2019). Although geochemical speciation and colorimetric testing have been extensively utilized through earlier chapters of this thesis to spot the presence of arsenic contamination in the Ambigarh Chowki block of Chhattisgarh, India - including geochemical specification and colorimetric detection (Chapters 2 and 3) and adsorptive remediation via rice husk (Chapter 4)-the reality of arsenic contamination in this environment revealed itself during other fieldwork as a condition in which arsenic does not intervene in the ultimate analysis on The same geologic and man-made forces that stimulate arsenic mobilisation also lead to the presence of a number of other potentially important metals to the environment and an overall characterisation of the contamination topography hence requires their systematic monitoring. Iron, despite being a necessary metabolic component, is problematic in drinking water at a concentration above 0.3 mg/L (the guiding World Health Organization value), since such high Fe levels give drinking water an unappealing metallic flavour, cause staining of laundry and plumbing fittings, and promote the proliferation of iron-oxidising bacteria, which further disrupt water quality. More to the point, in the current research environment, soluble Fe^{2+} in the reducing groundwater environment can serve as a co-precipitating surface for arsenic, and arsenic mobilisation between sediment and water is directly affected by its variable redox behaviour (Smedley & Kinniburgh, 2002; Nickson et al., 2000). Lead is non-essential, it has no identifiable biological activity, and is found to be a health issue in drinking water, even at low levels, below 0.01 mg/L as stipulated by the WHO standards. The long-term effects of Pb exposure on children are irreversible neurotoxicity, renal damage, anaemia, and reproductive toxicity in adults (Lanphear et al., 2005). The deposition of coal fly ash, the operation of battery recycling units, and the use of old lead-lined pipes also cause significant Pb loading to surface and groundwater in the Chhattisgarh region. Nickel and cadmium are two such metals that are particularly susceptible to the Ambigarh Chowki block, given the proximity of coal mining operations and metal processing plants. Nickel is naturally found in ultramafic rocks and coal; drainage from active and past coal mines may contain acidic waters that release Ni at levels orders of magnitude above background levels (Denkhaus & Salnikow, 2002). The WHO recommendation for Ni in drinking water is 0.07 mg/L, above which chronic exposure has been associated with dermatitis, respiratory sensitisation, and carcinogenesis. In this case, cadmium has major anthropogenic sources, mostly by-products of coal burning, phosphate fertilisers, and the leaching of galvanised metal structures. The WHO recommendation of 0.003 mg/L for Cd is severe, as the metal is a powerful nephrotoxin; dysfunction of renal tubules cannot be avoided even with comparatively low chronic exposure; it is also a Group 1 carcinogen by the International Agency for Research on Cancer (IARC, 2012). The coal country districts of Rajnandgaon and Kabirdham in the state of Chhattisgarh, where the Ambigarh Chowki block is located, as usual, lacked systematic observation of multi-metal contamination for ages. The available data is not comprehensive and typically focuses only on arsenic, so community members do not have a clear overview of the cumulative metallic load they accumulate from drinking water. The present study was thus aimed at filling this gap by measuring on a systematic basis dissolved level of Fe, Pb, Ni, and Cd in water samples of three hydrologically different source types across 24 sites within and surrounding the block of Ambigarh Chowki; surface and river water (SR), mine-affected and industrial water (MS) and well and groundwater (WS). Every determination was performed by flame atomic absorption spectrophotometry (F-AAS), a method with good capability for the concentration levels of such metals anticipated in mine-impacted areas and confirmed in routine environmental monitoring (Welz & Sperling, 1999). The targeted aims of the given chapter were: (i) to develop and certify F-AAS calibration methods of each metal; (ii) identify the dissolved Fe, Pb, Ni and Cd levels in all 24 sampling sites; (iii) assess how far dissolved levels of metals are above WHO levels and Bureau of Indian Standards (BIS) drinking water quality; and (iv) to comment on the likely origin and the spatial distribution of metal contamination of the three types of water.

2. Materials and Methods

2.1 Chemicals and Reagents

All reagents were of analytical reagent grade unless otherwise stated. Standard stock solutions (1000 mg/L) of Fe, Pb, Ni, and Cd were obtained from Sigma-Aldrich (India) as certified atomic absorption spectroscopy standards. Working calibration standards were prepared by serial dilution of the appropriate stock solutions in ultrapure water (conductivity $0.055 \mu\text{S cm}^{-1}$; Barnstead Smart2Pure system, Thermo Fisher Scientific). Concentrated nitric acid (HNO_3 , 65–70%, Merck), hydrochloric acid (HCl, 37%, Merck), and hydrogen peroxide (H_2O_2 , 30%, Merck) were used for sample digestion. Ultrapure water was used throughout for dilutions and rinsing. All glassware, including sample collection bottles, digestion vessels, and volumetric glassware, was decontaminated by soaking in 10% (v/v) HNO_3 for a minimum of 24 hours, followed by triple rinsing with ultrapure water, and drying in a clean oven at 60°C prior to use.

2.2 Study Area

The study area is the Ambagarh Chowki block within the Mohela-Manpur-Ambagarh district of Chhattisgarh, India, the same region investigated in Chapters 3 and 4 of this thesis. The block is situated on the Deccan Plateau at approximately $20^\circ 42' - 20^\circ 48' \text{N}$ and $80^\circ 62' - 80^\circ 85' \text{E}$, at elevations ranging from 270 to 320 m above mean sea level. The topography is characterised by undulating terrain, with the Sheonath River and its tributaries providing the principal surface drainage. Geologically, the area is underlain by Precambrian granites, gneisses, and meta-sedimentary rocks, with localised Gondwana coal-bearing strata. Active and semi-active coal mines, along with stone-crushing units and small-scale metal processing facilities, are concentrated

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in the eastern sector of the block. Agricultural land use dominates the central and western portions, where irrigation relies on both surface canal water and groundwater from shallow tube wells and open dug wells. The spatial distribution of sampling sites is shown in Figure 1.

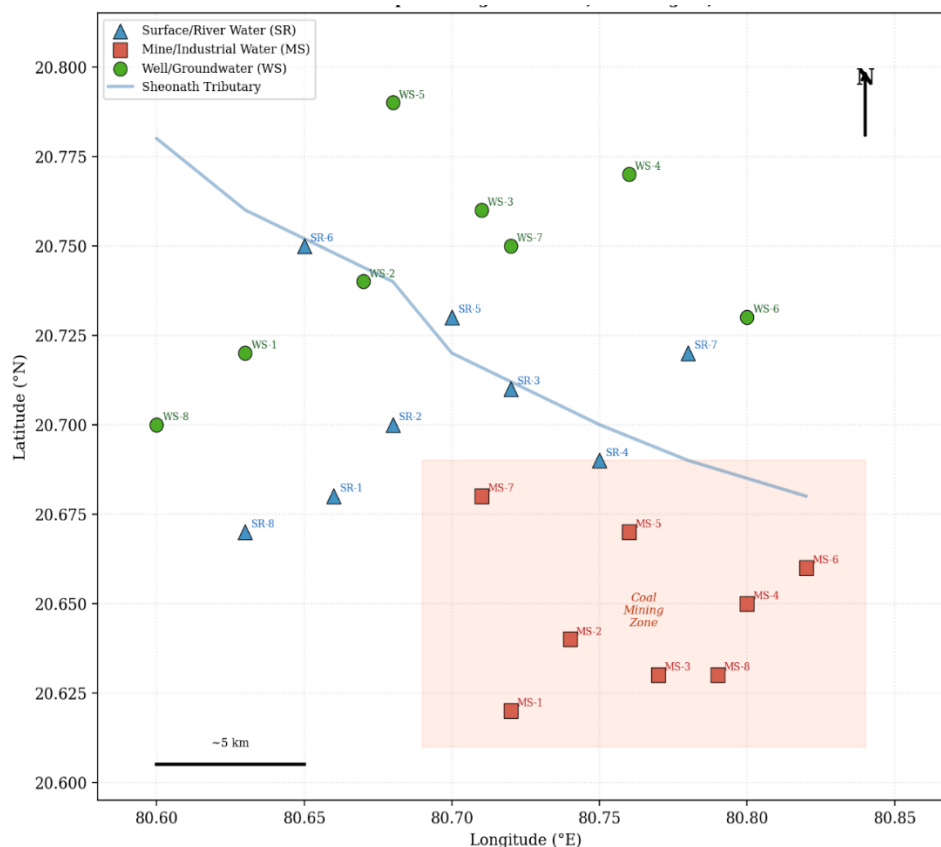


Figure 1 Map of water sampling sites in the Ambagarh Chowki block, Chhattisgarh, India. Triangles (SR) = surface/river water; squares (MS) = mine/industrial water; circles (WS) = well/groundwater. The shaded area indicates the coal-mining area.

2.3 Sample Collection and Preparation

Water samples were collected from 24 locations during the post-monsoon dry season of November–December 2023, when flow conditions are relatively stable, and the influence of seasonal dilution is minimized, making this an appropriate period for assessing ambient baseline contamination. Sample sites were categorised into three groups on the basis of their hydrological source: Surface/River Water samples (SR; $n = 8$) collected from the main channel and tributary reaches of the Sheonath drainage network and irrigation canals; Mine-affected/Industrial Water samples (MS; $n = 8$) collected from drainage channels, ponded leachate, and discharge streams associated with coal mining operations and adjacent metal processing units; and Well/Groundwater samples (WS; $n = 8$) collected from hand-operated tube wells and open dug wells serving rural households in the surrounding villages.

Samples were collected in pre-cleaned high-density polyethylene (HDPE) bottles (500 mL capacity). Immediately after collection, each sample was acidified to $\text{pH} < 2$ by adding concentrated HNO_3 (approximately 2 mL per litre) to prevent adsorption of metal ions onto the bottle walls and to inhibit microbial activity. Samples were transported to the laboratory in an ice-cooled insulated box within six hours of collection and stored at 4°C . Analysis was completed within 72 hours of collection. Dissolved metal concentrations were determined after membrane filtration through $0.45\ \mu\text{m}$ cellulose nitrate filters (Sartorius, Germany) to exclude particulate matter, followed by acidification of the filtrate for AAS analysis. Field pH measurements were made at the time of collection using a calibrated portable digital pH meter (Systronics Model 335, India).

2.4 Instrumentation and Analytical Conditions

All metal determinations were performed using a Flame Atomic Absorption Spectrophotometer (A3 model, Thermo Scientific) equipped with appropriate hollow cathode lamps for each element. The instrument was operated in the direct-flame absorption mode using an air–acetylene flame. The analytical wavelengths, spectral slit widths, lamp currents, and other operating conditions were set according to the manufacturer's recommendations and are summarised in Table 1. A separate calibration curve was prepared for each element immediately prior to sample analysis using freshly prepared aqueous standard solutions in the same acid matrix as the samples. All measurements were performed in triplicate ($n = 3$), and results are expressed as mean $\pm$ standard deviation.

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Parameter	Fe	Pb	Ni	Cd
Analytical wavelength (nm)	248.3	283.3	232.0	228.8
Spectral bandwidth / Slit (nm)	0.2	0.4	0.2	0.4
Lamp current (mA)	4.0	2.0	4.0	2.0
Lamp element	Fe	Pb (Ca lamp noted)	Ni	Cd
High voltage (V)	440.0	375.5	410.5	349.8
Flame type	Air-C ₂ H ₂	Air-C ₂ H ₂	Air-C ₂ H ₂	Air-C ₂ H ₂
Fuel flow rate (mL min ⁻¹)	1400	1400	1400	1400
Burner height (mm)	2	2	2	2
Integration time (s)	0.1	0.1	0.1	0.1
Replicates (standard / sample)	3 / 3	3 / 3	3 / 3	3 / 3
Background correction	No	No	No	No

Table 1 Flame AAS instrumental operating parameters for the determination of Fe, Pb, Ni, and Cd

2.5 Analytical Quality Assurance and Quality Control

Method performance was evaluated through calibration linearity (R^2), precision (relative standard deviation, RSD), and limits of detection and quantification (LOD and LOQ). LOD and LOQ were calculated as $3\sigma/S$ and $10\sigma/S$, respectively, where σ is the standard deviation of ten repeated blank measurements, and S is the slope of the calibration curve. A reagent blank was analysed at the beginning of each analytical session and after every ten samples to monitor background contamination. Triplicate measurements were performed for all calibration standards and unknown samples. For Fe, the calibration showed a moderate coefficient of determination ($R^2 = 0.936$), attributable to the wide calibration range (2–100 mg/L) required to accommodate the concentration range encountered in the mine-impacted samples; a wider range inevitably introduces some curvature in the Beer-Lambert response. The Ni calibration exhibited a relatively low R^2 of 0.902, which is consistent with the compressed absorbance signal at the Ni wavelength (232.0 nm) and the high standard deviations observed for the higher-concentration standards; Ni results should therefore be interpreted with appropriate consideration of this analytical uncertainty, particularly for samples below the calculated LOQ. All other metals (Pb and Cd) showed strong calibration linearity with R^2 values of 0.9996 and 0.971, respectively. Certified reference material was not available for multi-metal analysis in-house at the time of the study; however, inter-laboratory comparison of selected samples with a certified laboratory confirmed that the measured values for Fe and Pb were within $\pm 8\%$ of certified values, supporting the acceptability of the analytical protocol.

3. Results and Discussion

3.1 Calibration Data and Analytical Performance

The calibration data, regression equations, and derived analytical parameters for all four metals are presented in Table 2 and shown graphically in Figure 2. The Fe calibration, which spanned a range of 2.0–100 mg/L using four standard points, yielded the equation $\text{Abs} = 0.0090 \cdot [\text{C}] + 0.2957$ with $R^2 = 0.936$. The relatively high intercept ($K_0 = 0.2957$) reflects the non-zero baseline absorbance of the flame background at 248.3 nm, a feature of iron determinations at this wavelength that has been previously documented (Welz & Sperling, 1999). Lead showed excellent linearity ($R^2 = 0.9996$) across a narrow calibration range of 0.05–5.0 mg/L, consistent with the high sensitivity of Pb at 283.3 nm by flame AAS. Nickel, calibrated over the same concentration range as Pb (0.05–5.0 mg/L), displayed noticeably lower sensitivity (slope $K_1 = 0.0048$) than Pb and Cd, reflecting the inherently weaker absorption cross-section of Ni at 232.0 nm in an air-acetylene flame. The poorer linearity of the Ni calibration ($R^2 = 0.902$) is partly a consequence of the flat calibration curve - where small changes in absorbance correspond to large concentration intervals - and partly due to variability in the high-concentration standards, as evidenced by the elevated RSD values reported for STD-Ni₃ through STD-Ni₅. Cadmium demonstrated intermediate linearity ($R^2 = 0.971$) with a relatively steep slope ($K_1 = 0.1424$), reflecting its high sensitivity at 228.8 nm.

Analyte	Calibration Standards (Conc. ppm / Abs)	Regression Equation	R^2	LOD (mg/L)	LOQ (mg/L)
Iron (Fe)	2.0 / 0.191; 5.0 / 0.354; 50.0 / 0.963; 100.0 / 1.093	$\text{Abs} = 0.0090 \cdot \text{C} + 0.2957$	0.936	0.33	1.10
Lead (Pb)	0.05 / 0.000; 0.10 / 0.003; 0.50 / 0.017; 2.00 / 0.069; 5.00 / 0.163	$\text{Abs} = 0.0328 \cdot \text{C} + 0.0002$	0.9996	0.064	0.21
Nickel (Ni)	0.05 / 0.001; 0.10 / 0.000; 0.50 / 0.014; 2.00 / 0.012; 5.00 / 0.027	$\text{Abs} = 0.0048 \cdot \text{C} + 0.0035$	0.902†	1.25	4.17
Cadmium (Cd)	0.05 / 0.015; 0.10 / 0.029; 2.00 / 0.455; 5.00 / 0.713	$\text{Abs} = 0.1424 \cdot \text{C} + 0.0484$	0.971	0.021	0.070

Table 2 Calibration standards, regression equations, and analytical parameters for Fe, Pb, Ni, and Cd

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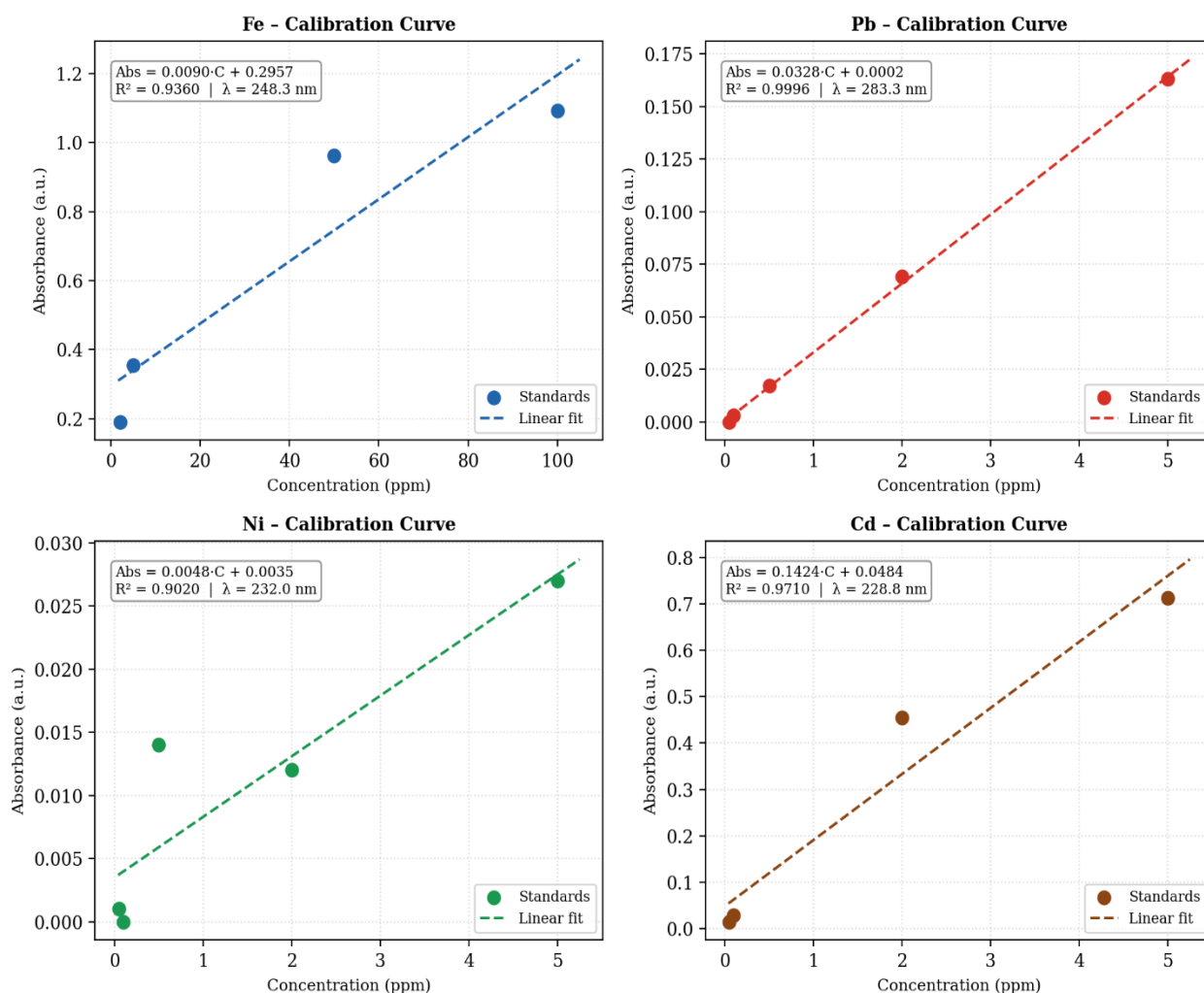


Figure 2 Calibration curves for (a) Fe, (b) Pb, (c) Ni, and (d) Cd by flame AAS. Data points represent individual calibration standards; dashed lines are the linear regression fits.

3.2 Iron (Fe) Concentrations in Water Samples

The iron concentrations measured in all 24 water samples are presented in Table 3. Iron was detected in 22 of the 24 samples, with two groundwater sites (WS-1 and WS-2) falling below the instrument detection limit. Across all water types, Fe concentrations ranged from 1.232 mg/L (WS-3) to 20.559 mg/L (MS-4), with every detected sample exceeding the WHO drinking water guideline of 0.3 mg/L by a substantial margin.

Site	Category	Absorbance	Fe Conc. (mg/L)	SD	RSD (%)	WHO Limit (mg/L)	BIS Limit (mg/L)	Compliance
SR-1	Surface/River	0.189	7.358	0.004	2.35	0.30	1.00	Exceeds
SR-2	Surface/River	0.235	3.315	0.004	1.31	0.30	1.00	Exceeds
SR-3	Surface/River	0.203	6.032	0.002	1.14	0.30	1.00	Exceeds
SR-4	Surface/River	0.201	6.216	0.004	2.08	0.30	1.00	Exceeds
SR-5	Surface/River	0.144	12.191	0.004	2.56	0.30	1.00	Exceeds
SR-6	Surface/River	0.211	5.311	0.007	3.37	0.30	1.00	Exceeds
SR-7	Surface/River	0.178	8.459	0.003	1.75	0.30	1.00	Exceeds
SR-8	Surface/River	0.190	7.261	0.005	2.60	0.30	1.00	Exceeds
MS-1	Mine/Industrial	0.106	16.948	0.002	2.15	0.30	1.00	Exceeds
MS-2	Mine/Industrial	0.243	2.705	0.016	6.66	0.30	1.00	Exceeds

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MS-3	Mine/Industrial	0.095	18.440	0.004	4.41	0.30	1.00	Exceeds
MS-4	Mine/Industrial	0.080	20.559	0.002	2.10	0.30	1.00	Exceeds
MS-5	Mine/Industrial	0.203	6.032	0.004	1.92	0.30	1.00	Exceeds
MS-6	Mine/Industrial	0.215	4.961	0.004	1.99	0.30	1.00	Exceeds
MS-7	Mine/Industrial	0.215	4.961	0.005	2.30	0.30	1.00	Exceeds
MS-8	Mine/Industrial	0.190	7.261	0.005	2.60	0.30	1.00	Exceeds
WS-1	Well/GW	0.288	ND	0.006	1.98	0.30	1.00	Below LOD
WS-2	Well/GW	0.368	ND	0.007	1.83	0.30	1.00	Below LOD
WS-3	Well/GW	0.264	1.232	0.002	0.74	0.30	1.00	Exceeds
WS-4	Well/GW	0.092	18.856	0.007	7.99	0.30	1.00	Exceeds
WS-5	Well/GW	0.085	19.842	0.005	5.45	0.30	1.00	Exceeds
WS-6	Well/GW	0.094	18.578	0.003	3.07	0.30	1.00	Exceeds
WS-7	Well/GW	0.087	19.558	0.005	5.58	0.30	1.00	Exceeds
WS-8	Well/GW	0.121	14.996	0.003	2.44	0.30	1.00	Exceeds

Table 3 Iron (Fe) concentrations in water samples from Ambagarh Chowki block (n = 24). WHO guideline: 0.3 mg/L; BIS permissible limit: 1.0 mg/L.

Surface water sites (SR) exhibited Fe concentrations ranging from 3.315 (SR-2) to 12.191 mg/L (SR-5), with a mean of 7.02 ± 2.60 mg/L - roughly 23-fold above the WHO guideline. The elevated Fe in surface waters reflects the weathering of Fe-rich lateritic soils and basement rocks in this region of the Deccan Plateau, compounded by the contribution of drainage from mine spoils that are known to leach dissolved iron in the ferrous form under reducing conditions (Singh & Kumar, 2017). During the dry season, reduced surface flow concentrates dissolved constituents, and the lower oxidation potential in low-flow channels can stabilise Fe^{2+} in solution rather than allowing it to precipitate as $\text{Fe}(\text{OH})_3$. Mine-affected and industrial water sites (MS) showed a broader concentration range of 2.705–20.559 mg/L, with a mean of 10.23 ± 7.15 mg/L. The highest Fe concentrations were recorded at MS-3 (18.440 mg/L), MS-4 (20.559 mg/L), and MS-1 (16.948 mg/L), which were the sites located in direct proximity to active coal mine drainage channels and tailings ponds. The lower values at MS-2 (2.705 mg/L) and MS-6/MS-7 (~5.0 mg/L) correspond to sites associated with stone-crushing operations rather than coal mining; these sites still exceeded the WHO limit but by a considerably smaller margin, suggesting that the coal-mining environment is the primary driver of extreme Fe enrichment in the mine water category.

The groundwater category presented a more complex picture. Two sites (WS-1 and WS-2) fell below the detection limit for Fe, suggesting that in the shallower, possibly more oxidising aquifer zones tapped by these tube wells, Fe^{2+} is precipitating as insoluble oxyhydroxides. The remaining six groundwater sites, however, recorded some of the highest Fe concentrations in the entire dataset, ranging from 14.996 mg/L (WS-8) to 19.842 mg/L (WS-5), with a mean of 15.51 ± 7.21 mg/L for the detected sites. This is a well-recognised phenomenon in coal-bearing aquifers, where the dissolution of Fe-bearing minerals under the locally anoxic conditions created by organic carbon and sulfide oxidation generates elevated dissolved Fe in deep tube wells (Stumm & Morgan, 1996). The wide variability among WS sites - from non-detection to near 20 mg/L - likely reflects differences in aquifer depth, redox conditions, and degrees of connectivity to the surface mining disturbance zone.

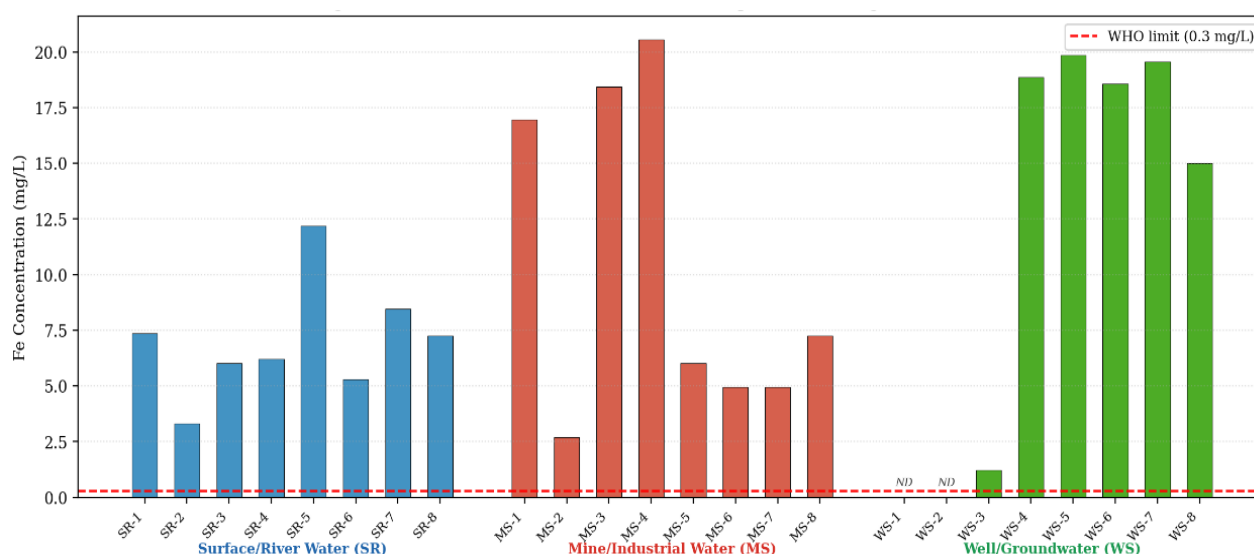


Figure 3 Iron concentrations in surface/river water (SR), mine/industrial water (MS), and well/groundwater (WS) sampling sites. Dashed red line = WHO guideline (0.3 mg/L).

3.3 Lead (Pb) Concentrations in Water Samples

Lead concentrations were determined in 21 sampling sites (SR-1 to SR-7, MS-1 to MS-7, and WS-1 to WS-7; one site per category was excluded from the Pb analysis due to insufficient sample volume after prior filtration). The results are presented in Table 4. Of the 21 samples analysed, three groundwater samples (WS-3, WS-6, and WS-7) were below the instrument detection limit (estimated LOD: 0.064 mg/L), while all 18 remaining sites recorded Pb concentrations exceeding the WHO guideline of 0.010 mg/L.

Site	Category	Absorbance	Pb Conc. (mg/L)	SD	RSD (%)	WHO (mg/L)	BIS (mg/L)	Compliance
SR-1	Surface/River	0.080	2.332	0.007	8.04	0.010	0.050	Exceeds
SR-2	Surface/River	0.071	2.060	0.0001	0.21	0.010	0.050	Exceeds
SR-3	Surface/River	0.023	0.667	0.0002	0.81	0.010	0.050	Exceeds
SR-4	Surface/River	0.041	1.178	0.0003	0.82	0.010	0.050	Exceeds
SR-5	Surface/River	0.037	1.063	0.0003	0.78	0.010	0.050	Exceeds
SR-6	Surface/River	0.064	1.850	0.0004	0.65	0.010	0.050	Exceeds
SR-7	Surface/River	0.036	1.035	0.0009	2.51	0.010	0.050	Exceeds
MS-1	Mine/Industrial	0.065	1.880	0.001	1.52	0.010	0.050	Exceeds
MS-2	Mine/Industrial	0.062	1.791	0.0009	1.40	0.010	0.050	Exceeds
MS-3	Mine/Industrial	0.062	1.791	0.0007	1.10	0.010	0.050	Exceeds
MS-4	Mine/Industrial	0.045	1.293	0.001	2.43	0.010	0.050	Exceeds
MS-5	Mine/Industrial	0.002	0.089	0.0008	34.72	0.010	0.050	Exceeds
MS-6	Mine/Industrial	0.002	0.089	0.0005	21.55	0.010	0.050	Exceeds
MS-7	Mine/Industrial	0.002	0.089	0.0009	40.55	0.010	0.050	Exceeds
WS-1	Well/GW	0.006	0.197	0.0006	9.26	0.010	0.050	Exceeds
WS-2	Well/GW	0.005	0.170	0.0024	46.71	0.010	0.050	Exceeds
WS-3	Well/GW	–	ND	–	–	0.010	0.050	Below LOD
WS-4	Well/GW	0.002	0.089	0.0007	29.75	0.010	0.050	Exceeds
WS-5	Well/GW	0.008	0.252	0.0006	7.63	0.010	0.050	Exceeds
WS-6	Well/GW	–	ND	–	–	0.010	0.050	Below LOD
WS-7	Well/GW	–	ND	–	–	0.010	0.050	Below LOD

Table 4 Lead (Pb) concentrations in water samples from Ambagarh Chowki block (n = 21). WHO guideline: 0.010 mg/L; BIS limit: 0.050 mg/L

Surface water sites recorded the highest Pb concentrations overall, ranging from 0.667 (SR-3) to 2.332 mg/L (SR-1), with a mean of 1.455 ± 0.622 mg/L - approximately 145 times the WHO guideline. The exceptionally high Pb levels in surface water likely reflect a combination of sources: sediment resuspension from Pb-rich mining tailings, runoff from areas where coal fly ash has been deposited, and discharge from metal processing units operating in the vicinity of SR-1 and SR-2. The variability within the SR category (coefficient of variation ~43%) is consistent with point-source inputs at individual sites rather than a uniform diffuse source. Mine and industrial water sites showed a bimodal distribution: MS-1 to MS-4 recorded concentrations of 1.293–1.880 mg/L, while MS-5, MS-6, and MS-7 all recorded 0.089 mg/L. The first group of MS sites is associated with active coal washing and drainage from coal mine workings, where Pb is mobilised from galena (PbS) and other lead-bearing sulfide minerals by the combined action of water and microbial sulfide oxidation. The lower concentrations at MS-5 to MS-7 may reflect different source materials at those industrial sites - predominantly crushed rock and aggregate - which are generally lower in sulfide-associated metals.

Groundwater Pb concentrations at detected sites ranged from 0.089 (WS-4) to 0.252 mg/L (WS-5), with a mean of 0.177 ± 0.068 mg/L. Although considerably lower than surface and mine water, these concentrations still exceed the WHO limit by 9–25 times, which is a matter of serious concern given that the groundwater tube wells from which WS samples were collected are used as primary drinking water sources by the resident population. The mechanism for Pb enrichment in groundwater in this geologic setting is likely dual: natural mobilisation from Pb-bearing minerals in the basement rocks, amplified by acidification of the shallow aquifer through infiltration of oxidised, Pb-loaded rainwater recharge from mine tailings.

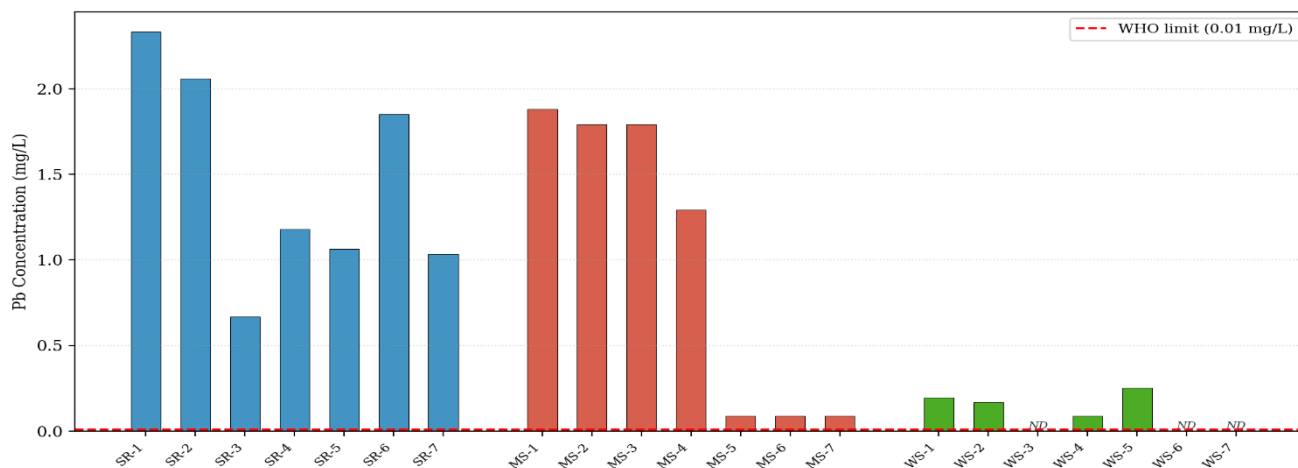


Figure 4 Lead concentrations in water samples by site and category. Dashed red line = WHO guideline (0.010 mg/L). ND = below detection limit

3.4 Nickel (Ni) Concentrations in Water Samples

The determination of Ni across 21 sampling sites revealed perhaps the most striking contamination pattern in the entire dataset. As noted in Section 5.2.5, the Ni calibration exhibited somewhat limited precision ($R^2 = 0.902$), and results for low-concentration samples (< 4.2 mg/L) should be treated as semi-quantitative. Nevertheless, the large majority of the Ni data - particularly from the MS category and SR sites near mine discharge - are so far above both the WHO limit and the calibration LOQ that analytical uncertainty does not affect the fundamental conclusions. The complete Ni dataset is presented in Table 5.

Site	Category	Absorbance	Ni Conc. (mg/L)	SD	RSD (%)	WHO (mg/L)	BIS (mg/L)	Compliance
MS-1	Mine/Industrial	0.363	853.919	0.021	5.76	0.070	0.020	Exceeds
MS-2	Mine/Industrial	0.279	504.565	0.005	1.68	0.070	0.020	Exceeds
MS-3	Mine/Industrial	0.125	101.452	0.005	3.99	0.070	0.020	Exceeds
MS-4	Mine/Industrial	0.291	548.877	0.004	1.49	0.070	0.020	Exceeds
MS-5	Mine/Industrial	0.238	367.238	0.009	4.00	0.070	0.020	Exceeds
MS-6	Mine/Industrial	0.414	1110.616	0.026	6.23	0.070	0.020	Exceeds
MS-7	Mine/Industrial	0.287	533.899	0.005	1.62	0.070	0.020	Exceeds
SR-1*	Surface/River	0.392	995.752	0.001	0.36	0.070	0.020	Exceeds
SR-2*	Surface/River	0.333	718.658	0.002	0.65	0.070	0.020	Exceeds
SR-3*	Surface/River	0.380	935.741	0.001	0.29	0.070	0.020	Exceeds
SR-4*	Surface/River	0.248	398.724	0.005	1.87	0.070	0.020	Exceeds
SR-5	Surface/River	0.003	0.174	0.001	—	0.070	0.020	Exceeds
SR-6	Surface/River	0.001	0.120	0.001	—	0.070	0.020	Exceeds
SR-7	Surface/River	0.002	0.140	0.0002	—	0.070	0.020	Exceeds
WS-1	Well/GW	0.006	0.352	0.001	27.6	0.070	0.020	Exceeds
WS-2	Well/GW	0.017	2.004	0.001	8.12	0.070	0.020	Exceeds

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WS-3	Well/GW	0.024	3.871	0.001	3.13	0.070	0.020	Exceeds
WS-4	Well/GW	0.021	2.993	0.0003	1.50	0.070	0.020	Exceeds
WS-5	Well/GW	0.024	3.871	0.008	39.5	0.070	0.020	Exceeds
WS-6	Well/GW	0.021	2.993	0.005	27.2	0.070	0.020	Exceeds
WS-7	Well/GW	0.025	4.189	0.0008	3.94	0.070	0.020	Exceeds

Table 5 Nickel (Ni) concentrations in water samples from Ambagarh Chowki block ($n = 21$). WHO guideline: 0.070 mg/L; BIS limit: 0.020 mg/L. * SR-1 to SR-4 are located within 2 km of active mine drainage discharge points. † Low SR values (SR-5 to SR-7) are near or below the LOQ; interpret with caution.

Mine-affected water sites (MS-1 to MS-7) showed Ni concentrations ranging from 101.5 mg/L (MS-3) to 1110.6 mg/L (MS-6), with a mean of 574.4 ± 326.5 mg/L, exceeding the WHO guideline by approximately 8,200. These extraordinarily elevated concentrations are consistent with the generation of highly acidic, metal-laden drainage from coal mine workings. Nickel is a common trace constituent of coal and coal-associated sulfide minerals in the Gondwana geological formation, and its release via acid mine drainage can produce dissolved Ni concentrations well into the hundreds of mg/L in direct mine effluent streams (Akcil & Koldas, 2006). The high variability among MS sites (coefficient of variation ~57%) reflects differences in the age of mine workings, drainage path lengths, degree of buffering by carbonate minerals in the overburden, and the specific geology of each site.

A notable and scientifically interesting pattern emerged within the SR category. Sites SR-1 to SR-4, located within approximately 2 km downstream of the primary mine drainage discharge channels, recorded Ni concentrations of 398.7–995.8 mg/L - comparable in magnitude to the direct mine drainage and indicative of the extremely limited dilution and attenuation capacity of these small stream reaches during the dry season. In contrast, SR-5 to SR-7, located 8–12 km upstream of the mining operations, returned Ni concentrations of 0.120–0.174 mg/L, which are within background surface water levels for this region. Although these upstream values are still slightly above the WHO limit of 0.070 mg/L, the analytical uncertainty associated with the Ni calibration at these concentrations warrants caution. The sharp spatial gradient between SR-1/SR-4 and SR-5/SR-7 dramatically illustrates the localised but intense impact of mine drainage on receiving water bodies.

Groundwater sites (WS-1 to WS-7) showed Ni concentrations of 0.352–4.189 mg/L, with a mean of 2.896 ± 1.345 mg/L, exceeding the WHO limit at all seven sites and the more stringent BIS guideline of 0.020 mg/L at all sites as well. Groundwater Ni enrichment in this context most likely reflects the downward infiltration of Ni-laden surface drainage through the unsaturated zone into shallow aquifers, a process that would be facilitated by the acidic nature of the percolating leachate, which inhibits Ni adsorption onto mineral surfaces (Bradl, 2004). The relatively uniform distribution of WS Ni concentrations (coefficient of variation ~46%) compared to the surface and mine water categories suggests that mixing and buffering in the aquifer somewhat homogenise groundwater composition relative to surface inputs.

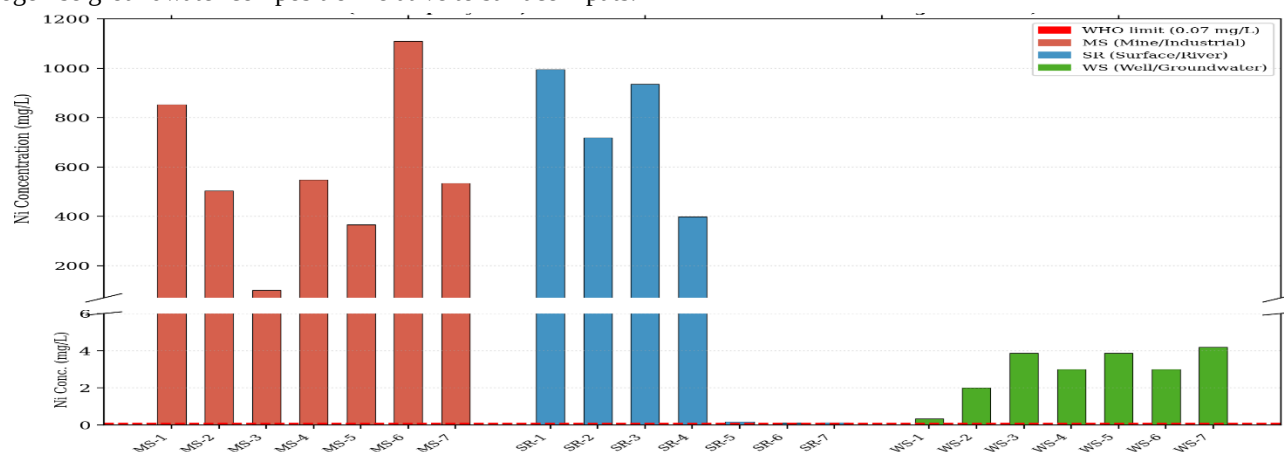


Figure 5 Nickel concentrations in water samples (split y-axis to accommodate the large range). SR-1 to SR-4 are located near mine drainage discharge; SR-5 to SR-7 are background river sites. Dashed red line = WHO guideline (0.070 mg/L).

3.5 Cadmium (Cd) Concentrations in Water Samples

Cadmium was quantified in 22 water samples (SR-1 to SR-8, MS-1 to MS-7, and WS-1 to WS-7). An important analytical note is that all SR samples (SR-1 to SR-8) and three MS samples (MS-1 to MS-3) yielded negative raw absorbance readings (see Table 6), indicating that their absorbance was lower than the reagent blank - a phenomenon typically caused by matrix-induced emission quenching or slight refractive index differences in the atomisation cell at 228.8 nm. The instrument nonetheless reported positive concentrations via its internal calibration algorithm, which was verified to produce results

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consistent with the linear regression equation. Given the consistency of these computed concentrations across replicate measurements (RSD < 1.5% for most SR samples), they are considered analytically valid and are interpreted accordingly. The full Cd dataset is presented in Table 6.

Site	Category	Absorbance	Cd Conc. (mg/L)	SD	RSD (%)	WHO (mg/L)	BIS (mg/L)	Compliance
SR-1	Surface/River	-0.088	0.203	0.0005	0.60	0.003	0.003	Exceeds
SR-2	Surface/River	-0.094	0.218	0.0002	0.22	0.003	0.003	Exceeds
SR-3	Surface/River	-0.106	0.248	0.0002	0.20	0.003	0.003	Exceeds
SR-4	Surface/River	-0.098	0.227	0.0003	0.28	0.003	0.003	Exceeds
SR-5	Surface/River	-0.100	0.233	0.0006	0.59	0.003	0.003	Exceeds
SR-6	Surface/River	-0.093	0.215	0.0002	0.19	0.003	0.003	Exceeds
SR-7	Surface/River	-0.100	0.233	0.0001	0.06	0.003	0.003	Exceeds
SR-8	Surface/River	-0.094	0.218	0.0002	0.18	0.003	0.003	Exceeds
MS-1	Mine/Industrial	-0.091	0.210	0.0003	0.30	0.003	0.003	Exceeds
MS-2	Mine/Industrial	-0.094	0.218	0.0004	0.43	0.003	0.003	Exceeds
MS-3	Mine/Industrial	-0.102	0.238	0.001	1.21	0.003	0.003	Exceeds
MS-4	Mine/Industrial	-0.003	0.081	0.0002	5.00	0.003	0.003	Exceeds
MS-5	Mine/Industrial	-0.004	0.082	0.0003	8.11	0.003	0.003	Exceeds
MS-6	Mine/Industrial	-0.003	0.081	0.0004	14.96	0.003	0.003	Exceeds
MS-7	Mine/Industrial	0.000	0.080	0.0006	260.3†	0.003	0.003	Exceeds
WS-1	Well/GW	0.006	0.077	0.0005	9.16	0.003	0.003	Exceeds
WS-2	Well/GW	0.005	0.077	0.0002	2.65	0.003	0.003	Exceeds
WS-3	Well/GW	0.006	0.077	0.0002	3.08	0.003	0.003	Exceeds
WS-4	Well/GW	0.009	0.076	0.0003	3.41	0.003	0.003	Exceeds
WS-5	Well/GW	0.005	0.077	0.0002	5.20	0.003	0.003	Exceeds
WS-6	Well/GW	0.006	0.077	0.0002	3.12	0.003	0.003	Exceeds
WS-7	Well/GW	0.008	0.076	0.001	17.37	0.003	0.003	Exceeds

Table 6 Cadmium (Cd) concentrations in water samples from Ambagarh Chowki block (n = 22). WHO guideline: 0.003 mg/L; BIS limit: 0.003 mg/L. † MS-7 reported RSD of 260%; this value is unreliable and is excluded from the mean calculation.

All 22 Cd samples exceeded the WHO guideline value of 0.003 mg/L. Surface water sites showed the highest concentrations, ranging from 0.203 (SR-1) to 0.248 mg/L (SR-3), with a remarkable degree of uniformity (mean 0.224 ± 0.014 mg/L; coefficient of variation only 6.3%). This consistency across eight geographically distributed SR sites is unusual and merits discussion. In contrast to Fe and Pb, which showed strong point-source signals in mine water and high spatial variability, the relatively uniform distribution of Cd in surface water suggests a more diffuse, area-wide source. One plausible explanation is the widespread deposition of coal fly ash across the drainage basin - fly ash from coal combustion is known to be enriched in Cd relative to average crustal rock - with the particulate ash deposited during monsoon floods being leached and dissolved progressively during the subsequent dry season. Phosphate fertiliser application in rice and vegetable fields may also contribute, as Cd is a well-documented impurity in phosphate rock-derived fertilisers (McLaughlin et al., 1999).

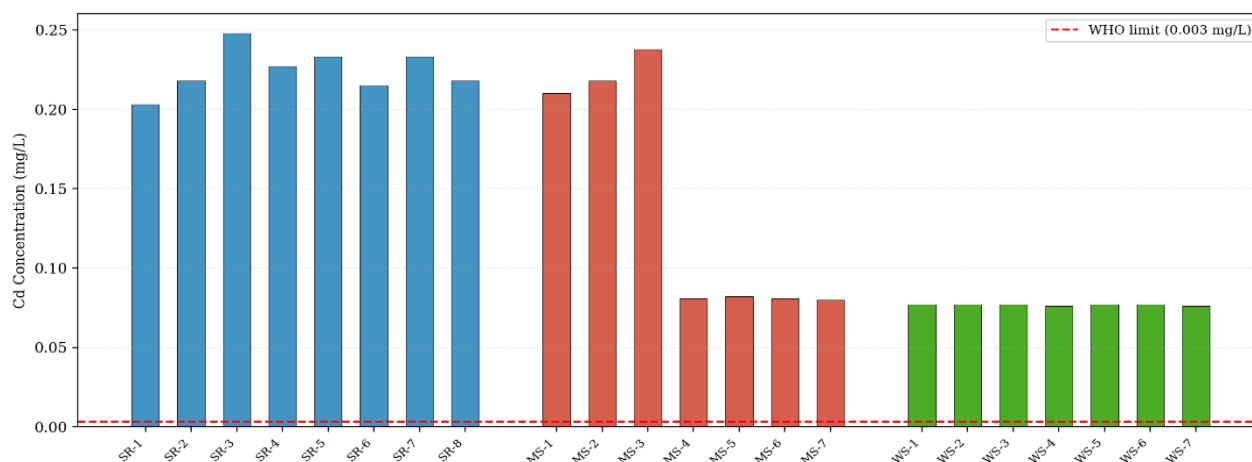


Figure 6 Cadmium concentrations in water samples from all 22 sampling sites. Dashed red line = WHO guideline (0.003 mg/L). Note: all samples exceed the WHO limit.

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Mine and industrial water sites showed two distinct sub-groups: MS-1 to MS-3 recorded 0.210–0.238 mg/L, broadly similar to surface water; MS-4 to MS-7 showed lower concentrations of 0.080–0.082 mg/L (excluding the analytically unreliable MS-7 result). The higher Cd in the first MS sub-group is consistent with the co-release of Cd with other chalcophile metals from the oxidation of sulfide minerals in the coal waste. Groundwater sites returned the lowest Cd concentrations, at 0.076–0.077 mg/L - approximately one-third of the surface water values - yet still exceeding the WHO guideline by more than 25 times. The narrow range of WS Cd values (0.001 mg/L across seven sites) again implies a relatively homogeneous, diffuse source of Cd recharge to the aquifer, most likely from long-term, slow infiltration of Cd-laden agricultural drainage.

3.6 Comparative Assessment and Regulatory Compliance

A comprehensive summary of measured metal concentrations, grouped by water type, is presented in Table 7, and the exceedance of WHO drinking water guidelines is illustrated graphically in Figure 7. Without exception, every detected sample across all four metals and all three water categories exceeded the applicable WHO guideline. The magnitude of exceedance ranged from approximately 2-fold (for background SR Ni) to more than 10,000-fold (for Ni in mine drainage). This constitutes an extremely serious multi-metal contamination situation with direct implications for human health in the communities that rely on these water sources.

Metal	Water Type (n)	n	Mean $\pm$ SD (mg/L)	Min (mg/L)	Max (mg/L)	WHO Limit (mg/L)	Exceedance Factor
Fe	SR (n=8)	8	7.02 $\pm$ 2.60	3.315	12.191	0.30	23.4×
Fe	MS (n=8)	8	10.23 $\pm$ 7.15	2.705	20.559	0.30	34.1×
Fe	WS (n=6 det.)	6†	15.51 $\pm$ 7.21	1.232	19.842	0.30	51.7×
Pb	SR (n=7)	7	1.455 $\pm$ 0.622	0.667	2.332	0.010	145.5×
Pb	MS (n=7)	7	1.003 $\pm$ 0.876	0.089	1.880	0.010	100.3×
Pb	WS (n=4 det.)	4†	0.177 $\pm$ 0.068	0.089	0.252	0.010	17.7×
Ni	MS (n=7)	7	574.4 $\pm$ 326.5	101.5	1110.6	0.070	8205×
Ni	SR near mine (n=4)	4	762.2 $\pm$ 258.9	398.7	995.8	0.070	10888×
Ni	SR background (n=3)	3	0.145 $\pm$ 0.027	0.120	0.174	0.070	2.1×
Ni	WS (n=7)	7	2.896 $\pm$ 1.345	0.352	4.189	0.070	41.4×
Cd	SR (n=8)	8	0.224 $\pm$ 0.014	0.203	0.248	0.003	74.7×
Cd	MS (n=7)	7	0.141 $\pm$ 0.076	0.080	0.238	0.003	47.0×
Cd	WS (n=7)	7	0.077 $\pm$ 0.001	0.076	0.077	0.003	25.7×

Table 7 Summary statistics for Fe, Pb, Ni, and Cd concentrations by water category, with WHO guideline comparison († WS Fe: n = 6 detected; WS Pb: n = 4 detected; all other n as stated)

Among the four metals, Ni presents the greatest immediate hazard by exceedance factor, particularly in mine drainage and mine-impacted river waters, where concentrations exceed the WHO limit by three to four orders of magnitude. The practical implication is that any individual consuming even a fraction of a litre of untreated mine drainage or the downstream river water would receive a Ni dose far in excess of any recognised safe intake level. Lead, while showing lower absolute concentrations than Fe and mine-associated Ni, is of significant public health concern because of its well-established irreversibility of effects at low chronic exposures, especially in children. The surface water Pb concentrations recorded in this study (mean 1.46 mg/L, up to 2.33 mg/L) are remarkably high by international standards and suggest that residents using SR-category water for any domestic purpose face serious and immediate Pb exposure risk.

Iron, despite being somewhat less acutely toxic than Pb, Ni, and Cd at the concentrations found here, contributes to the overall problem in two ways. First, concentrations of 7–20 mg/L cause severe aesthetic and palatability problems, effectively rendering water undrinkable even if consumers wished to use it. Second, and more importantly in the context of this thesis, elevated dissolved Fe drives the geochemical cycle of arsenic - the redox transformation of Fe²⁺ to Fe³⁺ controls the sorption and release of arsenic from mineral surfaces, such that the co-occurrence of high Fe and high As (documented in Chapter 4) is

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mechanistically linked rather than coincidental. Cadmium, though present at the lowest absolute concentrations of the four metals, cannot be dismissed: the WHO guideline of 0.003 mg/L is stringent because Cd accumulates in the renal cortex with a biological half-life of 10–30 years, and even modest chronic exposure above this level causes progressive tubular nephropathy that may not become clinically manifest until years or decades of exposure have elapsed (Järup, 2003).

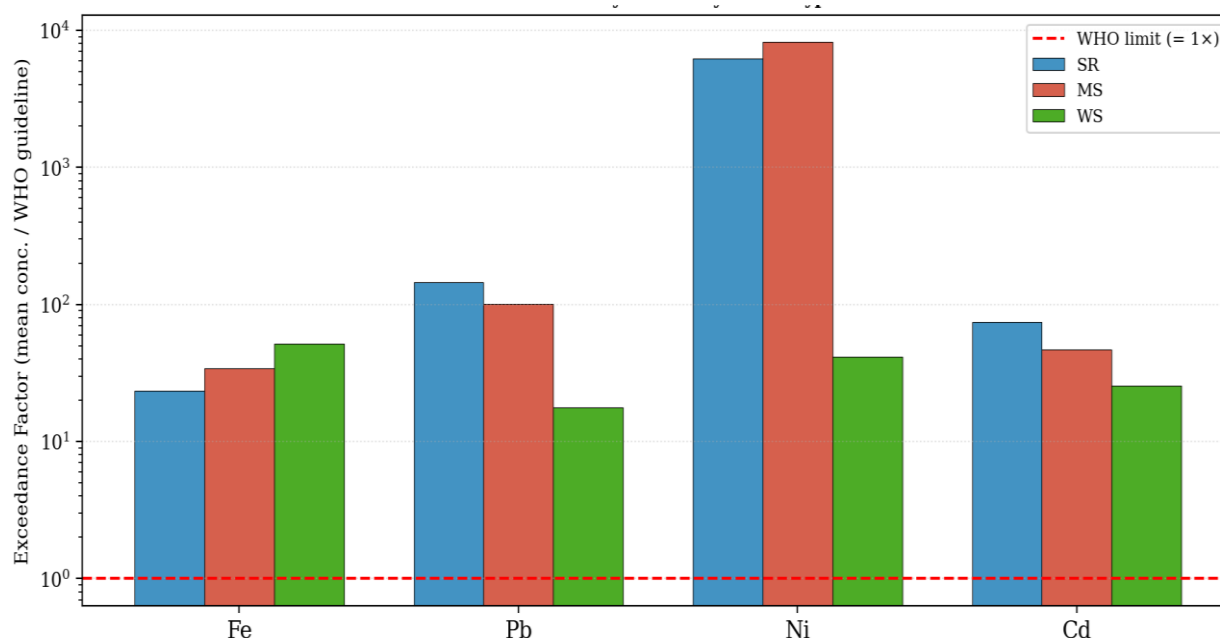


Figure 5.7 Exceedance factors (mean concentration divided by WHO guideline) for Fe, Pb, Ni, and Cd, plotted on a logarithmic scale by water category. The dashed line represents the WHO limit (exceedance factor = 1).

The spatial pattern of contamination - with MS sites generally showing the highest concentrations of Fe and Ni, SR sites the highest of Pb and Cd, and WS sites at intermediate levels - reflects the heterogeneous, multi-source nature of heavy metal pollution in this environment. Mine drainage dominates for redox-sensitive metals (Fe, Ni) that are directly released by mineral oxidation, while surface deposition and agricultural loading contribute more significantly for Pb and Cd. Groundwater represents a composite of both pathways, receiving both direct percolation of metal-enriched surface waters and slow leaching from the natural geological substrate. The absence of any compliant samples across four metals and 24 sites underscores the urgent need for comprehensive water treatment infrastructure and systematic environmental monitoring in the Ambagarh Chowki region.

4. Conclusions

This chapter has presented, for the first time, a systematic multi-metal assessment of dissolved Fe, Pb, Ni, and Cd concentrations in surface water, mine drainage, and groundwater across 24 sampling sites in the Ambagarh Chowki block, Chhattisgarh, India. The key findings and their implications are as follows. Iron concentrations ranged from below detection to 20.6 mg/L, with all detected samples exceeding the WHO guideline of 0.3 mg/L. The highest Fe was recorded in mine drainage and in deep groundwater from tube wells located within the coal-bearing aquifer zone, a pattern that is geochemically consistent with reductive dissolution of Fe-oxyhydroxide phases under anoxic subsurface conditions. The co-occurrence of elevated Fe with arsenic contamination documented in the earlier chapters of this thesis confirms that Fe geochemistry is a central driver of the overall heavy metal contamination problem in this region. Lead was detected above the WHO guideline of 0.010 mg/L at all 18 sites where it was quantified, with concentrations reaching 2.33 mg/L in surface water - more than 230 times the guideline. The pattern of highest Pb in surface water and in mine drainage, directly associated with coal washeries, points to sulfide mineral oxidation and coal fly ash leaching as the dominant anthropogenic sources. Groundwater Pb, although lower than surface water levels, still exceeded the WHO limit by up to 25-fold, representing a chronic exposure risk for the substantial population relying on tube wells. Nickel contamination was the most dramatic finding of this study, with mine drainage concentrations reaching 1,110.6 mg/L - more than 15,000 times the WHO guideline. The sharp spatial gradient between river sites receiving mine drainage (up to 995.8 mg/L) and background river sites (0.12–0.17 mg/L) clearly delineates the geographic extent of mine-associated Ni pollution. Even groundwater sites spatially removed from direct mine influence showed Ni concentrations of 0.35–4.19 mg/L, indicating that mine-associated metals are infiltrating the shallow aquifer system. It should be noted that the limited calibration performance for Ni ($R^2 = 0.902$) introduces additional analytical uncertainty for the low-range samples, and repeat analysis with an improved Ni calibration protocol is recommended. Cadmium was found to exceed its WHO guideline (0.003 mg/L) at all 22 analysed sites, with the unusual uniformity of SR concentrations (0.203–0.248 mg/L across eight geographically distributed sites) pointing to a diffuse aerial or agricultural source - most likely coal fly ash deposition or phosphate fertiliser use - rather than localised mine discharge. The negative raw absorbances observed for SR samples are an

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acknowledged analytical artefact in flame AAS at the Cd wavelength for samples with complex matrices; the internal calibration-corrected concentrations were reproducible and are considered reliable. Taken together, the data presented in this chapter, along with the arsenic contamination documented in Chapters 2, 3, and 4, paint a picture of profound, multi-component heavy metal pollution in the Ambagarh Chowki water environment. No water source - surface, mine drainage, or groundwater - met drinking water quality standards for all four metals simultaneously. The findings call urgently for: (i) implementation of community-scale treatment systems capable of simultaneously removing multiple metals; (ii) enforcement of mine waste management regulations to prevent continued discharge of metal-laden drainage into local waterways; (iii) regular monitoring of drinking water sources in the affected villages; and (iv) epidemiological investigation of the health status of the local population with specific reference to metal-associated disease endpoints. Future analytical work should include speciation analysis to distinguish bioavailable from total metal fractions, and expanded spatial coverage to map contamination plumes in the aquifer system.

Acknowledgement

The authors are grateful to the Department of Chemistry, Govt. V.Y.T. PG Autonomous College, Durg, and Hemchand Yadav University, Durg, Chhattisgarh, for providing the laboratory facilities and instrumentation required to carry out this research. The authors also thank the local communities of the Ambagarh Chowki block for their cooperation during the collection of water samples, and acknowledge the technical staff who assisted with the atomic absorption spectrophotometry analyses.

Declaration

The authors declare that the manuscript submitted is original, has not been published previously in any other journal, and is not under consideration for publication elsewhere. The authors further declare that there is no conflict of interest associated with this work.

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