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Retention Behaviour of Heavy Metals from Industrial Sludge Amended with Admixtures to Use Them as Liners for Landfill Facilities

S. S. S. Saranya*†, S. N. Maya Naik* and Shankara**
*Department of Civil Engineering, BMS College of Engineering, Bengaluru, India
**Department of Civil Engineering, Amrita School of Engineering, Bengaluru, India
†Corresponding author: S. S. S. Saranya; saranyasagiri8@gmail.com

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ABSTRACT

The solidification of contaminants within the soil/waste has proved to be a versatile technique to de-contaminate them and make them usable for several applications. In this method, the development of binder provisions leads to the conversion of the environmentally unstable condition of waste materials into a nearly stable material. Further, these materials pose a minimum threat that can be absorbed into the environment. Normally lime/cement and other pozzolanic materials are used as binder materials. In this work, it is proposed to use the efficiency of binding fly ash to improve the unconfined compressive strength (UCC) of soils, particularly during the curing period. This is because improvement in strength is a reflection of the improvement of bonding soil particles. Fly ash as the main source material, in addition to a minor proportion of cement and lime, is used to determine the strength. UCC test results revealed that as the percentage of fly ash increases there is an increase in compressive strength. It is also observed that with an increase in lime content and an increase in cement content, the UCC strength also increases. The strength in cement-stabilized compacted specimens is more compared to lime-stabilized mixtures. To confirm that the improvement in strength is related to the solidification of contaminated metals, particularly for soils containing copper and chromium, the stabilized mixture is tested for the leaching of these metals. Leaching tests were conducted on various stabilized mixtures at different curing periods. The leachate was examined for metal ion concentration using Atomic Absorption Spectrophotometer. The leaching behavior of heavy metals from different proportions of soil matrix revealed that with an increase in lime or cement percentage, a decrease in leachability is observed. It is found that the leaching of heavy metals from cement-stabilized soils was lower than in lime mixture combinations. However, minimum strength improves the solidification and retention of heavy metals effectively.

INTRODUCTION

Urbanization/industrialization and the economy of a country are always interconnected. Industrial operations are linked to environmental pollution. Therefore, various types of waste generated in the industry require treatment for their disposal facilities. A treatment facility concentrates/separates all the wastes and generates sludges and solid wastes. These end products at treatment facilities, are either hazardous or non-hazardous but consist of organic and/or inorganic substances (Voglar & Lestan 2011). Developed/industrialized countries export their wastes to developing countries due to their strict environmental regulations that make waste management in developed countries expensive compared to exporting wastes to developing countries where environmental regulations are lax and waste disposal is safe. Disposal and re-processing of these wastes have resulted in various contamination issues in soils in developing countries (Moon & Dermatas 2006). The impact of hazardous wastes on human wellness and strict policies for disposing of hazardous waste on land is leading to the development of scientific management of hazardous waste facilities like sanitary landfill areas, S/S techniques, etc. Methods to dispose of industrial waste have their limitations based on the properties of wastes, policies, the cost involved, and technology. The S/S technology has given relief in many directions from all the limitations of industrial waste treatment disposals and their complexities. Heavy metals like nickel, arsenic, copper, chromium, cadmium, zinc, and mercury are extremely toxic even at low concentrations or beyond the pollution control board's limits (Minocha et al. 2003, Wentz 2005).

Previous works on solidification/stabilization (S/S) processes have clearly shown that the technique applies to most metallic waste streams. This technique involves blending the waste along a binder material to enrich the

ORCID details of the authors:

S. S. S. Saranya
https://orcid.org/0000-0003-4919-366X

physical properties of the waste and immobilize contaminants that are a threat to humans and the environment (Sivapullaiah & Arif 2010, Malviya & Chaudhary 2006).

The solidification/stabilization technique involves physical and chemical actions to produce environmentally stable substances with enhanced pollutant holding capacity. Wastes containing inorganic contaminants normally require pre-treatment before disposing of in a sanitary landfill (Voglar & Lestan 2011, Shankara et al. 2012). The solidification/stabilization technique is being used all over the world to arrest the harmful contaminants that are present in various waste streams particularly industrial sludge by amending with fly ash, lime, and cement (Shankara et al. 2015, 2014a, 2014b). The challenging part of S/S technology is to quantify the reduction of negative environmental impacts in the real world after the treatment, proper disposal, reuse options, and so on. It is documented that the S/S technology is useful to treat sludge, solid waste contaminated sites, and other waste streams containing harmful heavy metals and metalloids. The in-situ steps are the mixing of binders and other materials directly with said waste streams/contaminated soils to obtain solid material that has transformed into low leachability characteristics compared to the original state (Venkata Ramaiah et al. 2014). The focus of the present work is to explore the practicability and efficacy of fly ash, soil, and other binders in stabilizing the toxic sludge collected from the Federal mogul industry. Unconfined compressive strength (UCC) tests were conducted for stabilizing the sludge and to obtain a minimum compressive strength required for disposal at a landfill site. Leaching tests were carried out to assess the leaching potential of metal ions from the sludge and the resulting leachates were analyzed for various heavy metal ions.

MATERIALS AND METHODS

In this paper an effort has been made to provide important insights on the compressive strength of the stabilized mixes and retention behavior of copper and chromium ions that are present in industrial sludge and the same has been evaluated using additives such as fly ash, lime, and cement blended with soil. The leaching tests were performed on stabilized mixtures at different curing periods for various combinations of additives and sludge.

Sludge

The sludge used in the present study was collected from the Federal-Mogul industry, situated in Yelahanka, Bangalore, with the generous consent of the Karnataka State Pollution Control Board. The toxic sludge obtained was so rich in chromium ions that it was considered in the category of hazardous waste and needs special care during the stabilization/solidification process so that it can convert the unstable condition of heavy metal ions into a stabilized state of ions within the stabilized soil matrix. Further, the stabilized industrial sludge can be disposed of in scientifically designed sanitary landfills. The collected sludge was characterized for p^H, moisture content, and concentration of heavy metal ions at the Bangalore test house. Table 1 shows the results of the characterization of the sludge.

Table 1: Properties of industrial sludge.

S. No	Parameter	Unit	Value
1.	pH	-	2.7
2.	Moisture content	[%]	9.5
3.	Chromium	[$mg.kg^{-1}$]	90.5
4.	Nickel	[$mg.kg^{-1}$]	7.3
5.	Copper	[$mg.kg^{-1}$]	24.2
6.	Aluminum	[$mg.kg^{-1}$]	82.9

Source results obtained from Bangalore Test House, Bangalore

Fly Ash

Fly ash is a by-product of coal-fired power stations. Fly ash

Table 2: Physical properties of fly ash (Venkata Ramaiah et al. 2014).

S. No.	Physical Property	Value
1.	Specific gravity G	2.0
2.	Liquid Limit (LL) [%]	35.0
3.	Plastic Limit(PL) [%]	--
4.	Plasticity Index(PI) [%]	--
5.	Shrinkage Limit (SL) [%]	18.5
	Compaction Characteristics	
1.	Maximum dry density (MDD) [$KN.m^{-3}$)	11.7
2.	Optimum moisture content (OMC) [%]	25.0
	Grain size distribution	
1.	Gravel [%]	00.0
2.	Sand [%]	58.0
3.	Silt and clay [%]	42.0

Table 3: Chemical composition of fly ash.

S. No.	Constituents	Percentage [%]
1.	SiO_2	61.1
2.	Al_2O_3	28.0
3.	TiO_2	1.3
4.	Fe_2O_3	4.2
5	MgO	0.8
6.	CaO	1.7
7.	K_2O	0.2
8.	Na_2O	0.2
9.	L.O.I	1.4

Table 4: Properties of black cotton soil.

S. No.	Property		Value
1.	Specific gravity G		2.7
2.	Grain size Analysis	Clay (C) [%]	54.2
		Silt (M) [%]	16.1
		Sand (S) [%]	26.7
		Gravel (G) [%]	3.3
3.	Consistency limits	Liquid limit (LL) [%]	49.1
		Plastic limit (PL) [%]	18.4
		Plasticity index(PI) [%]	21.3
		Shrinkage limit (SL) [%]	8.9
4.	Compaction characteristics (Proctor's)	Optimum moisture content (OMC) [%]	22.0
		Maximum dry density (MDD) $g.cm^{3-}$	2.1

used in the present research work is collected from Raichur Thermal Power Station (RTPS), in Karnataka, India. The fly ash used was classified as class "F" category and was grey in color. The physical properties and chemical composition of fly ash are given in Tables 2 and 3.

Soil

The black cotton soil (BCS) used was collected from Harihara in Karnataka state. The (BCS) is preferred as it contains more silt and clay which conveys plasticity properties in the process of stabilization. The topsoil was removed to a depth of 0.5m before the soil samples were taken by the undisturbed sampling method. Before pulverizing, the soil sample was oven-dried for 24 h to obtain soil particles that could pass through a sieve with a 4.75 mm aperture. The compaction characteristics and physical properties of the BCS are given in Table 4.

Cement and Lime

The cement and lime used in the present study were procured from local distributors in Bangalore, Karnataka.

Preparation of Sample and Mix Proportions

The sludge, fly ash and soil were oven-dried for 24 h at 105°C and sieved using 4.75 mm to avoid the coarser materials. Different proportions were prepared by mixing all the ingredients as shown in Table 4a. A total of 20 mixes were prepared with one control set of mixes in each series.

The series of mixes are labeled indicating Fly ash: F, Soil: S, Sludge: SL, Lime: L, and Cement: C. For example,

Table 4a: Mix proportions.

S. No.	Specimen	Fly ash	Soil	Sludge	Lime	Cement
		%	%	%	%	%
1.	10F90S	10	90	-	-	-
2.	10F85S5SL	10	85	5	-	-
3.	10F80S10SL	10	80	10	-	-
4.	10F75S15SL	10	75	15	-	-
5.	10F70S20SL	10	70	20	-	-
6.	20F80S	20	80	-	-	-
7.	20F75S5SL	20	75	5	-	-
8.	20F70S10SL	20	70	10	-	-
9.	20F65S15SL	20	65	15	-	-
10.	20F60S20SL	20	60	20	-	-
11.	30F40S30SL	30	40	30	-	-
12.	30F36S30SL4L	30	36	30	4	-
13.	30F32S30SL8L	30	32	30	8	-
14.	30F28S30SL12L	30	28	30	12	-
15.	30F24S30SL16L	30	24	30	16	-
16.	30F40S30SL	30	40	30	-	-
17.	30F36S30SL4C	30	36	30	-	4
18.	30F32S30SL8C	30	32	30	-	8
19.	30F28S30SL12C	30	28	30	-	12
20.	30F24S30SL16C	30	24	30	-	16

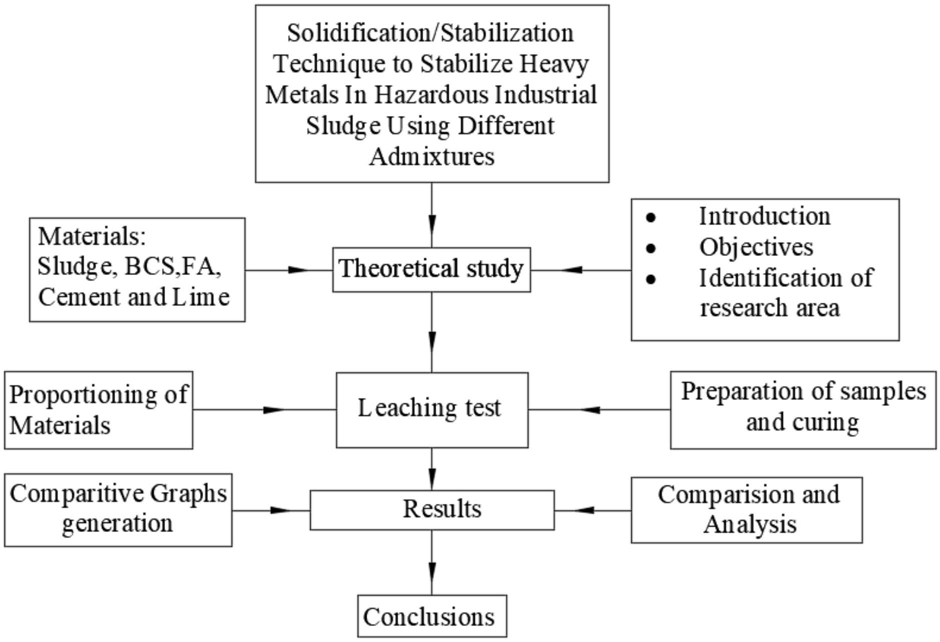

Fig. 1: Methodology used in the study.

30F36S40SL4C indicates a mixture of 30% fly ash, 36% soil, 40% sludge, and 4% cement.

Each combination of the series was compacted in a split mold and prepared cylindrical specimens (7.6 mm and diameter of 3.8mm) and cured for 1, 7, 14, and 28 days. The specimens are stored in sealed sample bags and just after the predetermined curing periods, the unconfined compressive strength was determined. The tested specimens are powdered and passed through a 4.75 mm sieve and the leaching test was conducted using the ASTM D3987-85 procedure. The leachate obtained after the test was analyzed for concentrations of the ions using an Atomic Absorption Spectrophotometer (AAS). The methodology followed in this study has been adopted and presented in the form of a flow diagram presented in Fig. 1.

RESULTS AND DISCUSSION

After completing the experimental work, the results obtained are tabulated concerning specified series, and the values are used to generate graphs. UCC test was conducted on different proportions of black cotton soil, sludge, and fly ash with and without the addition of lime in a small percentage, and the strength parameter is investigated. Among various recommendations for the minimum compressive strength required for solid waste disposal at landfill sites, 0.3 MPa is one according to the Resource Conservation and Recovery Act (RCRA). The efficiency and practicability of the containment of the heavy metal ions in the S/S method are measured by the leaching test of stabilized mixtures. There are various leaching-related documents available in this area, but it is very difficult to get the complex leaching behavior that involves surface complexation reactions.

Unconfined Compressive Strength of Fly Ash, Soil and Sludge Mixtures with Lime and Cement as Additives

Unconfined compressive strength of soil and sludge mixtures with 10% fly ash: Unconfined compressive strength tests were conducted on mixtures with 10% fly ash and varying proportions of soil and sludge. Sample "10F90S" is a blank (control) without the addition of sludge whereas for other samples the percentage of sludge increased from 5 to 20% and the respective strength of the specimens at 1, 7, 14, and 28 days of curing are presented in Table 5.

Fig. 2 shows the trend of UCC concerning varying days of curing. It is observed that the highest compressive strength is obtained for the control sample (10F90S) whereas the

Table 5: Unconfined compressive strength of soil and sludge mixtures with 10% fly ash.

Series of mixes	UCC strength in kPa			
	1-Day	7-Day	14-Day	28-Day
10F90S	184	211	242	255
10F85S5SL	176	202	230	247
10F80S10SL	163	190	222	235
10F75S15SL	151	176	201	219
10F70S20SL	134	160	179	192

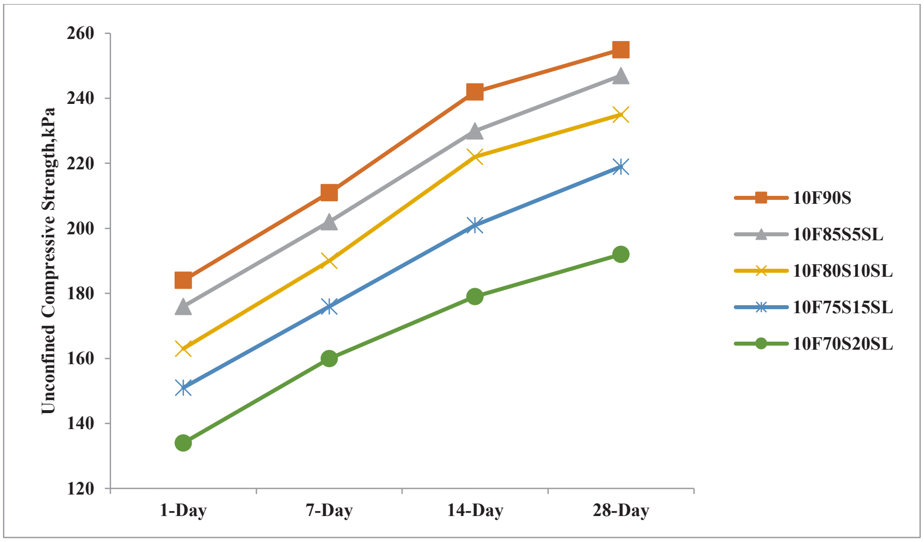

Fig. 2: Unconfined compressive strength of soil and sludge mixtures with 10% fly ash.

Table 6: Unconfined compressive strength of soil and sludge mixtures with 20% fly ash.

Series of mixes	UCC strength in kPa			
	1-Day	7-Day	14-Day	28-Day
20F80S	252	277	305	318
20F75S5SL	238	268	297	308
20F70S10SL	219	247	272	286
20F65S15SL	199	219	240	258
20F60S20SL	194	211	232	243

addition of 5,10,15 and 20% of sludge by weight resulted in a decrease in UCC strength. Reduced compressive strength with an increase in sludge percentage can be due to the changes in pore fluid viscosity and the effect of enhanced lubrication by an excessive amount of moisture present in the sludge. Also, the decrease in strength can be due to the physio-chemical effects caused by the dielectric constant.

Unconfined compressive strength of soil and sludge mixtures with 20% fly ash: UCC strength test results for a set of series with 20% fly ash and varying proportions of soil and sludge are shown in Table 6. Sample "20F80S" is a blank (control) without the addition of sludge, whereas for other samples the percentage of sludge increased from 5 to 20% by weight. From Fig. 3, plotted between UCC strength and the curing period, it is noticed that the compressive strength decreases with an increase in the percentage of sludge.

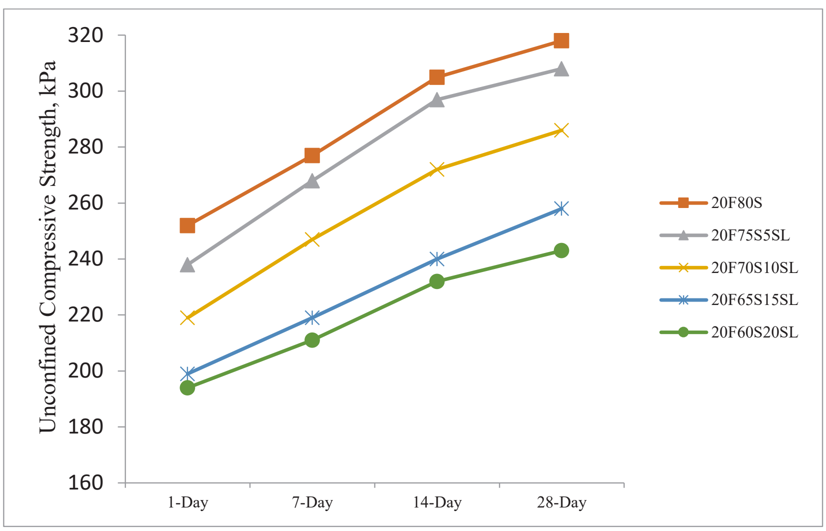

Fig. 3: Unconfined compressive strength of soil and sludge mixtures with 20% fly ash.

Table 7: Unconfined compressive strength of soil, sludge mixtures with 30% fly ash and varying percentage of lime.

Series of mixes	UCC strength in kPa			
	1-Day	7-Day	14-Day	28-Day
30F40S30SL	202	228	250	265
30F36S30SL4L	218	243	271	284
30F32S30SL8L	236	258	284	299
30F28S30SL12L	262	281	310	320
30F24S30SL16L	274	295	321	336

Table 8: Unconfined compressive strength of soil, sludge mixtures with 30% fly ash, and varying percentage of cement.

Series of mixes	UCC strength in kPa			
	1-Day	7-Day	14-Day	28-Day
30F40S30SL	203	229	251	265
30F36S30SL4C	225	238	269	288
30F32S30SL8C	245	262	294	325
30F28S30SL12C	277	288	315	348
30F24S30SL16C	290	304	337	375

Unconfined compressive strength of soil, sludge mixtures with 30% fly ash, and varying percentage of lime: UCC strength test results for the soil, sludge mixtures with 30% fly ash, and varying percentages of lime are shown in Table 7. Sample "30F40S30SL" is a blank (control) sample without the addition of lime, whereas for other samples the percentage addition of lime increases from 4 to 16% by weight. From Fig. 4, plotted between the unconfined compressive strength with respect to 1, 7, 14, and 28 days of curing, it is noticed that with an increase in lime percentage, the pozzolanic compounds are formed that bind the soil particles and results in an increase in compressive strength.

Unconfined compressive strength of soil, sludge mixtures with 30% fly ash, and varying percentage of cement: UCC strength test was carried out for the soil, sludge mixtures with 30% fly ash, and varying percentage of cement. Sample "30F40S30SL" is a blank (control) sample without the addition of cement, whereas for other samples the percentage addition of cement increases from 4 to 16% by weight and the respective compressive strengths at 1, 7, 14, and 28 days are shown in Table 8. From Fig. 5 plotted between UCC strength and the curing period, it is observed that the compressive strength increases with an increase in the cement percentage from 4 to 16, which results in the formation of pozzolanic compounds that bind the soil particles and result in strength increment.

Leaching of Chromium in Soil, Sludge Mixtures with 30% Fly Ash, and Varying Percentage of Lime

The soil, sludge mixtures with 30% fly ash and varying percentage of lime starts with a control specimen of a mixture of 40% of BCS, 30% of fly ash, and 30% of industrial sludge. The other four specimens represent the percentage of lime increased from 4 to 16% as given in Table 4.

It can be seen from Fig. 6 that as the percentage of lime and the curing period increases the leaching of chromium

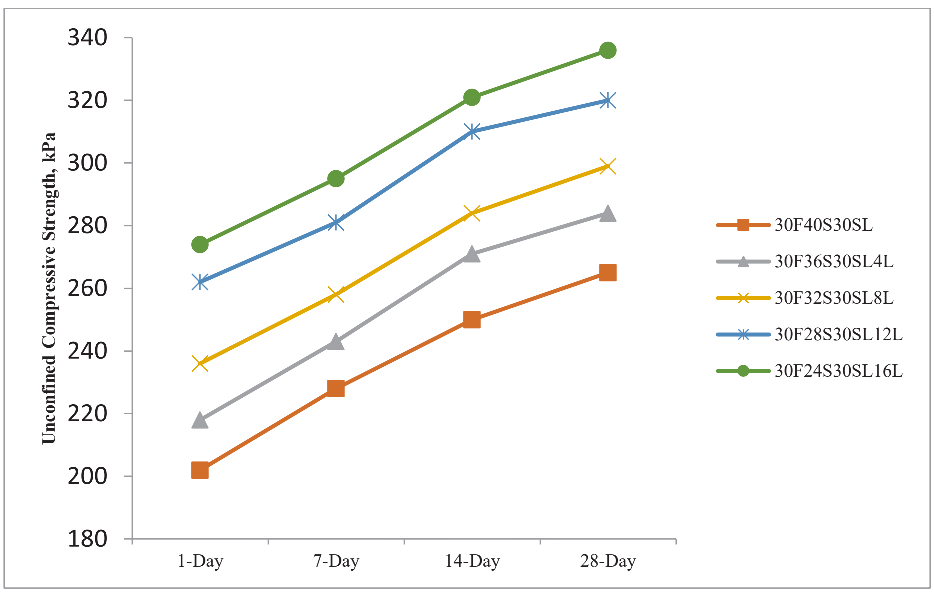

Fig. 4: Unconfined compressive strength of soil, sludge mixtures with 30% fly ash, and varying percentage of lime.

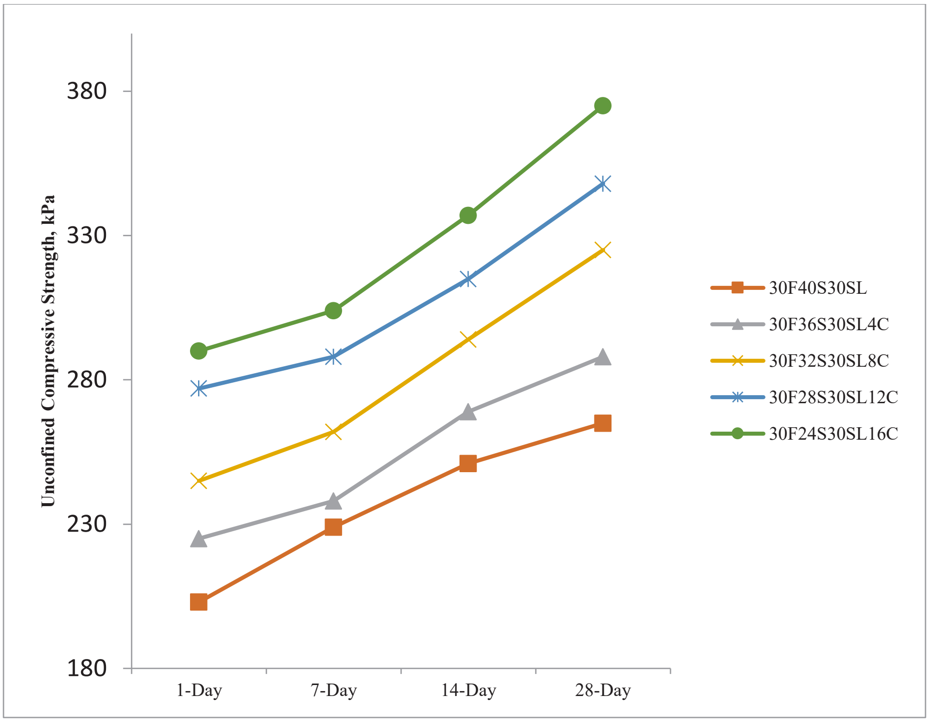

Fig. 5: Unconfined compressive strength of soil, sludge mixtures with 30% fly ash, and varying percentage of cement.

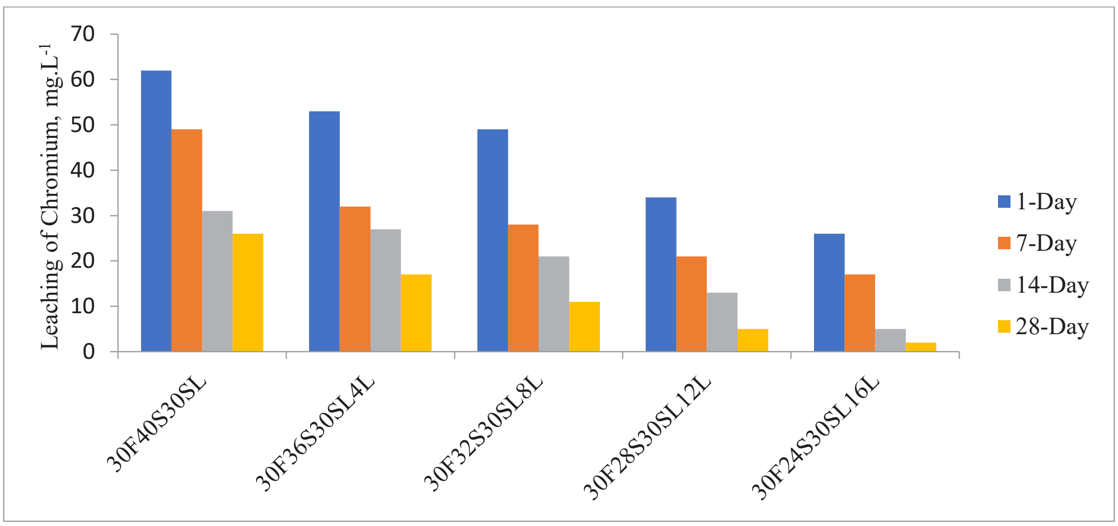

Fig. 6: Leaching of chromium in soil, sludge mixtures with 30% fly ash, and varying percentage of lime.

decreases. This retention behavior may be due to the formation of chromium hydroxide precipitate caused due to the increased lime content in the mixture and adsorption onto fly ash and BCS. The surface complexation and precipitation reactions are the reason behind the leaching behavior. It can also be seen that specimen 30F28S30SL12L gained the minimum leaching requirement of 5 $mg.L^{-1}$ at the curing of 28 days and the same is achieved by 30F24S30SL16L at 14 days of curing itself.

Leaching of Chromium in Soil, Sludge Mixtures with 30% Fly Ash, and Varying Percentages of Cement

The soil, sludge mixtures with 30% fly ash and varying percentage of cement starts with a control specimen of mixtures of 40% of BCS, 30% of Fly ash, and 30% of industrial sludge. And other four specimens represent the percentage of cement increased from 4 to 16% as given

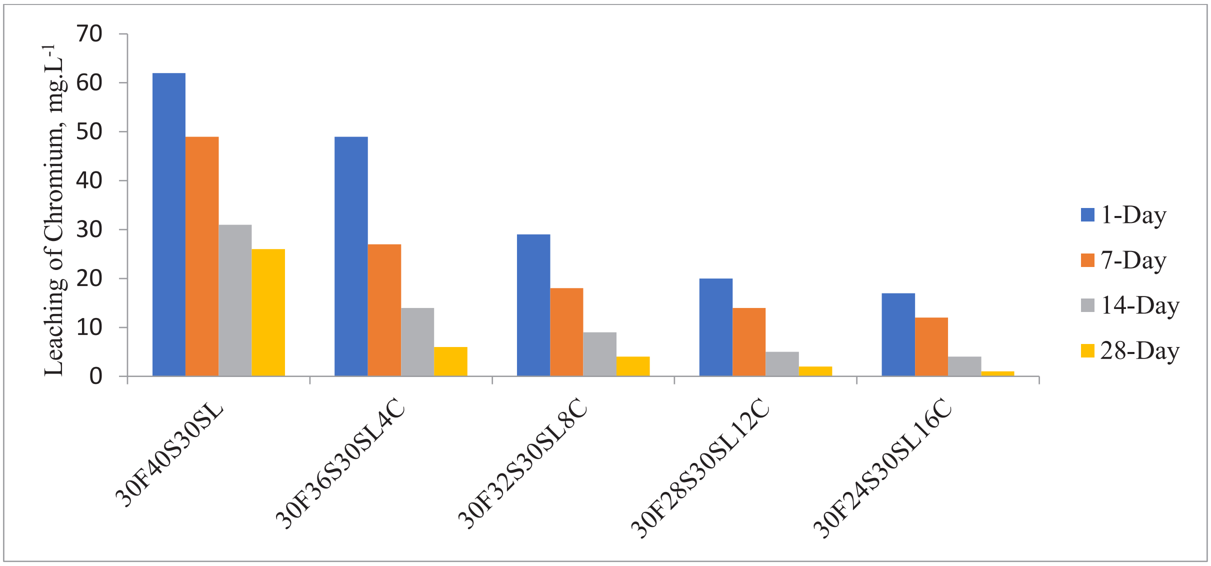

Fig. 7: Leaching of chromium in soil, sludge mixtures with 30% fly ash, and varying percentage of cement.

in Table 4. The leaching behavior of chromium against different curing periods has been presented in Fig. 7. It can be seen in the graph that as the percentage of cement and curing period increases the leaching of chromium decreases. This retention behavior may be due to the formation of hydroxide precipitate caused due to increase in cement content in the mixture and adsorption onto fly ash and BCS. The surface complexation and precipitation reactions are the reason behind the leaching behavior. It can also be seen that specimen 30F28S30SL12C established the minimum leaching requirement of 5 $mg.L^{-1}$ at the curing of 28 days and the same is achieved by 30F24S30SL16C at 14 days of curing itself.

This reduction in the leachability of chromium with an increase in the percentage of cement is observed because of hydrolysis and a series of chemical complexes developed over the time of curing.

Leaching of Copper in Soil, Sludge Mixtures with 30% Fly Ash, and Varying Percentages of Lime

The soil and sludge mixtures with 30% fly ash and varying percentages of lime start with a control specimen of mixtures of 40% of BCS, 30% of fly ash, and 30% of industrial sludge. And other four specimens represent an increase in lime percentage from 4 to 16%. It can be noticed that

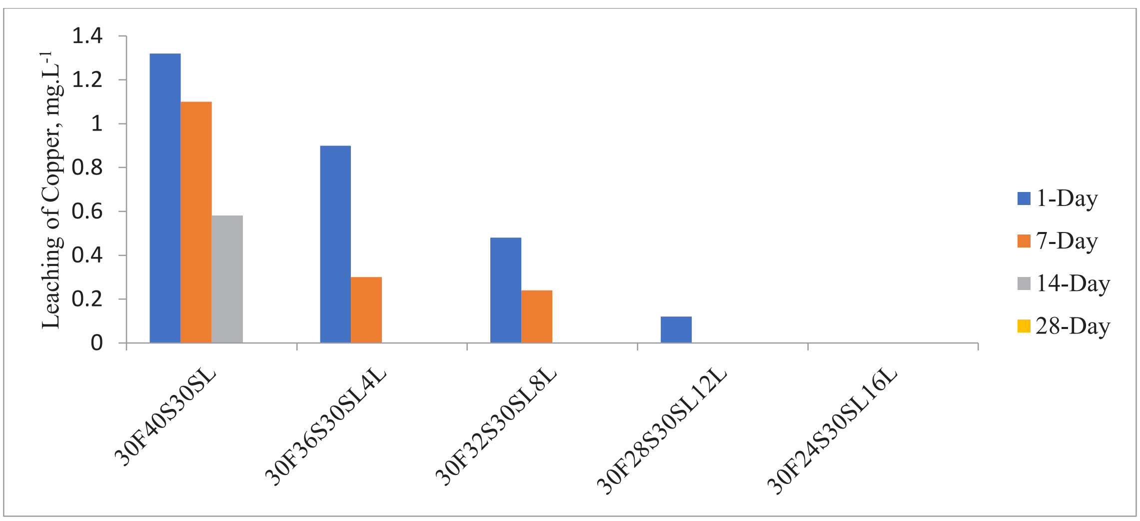

Fig. 8: Leaching of copper in soil, sludge mixtures with 30% fly ash, and varying percentage of lime.

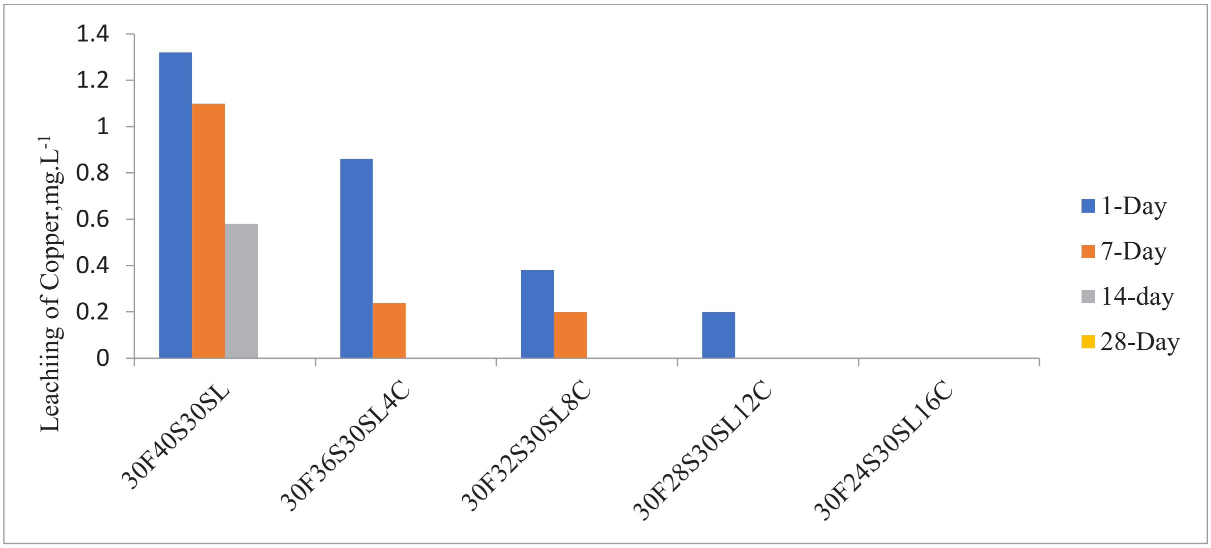

Fig. 9: Leaching of copper in soil, sludge mixtures with 30% fly ash and varying percentage of cement.

as the percentage of lime increases, a slight decrease in the leachability of copper is observed. The leaching behavior of chromium against the different curing periods has been presented in Fig. 8. It can be seen from the graph that as the percentage of lime and the curing period increases, the leaching of copper decreases. This retention behavior may be due to the precipitation of metal ions at higher p^H that prevails due to the increased lime content in the mixture and adsorption onto fly ash and BCS. The surface complexation and precipitation reactions are the reason behind the leaching behavior.

Leaching of Copper in soil, Sludge Mixtures with 30% Fly Ash, and Varying Percentage of Cement

The soil, sludge mixtures with 30% fly ash and varying percentage of cement starts with a control specimen of mixtures of 40% of BCS, 30% of fly ash, and 30% of industrial sludge. And other four specimens represent the percentage of cement increased from 4 to 16%. The combination of mixtures with an increase in cement from 4 to 16% has shown that the leaching of copper reduced drastically with an increased curing period. This may be because of the precipitation and a series of chemical complexes formed throughout curing time as depicted in Fig. 9. The stabilization process by the addition of cement is very effective at fixing copper ions and other ions. The 16% cement addition caused 100% efficiency to stop the leaching of copper as seen in the graph. It can also be noted from the graph that about 12% cement content in the matrix is enough to bring down the leaching of copper below 1 $mg.L^{-1}$.

Comparison Between Leaching of Chromium and Copper at 28 Days Curing Period

From a comparative study on the leaching of chromium and copper in the soil, sludge and fly ash mixtures in addition to lime or cement at the curing of 28 days, it is noticed that the leaching decreases with an increase in lime or cement percentage (Table 9 and 10). The retention capacity is more in cement-stabilized mixtures when compared to lime-stabilized mixtures. Also, it is noticed that in both lime and cement stabilized mixtures 100% retention of copper is noticed at curing of 28 days.

Table 9: Leaching of chromium and copper in the soil, sludge, and fly ash mixtures with lime.

Soil, sludge, and fly ash mixtures with lime	Leaching of chromium [$mg.L^{-1}$]	Leaching of copper [$mg.L^{-1}$]
30F40S30SL	26	0
30F36S30SL4L	17	0
30F32S30SL8L	11	0
30F28S30SL12L	5	0
30F24S30SL16L	2	0

Table 10: Leaching of chromium and copper in the soil, sludge and fly ash mixtures with cement.

Soil, sludge, and fly ash mixtures with cement	Leaching of chromium [$mg.L^{-1}$]	Leaching of copper [$mg.L^{-1}$]
30F40S30SL	26	0
30F36S30SL4C	6	0
30F32S30SL8C	4	0
30F28S30SL12C	2	0
30F24S30SL16C	1	0

CONCLUSIONS

Based on the results attained the UCC strength of the soil and sludge mixtures with 10% and 20% fly ash decreases with an increase in sludge content and the strength increases with an increase in fly ash percentage. The compounds formed due to pozzolanic reaction with the addition of fly ash, lime, and cement bind the soil particles increasing the strength of the soil. Also, an increase in p^H increases the availability of reactive silica in soil which will react with the lime present and enhances the formation of pozzolanic compounds which increases the strength. Finally, it is noticed that the compressive strength of cement-stabilized mixtures was higher when compared to lime-stabilized mixtures and also attained a minimum compressive strength required for safe disposal at a landfill site. The leaching of chromium and copper ions from the leachate was observed from soil and sludge mixtures containing 30% fly ash with lime and cement as additives. In this work, the practicability and efficacy of the fly ash (FA), cement, lime, and soil in the treatment process of industrial sludge which is rich in heavy metal contamination for different proportions have been evaluated. The binders are also proved to be most adaptable to arrest the heavy metal ions and other pollutants present in the contaminated sludge. To check the leaching efficiency, the control mixture was maintained for both lime and cement combinations separately. According to leaching test results of mixtures with soil, sludge, and 30% fly ash in addition to lime and cement, the leaching of the heavy metals from different proportions of soil matrix revealed that with the increase in the lime or cement percentage, the leachability of the heavy metal ions decreases. With the addition of 4% lime at 28 days of curing, the leaching of chromium is 17 $mg.L^{-1}$ whereas the leaching of copper is 0 $mg.L^{-1}$ with the same mixture. With a 4% addition of cement at 28 days of curing, leaching of chromium was $mg.L^{-1,}$ and 0 $mg.L^{-1}$ of copper is observed for the same. The highest chromium retention is noticed in a series of 16% lime or 16% cement whereas the leaching of Copper is below 1 $mg.L^{-1}$ with an addition of 4% cement at 1 day. The relative precipitation of metal ions depends on the solubility product of metal hydroxide. The lower the solubility of the product, the higher the precipitation. It is also found that the leaching of heavy metals from cement-stabilized mixtures was much lower than in lime mixture combinations.

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