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STABILIZATION OF HEAVY METALS FROM ETP SLUDGE THROUGH PORTLAND CEMENT AND LIME

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ABSTRACT

The potential of stabilization technology for the safe disposal of hazardous wastes has wide spread recognition. This study aimed to evaluate the efficiency of portland cement and lime based stabilization technology for the secure disposal of hazardous waste containing toxic metals and pH. Stabilized sludge is a beneficial material that contains 1% portland cement and 2% lime, resulting in a durable substance. Before stabilization, ETP-A, ETP-B, ETP-C, and ETP-D exceeded the CPCB direct landfill limits for Zinc (Zn) in ETP-A, Lead (Pb) in ETP-B and ETP-D, and Hexavalent Chromium Cr+6 in ETP-C. The outcome decreases the levels of zinc, lead, and chromium hexavalent in the stabilized sludge. Enhancing the mixture with varying amounts of 2% lime and 1% portland cement will establish a highly alkaline environment and enhance the binding capacity. This study aims to minimize the cost of stabilizing wastewater treatment sludge by employing portland cement, and to ensure the safe disposal of the sludge. The resultant material effectively controlled the heavy metal concentration and the leachability capability.

KEYWORDS: Stabilization, ETP sludge's, Hexavalent chromium, Lead, Zinc, Lime & Cement.

GRAPHICAL ABSTRACT

INTRODUCTION

Industries produce a significant volume of effluents containing various hazardous substances depending on the industry type. Several chemicals are hazardous, persistent, and have various health impacts (Patel and Pandey, 2012; Amit et al., 2014). Sludge is a byproduct of Effluent treatment plants. Chemicals included in effluents accumulate to levels that can cause environmental issues, as per the MoEF and Hazardous Waste Management Rules and Transboundary 2008. Currently, significant amounts of chemical sludge are still present at the CETP (Common Effluent Treatment Plant) site and are waiting to be disposed of at a landfill. Researchers have developed hazardous waste treatment and disposal systems, including stabilization and solidification (S/S) (Spence and Shi, 2004). Solidification can occur through mechanical means, consolidating the trash into a monolith, without necessarily including a chemical interaction between the waste and the solidifying agents, which is then followed by disposal in a landfill.

Hazardous wastes are often treated using solidification/stabilization processes to reduce contaminant leaching before being disposed of on land. Cement-based systems are widely used as solidification/stabilization binders due to their cost-effectiveness, availability, and versatility (Murat and Yuskel, 2001; Idachaba et al 2001, Gong and Bishop, 2003). The S/S approach can transform highly mobile heavy metals into less mobile forms. This technology is applicable to various waste types that are not amenable to chemical, physical, or biological treatment (Gollmann et al., 2010). Many researchers have tested different methods to control the release of heavy metals from the sludge. The study used different amounts of leaching solutions and found that heavy metals might be extracted from the sludge (Naveen and Malik 2019). Yesil and Tugtas (2019) showed that liquid membranes can be utilized to recover heavy metals. Lasheen and Ammar's 2009 argument said that the stabilized sludge sample from the wastewater treatment facility had 100% of Cd, 90.65% of Cu, 99.86% of Cr, 99.74% of Fe, 82.58% of Mn, 85.15% of Ni, and 75.17% of Zn in immobilized metal forms. After showing a significant reduction in metal content due to chemical treatment. Tan et al. (2021), Muneswara Rao et al. (2023), and Wang et al. (2024) shown that using different mixtures of cement and lime can effectively decrease the leaching of heavy metals over time. A similar study was conducted, and a significant reduction in leachability was noted.

This study intends to demonstrate the efficacy of stabilization technique in eliminating heavy metals from ETP sludge using a selected cement and lime concentration mixer. The research utilized a cement and lime concentration mixer with concentrations of 1% and 2%. The water leaching test is performed post-stabilization to identify any failed parameters such as Zn, Pb, and Cr+6, which distinguishes it from conventional tests. A stabilization software can be created on-site to process comparable sludge kinds by simulating the Waste Load Allocation and determining the parameters that are exceeded.

MATERIALS AND METHODS

The primary constituents of trash generated by Common Treatment Plants (CETP) or Effluent Treatment Plants (ETP) in different sectors. It is widely recognized that when treating industrial effluents, sludge is produced as a by-product. The sludge is created through different treatment phases and comprises solid materials extracted from the wastewater. The composition and properties of effluent sludge may differ based on the kind of wastewater treatment and the treatment methods used.

Sample Collection

We gathered four types of Effluent Treatment Plants Sludge from various areas in the industrial zone of Delhi, India. Each sample, weighing five kilograms, was labelled as ETP 'A', ETP 'B', ETP 'C', and ETP 'D'. The samples are correctly labeled on their sampling covers for improved identification and tracking, then stored in the laboratory at 4°C.

Sludge Characterization

Sludge characterization was conducted both before and after stabilization. The physico-chemical parameters examined included physical state, color, pH, bulk density, calorific value, moisture content, ash content, and heavy metals such as hexavalent chromium (Cr +6), zinc (Zn), lead (Pb), copper (Cu), cadmium (Cd), and nickel (Ni). The stabilizing process was carried out to address parameters that exceeded the CPCB guidelines.

Physical state and color are visually perceived. A 10 g sludge sample was mixed with 100 ml of distilled water and stirred continuously in a shaker for 1 hour. The mixture was then allowed to settle, and the supernatant was used to measure the pH using a pH meter (Orion Star, India) following the guidelines of the CPCB solid waste manual (Hema and Suneel 2008). The experiment on calorific value was carried out using a Bomb Calorimeter (Parr, India). A 1g sample of uniformly mixed dry sludge was assessed according to IS 1350 part 2 (1994).

Sample Preparation for Water Leaching Test

Weighed 50g of sludge sample was placed into an extraction vessel, mixed with 500ml of distilled water, and then sealed with a lid for identification. Extraction vessels are placed in the TCLP agitator and rotated at 30 ± 2 RPM for approximately 23 ± 1 hours. After the rotation process is completed, the sample will be filtered using a 41 mm Whatman filter paper in a conical flask. 100 ml of the supernatant will be collected. Acid digestion will then be carried out using a 1:3 ratio of Nitric acid to Hydrochloric acid until the sample digestion reaches 20 ml. It will then be filtered and adjusted to a final volume of 100 ml.

Sample Preparation for Total Metals

2.5g of sample was mixed with 100 ml of distilled water in a conical flask and placed in a rotary shaker (Remi, India) for 20 minutes. Acid digestion was carried out using a mixture of Nitric acid and Hydrochloric acid in a 1:3 ratio until the sample digestion reached 20 ml. The solution was then filtered and diluted to 250 ml. The results are reported in mg/kg.

Heavy Metal Analysis

The samples were analysed for Zn, Pb, Cd, Cu, and Ni using an Atomic Absorption Spectrophotometer (AAS) from Shimadzu, India, at various wavelengths. The analysis for Cr+6 was conducted on the digested sample using a Spectrophotometer (Hach DR 5000, India) at a specific wavelength.

Stabilization Trails

We treated sludge samples with lime and Portland cement at various doses and proportions of ETP sludge samples to stabilize them. The details are presented in Table 1.

Table 1: Stabilization trails details of ETP-A, ETP-B, ETP-C and ETP-D Sludge samples.

S.No.	ETP – A		ETP – B		ETP - C		ETP – D	
	Lime (%)	Cement (%)	Lime (%)	Cement (%)	Lime (%)	Cement (%)	Lime (%)	Cement (%)
Trail - 1	2	1	2	1	2	1	2	1
Trail - 2	2	2	2	2	2	2	2	2
Trail - 3	2	3	2	3	2	3	2	3
Trail - 4	2	4	2	4	2	4	2	4
Trail - 5	2	5	2	5	2	5	2	5

Following the modifications made in Yonghua and Changquan 2013, Trail-1, we initially used 50g of each ETP sludge (ETP-A, ETP-B, ETP-C, and ETP-D) and added some water to create a slurry. Subsequently, 1g of lime was evenly mixed with all ETP sludge samples. Additionally, 0.5g of Portland cement was added, thoroughly mixed, and left to cure for 8 hours. Trail-2, Trail-3, Trail-4, and Trail-5 were stabilized using various concentration cements listed in Table-1. We will solely analyse the remaining data from Trail-1 that has not been included in this paper for this experiment.

Table 2: Analytical techniques used for sludge sample analysis through IS, ASTM and USEP methods.

Parameter	Unit	Method
Physical State	-	Visual Observation
Color	-	Visual Observation
pH	-	USEPA 1998 SW-846,9045 C
Bulk Density	gm/cc	ASTM D 5057 -90
Calorific value	cal/g	IS 1350 (Part-2) 2000
Moisture content	%	IS 326 Part 21 : (2001)
Ash	%	ASTM D1102-84
Hexavalent Chromium (Total)	mg/Kg	USEPA 1998, SW-846- 3050B; 7196 A
Hexavalent Chromium (WLT)	mg/L	USEPA 1998, SW-846- 7196 A
Zinc (Total)	mg/Kg	USEPA 1998, SW-846- 3050B; 7950
Zinc (WLT)	mg/L	USEPA 1998, SW-846- 7950
Lead (Total)	mg/Kg	USEPA 1998, SW-846-3050B; 7420
Lead (WLT)	mg/L	USEPA 1998, SW-846-7420
Copper (Total)	mg/Kg	USEPA 1998, SW-846- 3050B; 7210
Copper (WLT)	mg/L	USEPA 1998, SW-846- 7210
Cadmium (Total)	mg/Kg	USEPA 1998, SW -846 – 3050B; 7130
Cadmium (WLT)	mg/L	USEPA 1998, SW -846 – 7130
Nickel (Total)	mg/Kg	USEPA 1998, SW -846 – 3050B; 7520
Nickel (WLT	mg/L	USEPA 1998, SW -846 – 7520

Statistical Analysis

The statistical analysis was conducted on a personal computer using triplicate data, and the standard error was calculated using Microsoft Excel version 2011 (Varaprasad et al., 2021).

RESULTS AND DISCUSSION

In the Present work, All ETP sludge samples (ETP-A, ETP-B, ETP-C, and ETP-D) were initially solid. ETP-A was dark gray, while the others were brown. After stabilization, all samples turned gray. Additionally, bulk density measurements were taken both before and after stabilization. The initial values were ETP-A 0.72 g/cc, ETP-B 1.21 g/cc, ETP-C 0.81 g/cc, and EPT-D 0.68 g/cc. The post-stabilization values were 0.81 g/cc, 1.24 g/cc, 0.83 g/cc, and 0.73 g/cc, respectively. The calorific value of all the sludge samples is significantly below 50 cal/g as per CPCB standards. Furthermore Moisture content was measured in all sludge samples. The results are steadily decreasing after stabilization, as seen in Table-2. Samaras et al. (2008) and Yonghua and Changquan (2013) highlighted the importance of lime and fly ash in controlling moisture. Ash content analysis was conducted on sludge samples before and after stabilization. It was observed that the ash content increased in the samples after stabilization compared to before stabilization. The results are presented in Table-2. The term pH indicates whether a sample is acidic or alkaline. We evaluated eight samples in the current study, all of which fall within the acceptable range set by the CPCB, which is between 4 to 12. (Bina et al., 2004) utilized varying dosages of lime to raise the pH.

Chromium in its +6 oxidation state is a very hazardous metal associated with causing cancer in humans by prolonged inhalation. It is also harmful to aquatic organisms even at low concentrations, according to the US Environmental Protection Agency (EPA) in 1998. Our investigation of Cr+6 revealed that all sludge samples had a minimal quantity, less than 0.1 mg/kg, both before and after stabilization. Zinc was quantified using Atomic Absorption Spectroscopy (AAS). The zinc level in an ETP-1 sludge sample was higher (272 mg/kg) before stabilization compared to all ETP samples before and after stabilization, as shown in Figure 1. Furthermore, the zinc concentration in all sludge samples decreased gradually following stabilization with a mixture of lime and cement, as depicted in Figure 1b. Lasheen and Ammar (2009) found that treating sludge with cement reduced the Zn concentration in the sludge. Additionally, we analyzed the presence of lead (Pb) in all samples of ETP sludge. The concentration of Pb varied from 2.29 mg/kg to 0.77 mg/kg before stabilization. There was a gradual decrease observed in lead concentrations from 0.3 mg/kg to 0.11 mg/kg following stabilization. All samples were found to be within the CPCB recommendations limit of 5 mg/kg.

Most metal ions form stable complexes when combined with lime and portland cement. The study examined the effectiveness of mixing it with different amounts to remove pollutants from ETP sludge (Table 1). Analyzed Cu levels are under the 25 mg/kg CPCB standards. In pre-stabilization ETP sludge samples, Cu levels vary from 11 mg/kg to 1.57 mg/kg. Combining lime with plain cement led to a reduction in concentrations. Naveen and Malik's (2018) experiment showed how leaching solutions bind to Cu metal and control concentrations when mixed with ETP sludge. Analysis of Cd and Ni in all four ETP sludge samples, both before and after stabilization, showed that they met the specified parameters outlined in the CPCB guidelines. The levels of Cd and Ni in the ETP sludge samples decreased gradually over time. Similarly, this was effectively shown in polluted soils containing different chemical mixtures (Bilgin and Tulun, 2016).

Table 3: Analysis results before and after Stabilization of ETP-A, ETP-B, ETP-C and ETP-D sludge sample.

S.No.	Test parameter	UOM	ETP - A		ETP – B		ETP - C		ETP – D		Landfill CPCB Limits
			Before	After	Before	After	Before	After	Before	After	
1	Physical state	-	Solid	Solid	Solid	Solid	Solid	Solid	Solid	Solid	Solid/Liquid
2	Colour	-	Dark grey	Grey	Brown	Grey	Brown	Grey	Brown	Grey	-
3	Bulk density	g/cc	0.72	0.81	1.21	1.24	0.81	0.83	0.68	0.73	-
4	Calorific value	cal/gm	1771	<50	<50	<50	<50	<50	<50	<50	<2500
5	Moisture	%	25	13	2.1	0.4	10	5.3	24	16.1	-
6	Ash	%	74	84	86	89	88	93	74	79	-
7	pH		7.87	8.13	7.9	8.25	7.11	8.48	7.8	8.42	>4 to <12
	Total Metals										
8	Hexavalent Chromium	mg/Kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 5
9	Zinc	mg/Kg	**272**	**21**	16.8	2.1	BDL	BDL	15.4	1.7	< 250
10	Lead	mg/Kg	1.67	0.3	2.29	0.23	0.72	0.11	1.83	0.22	< 5
11	Copper	mg/Kg	11	2.1	2.45	0.44	2.71	0.39	1.57	0.3	< 25
12	Cadmium	mg/Kg	0.47	0.2	0.15	0.03	0.05	0.01	0.24	0.12	< 1
13	Nickel	mg/Kg	9.15	1.4	2.67	0.48	12	1.9	3.77	0.52	< 20
	Water Leachate										

	Test										
14	Hexavalent Chromium	mg/L	< 0.1	< 0.1	< 0.1	< 0.1	**3**	**0.32**	< 0.1	< 0.1	< 0.5
15	Zinc	mg/L	**18**	**8.45**	5.7	3.6	BDL	BDL	5.75	3.1	< 10
16	Lead	mg/L	0.63	0.22	**2.89**	**1.82**	0.61	0.18	**2.52**	**1.76**	< 2
17	Copper	mg/L	0.38	0.11	1.04	0.56	0.18	0.08	2.61	0.76	< 10
18	Cadmium	mg/L	**0.14**	0.1	0.14	0.11	0.04	0.02	0.17	0.12	< 0.2
19	Nickel	mg/L	1.93	1.1	2.11	1.26	1.78	0.81	1.8	0.98	< 3

The current study primarily aims to reduce the leaching of harmful metals from ETP sludge by utilizing inexpensive reagents. The experiment was carried out in April, May, and June 2023. Three harmful elements, specifically Cr+6, Zn, and Pb, exceeded the Water Leachability Test Limits for landfills. These three hazardous metals are known for their chronic effects on animals and humans.

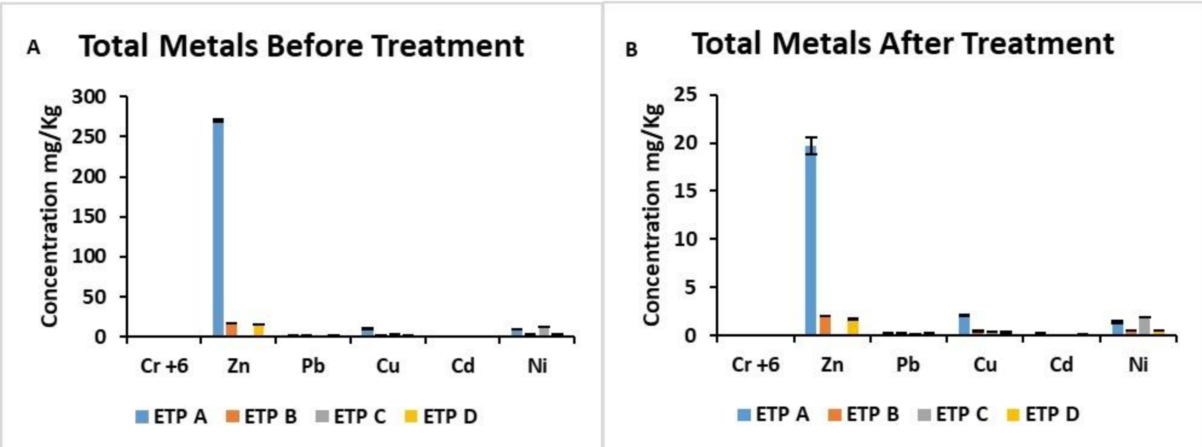

Figure 1: Graphical representation of total metals concentration before and after treatment of ETP sludge samples.

Investigating how lime and cement can restrict the leaching of heavy metals by acting as catalysts and binders for the heavy metals. The UV-visible spectrophotometer examination showed that the concentration of Cr+6 in the ETP-C sludge sample was 3.03 mg/L greater than the concentrations in the ETP-A (0.06 mg/L), ETP-B (0.04 mg/L), and ETP-D (0.04 mg/L) sludge samples. These values exceed the stated CPCB limit of less than 0.5 mg/L. Singh and Singh (2002) ETP sludge samples were treated with an acidic aqueous solution to control Cr+6. Upon stability, the Zn and Pb concentrations in the ETP sludge samples exceeded the defined CPCB criteria and were steadily decreasing. Prior to stabilization, the Zn contents in the ETP-A sludge sample are 18mg/L. Asavapisit and Chotklang (2004) demonstrated that alkali activated pulverized fuel ash can control the concentration in electroplating ETP sludge samples. The Pb concentration in ETP-B and ETP-D sludge samples was 2.89 mg/L and 2.52 mg/L, respectively. The ETP sludge sample underwent testing in the AAS both prior to and during stabilization. The Cu, Cd, and Ni test findings met the specified limits of <10 mg/L, <0.2 mg/L, and <3.0 mg/L, respectively. Pandey et al. (2012) shown that by working with various sludge samples and oxides like SiO2, Al2O3, Fe2O3, MgO, and SO3, heavy metal concentrations such as Cu, Cd, and Ni may be reduced. Lasheen and Ammar conducted research in 2009, while Patel and Pandey did so in 2012, using varying quantities of Portland cement to effectively control the leaching of heavy metals. Comparable outcomes were achieved in the leachability test once the ETP samples reached a stable state. The WLT limits also contain factors such as Cu, Cd, and Ni. The results are shown in Table 3.

Effects of Toxic metals

If not treat the aforementioned heavy metals can seep into adjacent water bodies. It leaches to contamination of surface water, ground water threats to aquatic ecosystems and potentially impacting drinking water.

Hexavalent Chromium, Lead & Zinc: (Cr^{+6}, Pb and Zn)

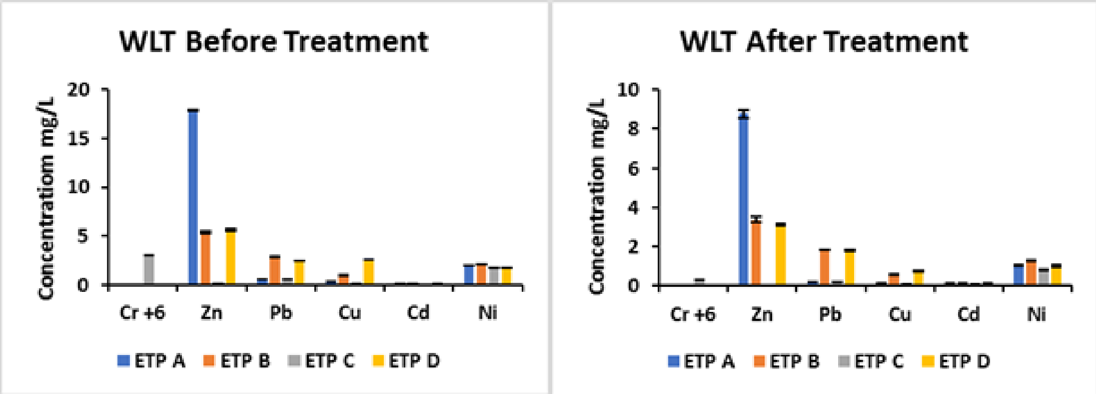

Figure 2: Graphical representation of WLT analysis before and after treatment of ETP sludge samples.

Hexavalent chromium is highly poisonous and carcinogenic type of chromium that can severe water pollution and represent a concern to human health. These metals stay in the soil for extended durations providing dangers to plant growth and potentially entering the food chain (Sherene ., 2010). These metals in soil can severely effect soil fertility and disturb soil microbial populations, hurting overall ecosystem heath. These metals are very harmful to wide range of creatures including aquatic organism, plants and soil microbes. If concentrations raised in the environment can cause to impaired growth, reproductive issues and even mortality in exposed balance and affects biodiversity in impacted habitats. To reduce the environmental consequences of Hexavalent chromium, Lead and Zinc in ETP Sludge's. It's to execute acceptable and cost efficient best mechanism for treatment of this metal stabilization and disposal approach as environmental way (Mitra et al., 2022).

CONCLUSIONS

The reagents such as Lime and Portblend Cement are utilized for stability of waste removal of Zinc, Lead and Hexavalent chromium for immobilization in different ETP sludge's. The following are findings that can arise the experimental inquiry.

i) In leachability of these metal tests finally Lime 2 % and Cement 1 % achieved our landfill criteria limitations. The maximum removal efficiency of Zinc, Lead and Hexavalent chromium metals optimization circumstances was found to be 96 and 99 % correspondingly.

ii) Final cost of stabilization also extremely low cost (Rs.220 per MT) for treatment of ETP sludge's usage of lime and cement economically after this studied.

iii) Based on the experimental results, it can be stated that economically feasibility of reagents may be successfully for immobilization of hazardous metals zinc, lead and hexavalent chromium present in the ETP sludge's.

iv) Toxic metals eliminated in different ETP sludge's with the above reagents appears to be technically possible, eco-friendly and with high efficiency additionally it helps to lessen the environmental contamination of soil and water.

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