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Original Research Article

Assessment of different metals using the ICP-OES technique in the contaminated water of the Sonipat

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1. Introduction

The central pollution control board has implemented numerous environmental laws in recent decades as part of the Environment Action Program 2020 with the goal of lowering pollution in the air, soil, and water [1]. In January 2014, the suggested plan came into effect, outlining long-term goals and measures to safeguard the environment and avoid any possible negative effects on human health and biodiversity. The Management of waste from extractive industries section describes the management of mining industry waste, both historical and present, with the goal of preventing or lessening negative environmental effects [2] and the directive on landfills [3]. At the national level, each EU member state has a set of rules for evaluating the environmental impact and recovering contaminated areas. The quality of soil, surface water, and groundwater is degraded by the migration of heavy metals, metalloids, and other toxic chemicals from trash. Regular monitoring of the condition of deposited garbage and the surrounding environment is necessary since it directly impacts human health and biodiversity. The sediments of the Potomac and Anacostia Rivers have historically had high concentrations of Pb, Hg, and other metals, among other environmental issues. It is challenging to determine Hg using ICP-OES; instead, cold vapor atomic absorption is usually used. Hg was not included in this investigation since the authors lacked a cold vapor atomic absorption device. Large tracts of land around the Potomac River have previously been used for both surface and underground mining [4]. The common cause of dissolved

The purpose of the study was to determine the levels of metals, including heavy metals and trace elements, in the water from the Sonipat City common effluent treatment plants (CETPs) in Barhi and Rai. The technique of Inductively Coupled Plasma-Optical Emission Spectroscopy was used to examine the metals or trace elements, including Ag, Al, B, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, and Zn. The results indicated that the concentration of approximately all the metals, heavy metals and trace elements in these water bodies were found within the maximum permissible limit set (IS:2196), environmental protection agency (EPA) and world health organization WHO for surface water quality standard. This study's goal was to quantify the elements present in tainted water because it is commonly known that metal-containing textiles might provide a health concern to consumers. Knowing the metal content may also be helpful in industrial settings because some metals in textiles might cause problems during the manufacturing process. Pollutants in water systems and sediments are crucial for assessing environmental health and reducing hazardous levels. ICP-OES provides a useful way to monitor the concentrations of metals in sediments and water. industrial related acidic drainage can release dissolved metals into streams, ICP-OES makes mercury measurement challenging.

> metals entering current streams is acidic drainage from mining, although a widely accepted definition and standards for soil quality have yet to be established [5]. At the national level, proposals are being made to regulate the amounts of heavy metals, metalloids, and other contaminants in soil. Given the landfill's characteristics, its surroundings, and the type of waste, the regulator may impose particular requirements on permitted values for specific parameters. The assessment must show that the landfill's emissions, including leachate, will not pose any additional environmental risks (Landfill Directive 1999/31/EC) [6]. The following are the primary dangerous metals and their compounds that require close monitoring: lead (Pb), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), zinc (Zn), and arsenic (As) [6, 7]. Romania's Tarnita-Suceava region is renowned for its extensive mining industry. A vast amount of sterile dump material has been accumulated in multiple sterile dumps and tailing ponds as a result of the long-term operations of a barite mine in this area. The environment is negatively impacted by the deposited rubbish. [6, 8] Furthermore the chemical makeup of the surrounding soils and sterile dump material should be determined on a regular basis in order to track its ecological fate. It requires a verified analytical technique to identify dangerous metals. In our earlier study [8], we used a stepwise extraction process using distillated water and Mg (NO₃)₂ together with an acid digestion approach to assess the total metal content of sterile waste material. According to the



Copyright: © 2025 by the authors. Licensee Research Plateau Publishers, India This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/). findings, the waste material samples had higher than normal concentrations of As, Cd, Cu, Pb, and Zn. Some conventional procedures are mentioned, including ICP-OES determination is widely recognized for multicomponent determination of soil components. The most popular digestion technique for determining base metals in geological samples, soils, and mine waste using ICP-OES multi-element analysis is aquaregia wet digestion. [9, 10, 11, 12, 13]. Any laboratory should, however, optimize and validate the proper analytical process for sample preparation and ICP-OES results. The main objective is to estimate the analytical features of ICP-OES measurement of metalloids and heavy metals in sterile dump material following wet sample digestion. Aqua regia was used to determine the wet sample digestion efficiency. By examining approved reference soil material, the efficacy of metal extraction is confirmed. The usefulness of the optimized method for determining the target analytes in sterile dump material is confirmed by estimating the limits of detection and quantification, sensitivity, accuracy, and repeatability. In the Tarnita-Suceava region of Romania, the technique was used to identify heavy metals and metalloids in waste sterile dump material and adjoining soils at a disused barite mine.

2. Materials and methods

All required reagents used for metal analysis were from Sigma Aldrich (Fluka) and HNO₃ (60%), H₂O₂ (30%), HCl (40%) and HClO₄ (70%) which were). These reagents are commonly used in digestion methods. Different mixtures of mineral acids with varying volume ratios for reagent mixture were used to ensure complete digestion of the samples. Polyethylene bottles were used for storing the solutions. All the glassware and plastic containers which were used throughout this experiment were immersed in a dilute HNO₃ solution overnight and rinsed with de-ionized water, and finally dried in an oven (Xi'an Unique Electronics, UQ 9053A Drying Oven). Cellulose acetate membrane (Separations Scientific SA Ltd) filters (0.45 μ m) and the acrodiscs (0.45 μ m) syringe filters (Pall Corporation, USA) were used to filter the digested solutions throughout the study. The acrodiscs used were composed of a hydrophilic polyvinylidene fluoride (PVDF) filter membrane housed in a polypropylene. A multi-elemental standard solution of 1000 mg/L containing the elements of interest (As, Cd, Fe, Mn, Pb, Zn, Al, Cu, Cr and Ni) was used to obtain the working standard solutions. All the methods were verified using standard reference material (SRM) CWW-TM-A and CWW-TM-B (Trace Metals in Certified Wastewater from High-Purity Standards, Charleston, SC, USA) (CWW-TM-B-). Sample preparation was conducted in controlled laboratory conditions at 22 °C and only hot plate digestions were undertaken in standard laboratory fume hoods.

3. Sampling - study area

Two different sample from Sonipat from birhi and rai collected in plastic bottle which is labelled and stored at 5° C sample were acidified with 3 ml of HNO₃ to pH 2.3.

4. Experimental and instrumentation section

An "Ultra scientific" multi-element standard solution of different elements (Al, Ba, Be, Bi, B, Cd, Ca, Cr, Co, Cu, Ga, Fe, Pb, Li, Mg, Mn, Ni, K, Se, Na, Sr, Te, Tl, and Zn) at a concentration of 105 \pm 6 mg in 60% HNO₃L⁻¹ in Labperlitre were used. Thermo Fisher Scientific analytical grade HNO₃ 60% and HCl 40% were utilized to digest the samples. ICP-OES experiments analyze sample elemental composition through sample preparation, plasma generation, emission detection, and calibration, using argon gas, wavelength measurements, and calibration curves to determine element concentrations. The ICP-OES (Agilent 725 ICP-OES) is a high-throughput instrument used to measure the concentrations of selected elements. It features an auto sampler, a nebulizer, a spray chamber, a peristaltic pump, a nebulizer, and a spray chamber. The instrument uses a nebulizer to convert liquid samples into fine aerosols, a spray chamber to remove larger droplets, a peristaltic pump to control flow rate, and an autosampler for high-throughput analysis. The plasma source is the Inductively Coupled Plasma Torch, which creates a plasma with temperatures up to 10,000 K. The optical system includes a monochromator/polychromator, detectors, and a data processing unit. The plasma is generated using high-purity argon gas to prevent interference and stabilize the plasma. Reagents used in ICP-OES include calibration standards, acids for sample digestion, and dilution solvents. The data collected by the detectors is processed and interpreted using specialized software.

5. Sample digestion procedures

Closed digestion methods employing various acid mixtures were performed using a hot plate and a microwave digestion system. The acid digestion procedures were investigated in order to determine their reliability in the extraction of heavy metals in the sample 1(Barhi) and sample 2(Rai). The three acid digestion mixtures were tested with both hot plate and microwave conventional methods with slight modification. Digestion vessels on the hot plate were covered with the watch glass to reduce the loss of analyte due to vaporization of the sample. The samples were digested in triplicate for each procedure in order to quantify the closeness of the results obtained under the same conditions. The blank solutions, with identical reagents, subjected to the same treatment as the reference material were prepared as well.

6. Microwave digestion

The microwave procedure included an acid pre-digestion step whereby a mixture of acid in a Teflon vessel placed with the test sample was allowed to react at room temperature for 30 min prior to loading the samples into the microwave carousel. This step was performed with all the samples that were digested, especially those samples with high organic matter to avoid pressure buildup in the microwave Teflon vessels which is normally caused by the release of gases from the acids reacting with the samples. All samples were microwave digested following the digestion program in Table 1.

Table 1: Microwave parameters used for sample digestion.

Step	Power(W)	Pressure(psi)	Hold Time (Min)	Temp. (°C)	Ramp time (min)
1	1000-1600	200	20	180	25

7. Hot plate digestion

All the methods used for hot plate digestion were modified from the previous cited work and modification was done in all the three methods used. The acid mixtures were applied to the spiked sample of the same quantity for all the methods, and the vessels were covered with the watch glasses to reduce loss of volatile metals and contamination. All digestion solutions were filtered off with both the flat sheet filter paper (0.45 μ m) and the syringe filter (0.45 μ m) acrodisc and finally analyzed with ICP-OES.in order to determine the permitted levels of metals in water and sediment samples, as well as to identify areas of potential toxicity and the drinking water quality. The results of values obtained for industrial areas were above the non-industrial areas, showing the effects of industrial activities on pollution of environments [14].

Analytes	microwave	Hot plate						
	Added (mg/l)	Found(mg/l)	Sample Barhi	Added (mg/l)	Found(mg/l)	Sample Rai		
		Sample Barhi			Sample Barhi			
Ag	0	1.5±.1	1.7±0.1	0	0.8±0.1	0.6±0.1		
-	5±0.1	6.8±.1	5.3±0.1	5±0.1	4.3±0.1	3.9±0.2		
Al	0	27.5±1.4	26.3±0.2	0	13.2±0.2	11.2±0.2		
	20±0.2	45.8±0.3	44.3±0.2	20±0.2	22.1±0.2	21.1±0.2		
В	0	1.9±0.1	1.7±0.1	0	1.1±0.1	1.09±0.1		
	5±0.1	5.7±0.1	4.8±0.1	5±0.1	2.1±0.1	1.8 ± 0.1		
Bi	0	2.2±0.1	2.1±0.1	0	1.3±0.1	1.1±0.1		
	5±0.1	7.1±0.1	6.3±0.1	5±0.1	3.8±0.1	3.1±0.1		
Ca	0	28.2±1.7	27.3±0.3	0	1.6±0.3	1.2±0.2		
	20±0.2	48.2±0.4	47.4±0.4	20±0.2	28.1±0.4	22.1±0.3		
Cd	0	1.4±0.1	1.2±0.1	0	0.8±0.1	0.6±0.1		
	5±0.1	1.9±.1	1.3±0.1	5±0.1	1.1±0.1	0.9 ± 0.1		
Cr	0	34.8±1.5	32±1.3	0	14.1±0.4	12.1±0.2		
	20±0.2	52.5±0.1	51 ± 0.2	20±0.1	29.2±0.3	24.2±0.2		
Fe	0	180±2.6	182±2.5	0	147±1.3	138±0.7		
	20±0.2	220.6±0.2	200±0.4	20±0.2	188.1±0.2	171±0.1		
K	0	28.4±0.1	26.3±0.3	0	19.2±0.1	23±0.1		
	20±0.2	44.2±0.2	39.3±0.2	20±0.2	23.1±0.2	37±0.2		
Se	0	1.2±0.1	1.1±0.1	0	0.6±0.1	0.5±0.1		
	5±0.1	4.1±0.1	5.1±0.1	5±0.1	2.3±0.1	2.1±0.1		
Pb	0	0.1±0.01	0.3±0.1	0	0.07±0.1	0.06±0.1		
	20±0.2	11.2±0.3	7.8±0.2	20±0.2	6.2±0.2	5.4 ± 0.1		
Mg	0	23.2±0.1	21.1±0.2	0	14.3±0.1	13.2±0.1		
C	20±0.2	34.3±0.2	31.7±0.2	20±0.2	21.3±0.2	19.2±0.1		
Zn	0	49.2±2.1	43±0.2	0	26.3±1.1	22.1±0.1		
	20±0.2	68.4±0.2	58±0.2	20±0.2	37.4±0.2	32.1±0.1		
Mn	0	7.3±0.4	5.4±0.2	0	3.7±0.3	2.8±0.2		
	20±0.2	24.3±0.5	23.3±0.2	20±0.2	16.1±0.4	21.1±0.2		
Ni	0	17.4±1.3	11.3±0.8	0	11.2±0.3	9±0.1		
	20±0.2	39.2±0.3	34±0.1	20±0.2	26.3±0.2	23.1±0.1		
Na	0	28.3±0.2	24.2±0.2	0	21.1±0.2	18.2±0.2		
	20±0.2	45.7±0.5	42.2±0.2	20±0.2	24.2±0.4	21.2±0.3		

Table 2: Concentration of metals in sample 1 and sample 2 digested by microwave and hot plate technique.

8. Results and discussion

Microwave and hot plate digestion systems were used for the digestion of sample 1 and sample 2 employing three acid mixtures (HNO₃/H₂O₂, HNO₃/HCIO₄/H₂O₂ and aqua regia + H₂O₂) for heavy metal (Al, Ag, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) extraction. The capabilities of these methods for analysis of sample1 and sample 2 taking into account the information obtained were evaluated. Due to the vital role filtration methods play in the removal of undigested particulates from the sample matrix, proper filtration system to block or to trap unwanted particulate prior to analyte determination, must be used. The metal and element concentration in sample 1 (CETP-Barhi) and sample 2 (CETP-Rai) were analysed by ICP-OES based on instrumentation method. The experimental result with their concentrations in mg/L in depicted in Table 2. According to the Surface Water Quality Standard (IS; 2296) [15], the Environmental Protection Agency (EPA), the United States, and the World Health Organization (WHO), the concentration of group- 1 metals (Na, K, Mg, and Ca) was found to be within the acceptable range for outdoor bathing, (Graph a) fish culture irrigation, industrial cooling, and controlled waste disposal, with the exception of drinking (Figure 1), whereas water quality of Yamuna River in Delhi is more polluted [16]. Water quality of Yamuna in summer season is high polluted and unfit for drinking and other purpose [17]. The experimental value shown in graph b shows the water quality in sample 1 is poor in comparison to sample 2 due the industry in area Barhi is more instead of Rai which affect the water quality.

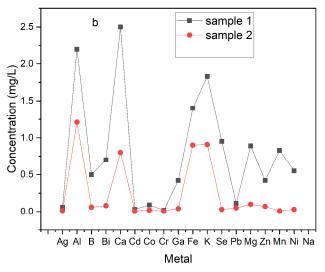


Figure 1: Graph (a) shows concentration of metal and trace element in sample 1 and sample 2.

9. Conclusions

Present research work gives information on concentration of metal in the sample from common effluent treatment plant(CETPs) Barhi and Rai. The probable sources of the heavy metals in the sample be caused by various industries outlet which comes to the common effluent treatment plant and also affected to the human health. The microwave and hot plate digestion methods (HNO₃/H₂O₂, HNO₃/HClO₄/H₂O₂ and aqua regia + H₂O₂) were successfully assessed and validated for the digestion of heavy metals (AI, As, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) using ICP-OES as the instrument for detection. Based on the results obtained demonstrated very good efficiency for the extraction of metals ions in influent wastewater.

Authors' contributions

The author read and approved the final manuscript.

Conflicts of interest

The author declares no conflict of interest.

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Data availability

No new data were created.

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