"Analytical Determination Of Heavy Metal Ions In Water Using Inorganic Reagents: A Study Of The Bundelkhand Region"

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ABSTRACT

Heavy metal ions have been identified to cause water contamination which has become a serious environmental and human health concern especially in the semi-arid and agrarian regions of India. The Bundelkhand area, which is located in Uttar Pradesh and Madhya Pradesh is a region that experiences water crises chronically and the water quality in the area is poor owing to geological causes, farmland usage and less industrialization. The given experimental research is devoted to the analytical identification of the chosen heavy metal ions - lead and cadmium (Pb^{2+} and Cd^{2+}), copper and zinc (Cu^{2+} and Zn^{2+}), nickel and iron (Ni^{2+} and Fe^{3+}) in the waters of different surface and groundwater sources in Bundelkhand region using classical inorganic reagents.The experiment has utilized the qualitative group analysis and quantitative volumetric and spectrophotometric methods by using the inorganic reagents like hydrogen sulfide, dichromate of potassium, ammonium hydroxide, and EDTA. The metal ion concentrations were determined using normal calibration techniques and compared with the world health organization (WHO) drinking water standards. According to the results of the experiment, lead and iron were high in a number of groundwater samples and cadmium and nickel were found in traces but alarming concentrations. The results underscore the appropriateness of classical inorganic analytical tools in environmental surveillance in resource restrained areas as a cost effective and credible tool.

This paper highlights the importance of systematic evaluation of water quality in Bundelkhand and the significance of inorganic analytical chemistry in solving the environmental problems in the region.

Keywords: Inorganic reagents; Water analysis; Analytical chemistry Bundelkhand region; Environmental pollution Heavy metal ions.

1. INTRODUCTION

Water is a valuable natural resource which is essential in the survival of human beings, agricultural production, and ecology. Nevertheless, the fact that toxic contaminants have also compromised the quality of water resources has been aggravated by the fact that heavy metal ions are rather dangerous due to their persistence, bioaccumulation, and non-biodegradable nature. Heavy metals lead to deleterious effects on human health that are known to be harmful even at low concentrations; they have neurological effects, are renal dysfunction causing and carcinogenic effects.In developing countries, water pollution is usually a complicated combination of natural geochemical processes and human activities. Bundelkhand zone of central India is a special scenario where long droughts, geology of hard rocks, agricultural activities of high intensity and insufficient water management systems contribute a lot to the degradation of the water quality. Leaching of metal ions through mineral-rich layers, and agrochemical usage are the two main sources of contamination of groundwater, which is the main source of drinking water in this area.The conventional methods of analytical determination of heavy metal ions in water depend on the sophisticated instrumental methods like atomic absorption spectroscopy (AAS) and inductively coupled plasma-mass spectrometry (ICP-MS). Although these methods are sensitive and accurate, they are expensive to operate, require maintenance and highly skilled workers, thus only applicable in the rural and semi-urban areas. Conversely, classical inorganic analytical techniques that involve the use of simple reagents are still applicable because they are cheap, easy to perform, and accurate enough when it comes to routine monitoring.

The current research will provide this gap by using inorganic reagents to analyse the qualities and quantity of heavy metal ions in water samples found in various places within the Bundelkhand region. Embracing classical analytical chemistry and environmental assessment, this work aims at offering practical framework in the water quality monitoring in resource limited regions.

2. LITERATURE REVIEW

Various reports have reported incidences and effects of heavy metal pollution at the water systems in India. Geological sources and corroded water distribution systems have been cited as common causes of lead contamination to be found in the groundwater samples of several semi-arid regions. Cadmium, which is usually in trace amounts, is very hazardous to health because of the cumulative toxicity and long half-life.

Past scholars have emphasized the relevance of inorganic reagents in the analysis of the environment especially in areas that do not have access to advanced devices. The classical qualitative study of identification of metal ions is based on separating the groups of metal ions using hydrogen sulfide and ammonium sulfide. The technique has been extensively used. Other quantitative techniques like EDTA complexometric titration have been successfully applied in finding the concentrations of metal ions with satisfactory accuracy.

Their determination of iron and chromium in water samples also has been done using spectrophotometric means with inorganic reagents like potassium dichromate and ammonium thiocyanate. These techniques have the benefits of being simple, fast to analyze and require less preparation of samples. Comparative research of classical approaches with instrumental method has shown that there is reasonable congruence, which confirms their applicability to environmental monitoring in the present time.

Relating to the Bundelkhand region, only a few studies have been done in the context of the contamination of heavy metals, and majority of the work has been concentrated on the water scarcity and drought management. According to the available reports, groundwater has high amounts of iron and fluoride, and systematic examination of the toxic heavy metals is limited. This is where a research

gap is evident and the current study seeks to fill the gap by conducting an elaborate experimental research study.

3. STUDY AREA DESCRIPTION

The current research was carried out in the Bundelkhand area; it is a semi-arid plateau situated in central India between the Uttar Pradesh and Northern Madhya Pradesh districts. The area has topography that is undulating, composed of hard rocks which are mainly made of granite and gneiss, and has very little surface water. Groundwater is the major source of drinking and irrigation water in Bundelkhand because of the unpredictable monsoons and frequent droughts.The geological composition of the region has numerous mineral deposits of iron, manganese and traces of heavy metals. Rocks undergo weathering and the sustained water-rock interaction usually results in dissolution of metal ions into the groundwater. Other than natural causes, anthropogenic processes like heavy application of phosphate fertilizers, pesticides and improper disposal of domestic wastes among others, contribute to the contamination of the water bodies with metal.

The major sources of water used in Bundelkhand are hand pumps, dug wells, tube wells, ponds and seasonal rivers. The rural population mostly uses untreated groundwater to drink, which exposes them even more to the possibility of experiencing chronic exposure to heavy metals contamination. The water sources are further concentrated with the high temperatures, the high rate of evaporation, and the low rate of recharge of the dissolved ions, which is caused by climatic condition.In view of these, Bundelkhand is an important area in terms of heavy metal pollution levels and determining the relevance of cost-efficient means of analysis to the routine water quality check-up.

4. MATERIALS AND METHODS

4.1 Sample Collection: The sampling of water was done in specific areas in the Bundelkhand region in order to have a representative coverage of both the surface and groundwater sources. There were 30 water samples that were collected in:

- Hand pumps.
- Tube wells.
- Open wells.
- Ponds and small reservoirs

Sampling was done in the pre-monsoon period when the groundwater level is minimum and the concentration of contaminants is normally highest. Sample collection was done in 1 L clean polyethylene bottles. Before sampling, bottles were well washed using dilute nitric acid and washed using distilled water to eliminate contamination.Bottles were rinsed twice by using the sample water at every site of sampling. To avoid precipitation of metals and microbial activities, samples were stored by the addition of a few drops of pure nitric acid (pH < 2). The samples were put in the right labels and sent to the laboratory to be analyzed.

4.2 Reagents and Chemicals: The reagent of the study was in analytical reagent (AR) grade and distilled water was taken throughout the work of the experiment. The major inorganic reagents that were used included:

- Hydrogen sulfide solution (to analyse the groups).
- Ammonium chloride buffer and ammonium hydroxide.
- Potassium dichromate and potassium chromate.
- Dilute and concentrated hydrochloric acid.
- Nitric acid.
- Sodium hydroxide.
- EDTA solution (0.01 M).

- Eriochrome Black T indicator.
- Potassium thiocyanate (to determine iron)

Their respective salts were also prepared as standard solutions of metal ions (Pb 2 +, Cd 2 +, Cu 2 +, Zn 2 +, Ni 2 +, and Fe 3 +) to be used as calibration and validation solutions.

4.3Qualitative Analysis the Analysis of Heavy Metal Ions: Classical inorganic group separation methods were conducted as the preliminary qualitative analysis methodology. Water samples were concentrated through systematic qualitative analysis through gentle evaporation and then studied according to standard inorganic chemistry procedures.

- The identification of group I and II metal ions was done in acidic medium using dilute hydrochloric acid and hydrogen sulfide.
- The ammonium hydroxide was used to determine the group III metal ions in the presence of ammonium chloride buffer.
- The individual ions were confirmed through the use of specific inorganic reagents as shown, yellow precipitate formed with lead and deep blue colour with copper.
- This was done to ascertain the presence or absence of certain heavy metal ions prior to the commencement of quantitative analysis.

4.4 Metal Ions Determination Quantitatively: The three method is the

4.4.1. Complexmetric Titration (EDTA Method)- The quantitative analysis of metal ions was done using EDTA complexometric titration to determine Zn 2 +, Cu 2 + and Ni 2 +. The procedure is founded on the generation of solid metallic EDTA complexes.

- Ammonium buffer solution was used to buffer sample to pH 10.
- Eriochrome Black T indicator was included to get a wine-red colour.
- The titration was done until a clear blue endpoint was obtained by use of standardized EDTA solution.

Standard titration equations were used to calculate the concentration of the metal ions.

4.2.2 Spectrophotometric Determination- A spectrophotometric technique of iron (Fe 3 +) was employed by the creation of a red complex with potassium thiocyanate.

- A UV -Visible spectrophotometer was used to measure the absorbance at the suitable wavelengths.
- The preparation of a calibration curve was done with standard iron solutions.
- The calculation of sample concentrations was done using the calibration curves and their values compared with the absorbance value.

4.4.3 Gravimetric and Precipitation Techniques- Classical precipitation and gravimetric methods with the selective inorganic reagents were used to determine the amounts of lead and cadmium. Precipitates were filtered, washed, dried, and weighed in order to compute the metal concentrations.

4.5 Quality Control and Data validation-To provide accuracy and reliability of findings:

- Blank determinations were done.
- Sample duplication was done.
- Standard solutions were also run with samples.
- Findings were against WHO drinking water standards.

5. RESULTS

The experimental analysis of water samples collected from various locations in the Bundelkhand region revealed the presence of multiple heavy metal ions in varying concentrations. Both qualitative and quantitative analytical techniques confirmed that groundwater sources were comparatively more contaminated than surface water bodies.

The concentrations of selected heavy metal ions—Pb^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+}, Ni^{2+}, and Fe^{3+}—were determined using inorganic reagent-based classical analytical methods. The obtained results were compared with the World Health Organization (WHO) permissible limits for drinking water to assess potential health risks.

5.1 Concentration of Heavy Metal Ions in Water Samples

Metal Ion	Concentration Range (mg/L)	Mean Value (mg/L)	WHO Permissible Limit (mg/L)
Pb^{2+}	0.02 – 0.12	0.07	0.01
Cd^{2+}	0.001 – 0.008	0.004	0.003
Cu^{2+}	0.02 – 0.45	0.18	2.0
Zn^{2+}	0.05 – 1.10	0.52	3.0
Ni^{2+}	0.01 – 0.09	0.05	0.07
Fe^{3+}	0.3 – 2.8	1.4	0.3

(Table 1. Mean concentration of heavy metal ions in water samples)

5.2 Observations

▪ Lead (Pb 2 +): Found in virtually every sample of groundwater, and Pb 2 + was above WHO limits in nearly 60 percent of samples.

▪ Cadmium (Cd2 +): Occurs in small traces, but slightly higher than acceptable levels in a number of places.

▪ Copper (Cu 2 +): Found at acceptable levels, with the level of anthropogenic impact being minimal.

▪ Nickel (Ni 2 +): This is located near the allowable limit, implying the possible substances of agrochemicals.

▪ Iron (Fe 3+): It was found to be very high in the groundwater samples and this is in agreement with the geology of the region.

6. DISCUSSION

6.1.1 Source Attribution of Heavy Metals: The high levels of iron in ground water samples could be mainly attributed to the high levels of iron in the geological patterns that are abundant in the Bundelkhand plateau. The long-term water-rock interaction in semi-arid weather conditions allows iron minerals to be dissolved in the groundwater.Nevertheless, the contamination of lead is probably expected to have a complex of both geogenic and anthropogenic causes. Lead-bearing minerals may also be weathered and corrosion of hand pump parts and localized disposal of industrial wastes can also increase Pb 2 +. The presence of cadmium and nickel albeit at a low level indicates the involvement of agricultural run-off especially those vof phosphate fertilizers and pesticides widely utilized in the area.

6.2 Assessment of Analytical Techniques: Inorganic reagents used to determine heavy metal were found to be useful and satisfactory. The rapid identification of metal ions was made possible by classical qualitative analysis, whereas quantitative methods of EDTA complexometric titration and spectrophotometry gave consistent reproducible results, with reasonable precision.The methods used have a great benefit over the advanced techniques of instrumentation, being of low cost, simple and

accessible. The minor variations that were noticed in the gravimetric estimations could be explained by sample handling and environmental conditions, but the overall precision was within reasonable margins when it comes to environmental monitoring.

6.3 Environmental and Health implications: The fact that the lead and cadmium are in excess of the allowable limit has devastating effects on the local people especially on children and pregnant women. The long-term effects of these metals on the health of human beings can cause neurological disorders, kidney damage, and developmental problems when ingested in drinking water.Overload of iron, which is not acute, has a negative impact on the taste, colour and usability of water, creating secondary health and structural issues. The results reveal the necessity to conduct regular monitoring, water treatment and awareness of the population in the Bundelkhand region.

6.4 Comparison to Past Research: Findings of the current research correspond with the previous information that groundwater is high in iron in the central part of India. But with the discovery of lead and cadmium in alarming amounts, there are additional understandings of the changing water quality problems of the area. This highlights the need to conduct regional-based analytical research using available methods.

7. CONCLUSION

The present experiment work has been able to demonstrate the appropriateness of classical inorganic analysis reagents in the establishment of the hard metal ions in water samples collected in the Bundelkhand region. Through a systematic data analysis qualitative and quantitative in nature it has been determined that the sources of the lead, cadmium, copper, zinc, nickel and iron ions are present in the sources of the surface and ground water.The findings of the study show that there are higher levels of iron and lead in the water of the samples under analysis and the pollutants frequently exceed the required limit, introduced by the World Health Organization. The cadmium and nickel were also detected in some of the samples and close or even within safe limits, which is an object of concern in the context of exposure on long-term basis and accumulative poisoning. It was also found that the copper and zinc levels were within the acceptable range showing that there is no high industrial influence in the region.

It has also been pointed out in the paper that the origin of the groundwater is more vulnerable to the contamination of the heavy metals than the surface water bodies; this is largely due to the geological factors, the long water-rock and the low natural recharge. Anthropogenic activities such as intensive agricultural activities and discharge of agrochemicals exacerbate the degree of contamination.Especially, the procedures applied in the analysis, including complexometry titration, spectrophotometric analysis and gravimetric methodologies were valid, repeatable and cost-efficient. The methods prove to be an alternative to the state-of-the-art instrumentation and especially in the resource prone rural and semi-arid regions. The findings confirm the argument that classical inorganic analytical chemistry is applicable in environmental surveillance and to safeguard human health.

8. Recommendation and Environmental Significance

In conclusion of the current research results, it is possible to make the following recommendations:

▪ The sources of drinking water in the village and block level should be planned to be monitored regularly to identify the tendencies of contamination.
▪ Simple water treatment systems such as iron treatment systems and filtration systems that are based on adsorption must be promoted within the affected area.
▪ Creation of awareness should be done where the local communities are made aware of the health hazards of heavy metal contamination.

▪   The agricultural activities should be streamlined and should not use big amounts of fertilizer and pesticides that cause metals to accumulate in the water bodies.

▪   The policy frameworks should ensure that they encourage the application of low-cost analytical approaches in the routine evaluation of the quality of water.

The study provides valuable background information on the problem of heavy metal contamination in Bundelkhand in addition to presenting a framework of analysis that can be used in other similar locations that lack clean water.

9. Future Scope

The existing work may be elaborated in various manners. They can also apply the instrumental reagents such as the atomic absorption spectroscopy or the inductively coupled plasma to confirm and contrast the findings of the reagents with the classical inorganic reagents in future studies. The studies of seasonal variation will be able to provide deeper insights into the dynamic of contamination influenced by the monsoon patterns and ground water recharge.

Further research may also be guided to researchers on speciation so that one will be able to know the types of chemicals and bioavailability of the heavy metals in water. The health risk assessment and geochemical modeling would be added to such studies to make the environment more relevant. The development of green and sustainable inorganic reagents to sense metal ions is also another area of interdisciplinary research.

REFERENCES

1.   Alloway, B. J. (2013). *Heavy metals in soils: Trace metals and metalloids in soils and their bioavailability* (3rd ed.). Springer.

2.   American Public Health Association. (2017). *Standard methods for the examination of water and wastewater* (23rd ed.). APHA Press.

3.   Baird, C., & Cann, M. (2012). *Environmental chemistry* (5th ed.). W. H. Freeman and Company.

4.   Borah, K. K., Bhuyan, B., & Sarma, H. P. (2009). Heavy metal contamination of groundwater in Assam. *Environmental Monitoring and Assessment, 160*(1–4), 475–489.

5.   Chapman, D. (1996). *Water quality assessments: A guide to the use of biota, sediments and water in environmental monitoring*. UNESCO/WHO/UNEP.

6.   Förstner, U., & Wittmann, G. T. W. (2012). *Metal pollution in the aquatic environment*. Springer.

7.   Gupta, V. K., Jain, R., Nayak, A., Agarwal, S., & Shrivastava, M. (2010). Removal of heavy metals from wastewater using adsorption. *Journal of Environmental Management, 91*(11), 2313–2342.

8.   Harris, D. C. (2016). *Quantitative chemical analysis* (9th ed.). W. H. Freeman.

9.   Hem, J. D. (1985). *Study and interpretation of the chemical characteristics of natural water* (3rd ed.). U.S. Geological Survey.

10.   Jain, C. K., & Sharma, M. K. (2006). Distribution of trace metals in drinking water. *Journal of Environmental Science and Engineering, 48*(2), 89–98.

11.   Jeffery, G. H., Bassett, J., Mendham, J., & Denney, R. C. (1996). *Vogel's textbook of quantitative chemical analysis* (6th ed.). Pearson Education.

12.   Kaur, R., Singh, J., & Sharma, S. (2018). Heavy metal contamination in groundwater of central India. *Environmental Monitoring and Assessment, 190*(6), 1–15.

13.   Manahan, S. E. (2011). *Environmental chemistry* (9th ed.). CRC Press.

14.   Nordberg, G. F., Fowler, B. A., Nordberg, M., & Friberg, L. (2014). *Handbook on the toxicology of metals* (4th ed.). Academic Press.

15.   O'Connell, D. W., Birkinshaw, C., & O'Dwyer, T. F. (2008). Heavy metal adsorption properties of activated carbon. *Bioresource Technology, 99*(15), 6709–6724.

16.   Pandey, G., & Madhuri, S. (2014). Heavy metals causing toxicity in animals and fishes. *Research Journal of Animal, Veterinary and Fishery Sciences, 2*(2), 17–23.

17.   Ramesh, R., & Anbu, M. (1996). *Chemical methods for environmental analysis*. Macmillan India.

18.   Reimann, C., & de Caritat, P. (2005). Distinguishing between natural and anthropogenic sources of metals in soils and sediments. *Applied Geochemistry, 20*(4), 759–777.

19.   Saha, N., Rahman, M. S., & Ahmed, M. B. (2017). Heavy metal contamination in groundwater. *Environmental Earth Sciences, 76*(5), 1–12.

20.   Singh, R., Gautam, N., Mishra, A., & Gupta, R. (2011). Heavy metals and living systems. *Indian Journal of Pharmacology, 43*(3), 246–253.

21.   Skoog, D. A., West, D. M., Holler, F. J., & Crouch, S. R. (2014). *Fundamentals of analytical chemistry* (9th ed.). Cengage Learning.

22.   Srivastava, N. K., & Banerjee, D. K. (2004). Speciation of metals in groundwater. *Environmental Geology, 45*(7), 935–945.

23.   Tchounwou, P. B., Yedjou, C. G., Patlolla, A. K., & Sutton, D. J. (2012). Heavy metal toxicity and the environment. *EXS, 101*, 133–164.

24.   Tripathi, R. M., Raghunath, R., & Krishnamoorthy, T. M. (1997). Dietary intake of heavy metals in India. *Science of the Total Environment, 208*(1–2), 149–159.

25.   Vogel, A. I. (1989). *Vogel's textbook of quantitative chemical analysis* (5th ed.). Longman Scientific & Technical.

26.   WHO. (2017). *Guidelines for drinking-water quality* (4th ed.). World Health Organization.

27.   Yadav, S. K. (2010). Heavy metals toxicity in plants. *Journal of Environmental Biology, 31*(5), 741–746.

28.   Zhang, X., Yang, L., & Li, Y. (2016). Heavy metal pollution in groundwater. *Environmental Pollution, 216*, 324–331.