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Effect of inorganic materials on the solidification of heavy metal sludge

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Abstract

Portland cement, cement–fly ash and lime–fly ash binders were used to solidify a synthetic heavy metal sludge containing nitrates of Cr, Ni, Cd and Hg. The sludge to binder (cement, cement–fly ash and lime–fly ash) ratio was kept at 3.33, 1.43 and 1.25, respectively. In addition inorganic substances like Cu, Zn, Pb, Sodium hydroxide and sodium sulfate were added. The molded samples were cured at room temperature for 28 days. The solidified samples with and without interference were examined for the change in their bulk density and compressive strength at definite time intervals during curing. All the metals and sodium salts added increased the average bulk density of the final product with increase in concentration (2% to 8%) with all the binder systems. The samples containing copper and lead decreased the compressive strength at all the concentrations added with CFA and LFA binders. Zn had the largest effect on all the three binder systems, lowering the strength of all samples at all the days and concentrations except the 2% Zn with CEM binder. However, Pb had only minor effect on the compressive strength with CEM binder and values remained almost constant at all the times and concentrations studied. In contrast, the effect of sodium sulfate was less marked while sodium hydroxide increased the rate of set and 28-day compressive strength of samples containing cement as binder. These observations confirm the need for specific studies of the waste and binder prior to the selection of a solidification process for the treatment of hazardous wastes. The results provide a better understanding of materials that may interfere with the immobilization of waste constituents and provide information on the possible mechanism of the interfering effects.

Keywords: Heavy metals; Lime; Fly ash; Cement; Compressive strength

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1. Introduction

As the quantity of hazardous industrial wastes increases significantly owing to rapid industrialization, its appropriate management is required to reduce adverse impacts on humans and ecosystems. The disposal options for industrial wastes are limited by the type of waste, cost, legislation and technology. Organic wastes with high calorific values are generally treated by oxidation or incineration, whereas inorganic wastes, commonly found in aqueous solution or suspension, often require pretreatment before landfill. One inorganic waste stream which requires treatment before disposal is metal plating waste, which is often found as sludge or filter cake composed of metal hydroxides with low organic contents [1]. Studies have shown that solidification/stabilization (S/S) processes are viable for most metallic waste streams [1]. S/S of hazardous waste involves mixing the waste with a binder material to enhance the physical properties of the waste and to immobilize contaminants that may be detrimental to the environment. Several binder systems are currently available and widely used for S/S [2–4]. The two main constituents of most commercial fixing agents are cementitious materials (Portland cements, fly ash, lime) and sodium silicate, but increasing research efforts are being directed towards using cement-based binders [1].

The leaching characteristics of solidified materials containing metal nitrates (Cd, Cr and Pb) were investigated under seawater conditions [5]. The results showed that final pH and metal concentration of leachate after 24 h extraction were strongly affected by the nature of leachant. The highest leachability was with Cd, and those of Cr and Pb were negligible in seawater. Cumulative Cadmium released from crushed sample under sea water increased linearly with time, but release rate of the other metal was decreased as leachant renewed. Dissolved heavy metals were considerably reprecipitated under alkaline seawater conditions.

Many studies show that alkalies accelerate early hydration, resulting in increasing the initial strength of

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Portland cement, but decreasing compressive strength later [6–8]. Singh et al. [9] also reported that the addition of alkali bypass kiln dust accelerated the hydration, causing decreased porosity in the hardened structure and increased compressive strength for Portland cement and blast furnace slag-blended cement. Many researchers have studied the effect of heavy metals on cement hydration [10–12]. Tashiro et al. [11] reported that the hydration of cement was retarded in presence of Zn, Pb and Cu.

Three cementitious materials were used to investigate solidifying and stabilizing hazardous wastes containing Cr, Cd and Pb [13]. High alkalies in clinker kiln dust (CKD) accelerated the setting and hydration of cement and quick setting agent (QSA) influenced quick setting and increased compressive strength of cement. The CKD and QSA modified cement presented the least amount of heavy metals leached and the highest compressive strength due to a large number of formation of hydrates and most effective stabilization of hazardous wastes containing multi-heavy metals.

The leachability characteristics of Cr(VI) from solidified Cr(VI)–Portland cement mixtures cured for 28 days were investigated [14]. Cr(VI) was solidified with Type I Portland cement at concentrations of 0.5%, 2% and 5% (based on K_2CrO_4) by weight of the cement. The addition of Cr(VI) increased the initial and final setting times of cement. X-ray diffraction (XRD) study indicates that Cr(VI) inhibits cement hydration process by reacting with calcium ions during the hydration of cement, which was also supported by increased setting times. Increasing the Cr(VI) content in cement reduced the compressive strength of the solidified cement.

Microchemical mechanism of S/S of metal nitrates in Portland cement/fly ash has been well understood using techniques like optical microscopy and X-ray diffractometer (XRD) [5,14], thermogravimetric (TGA), and Fourier-transform infrared (FTIR) analyses [15], atomic adsorption spectroscopy, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) [16].

A lot of work has been reported concerning the binding mechanism and leaching of metal ions during S/S, but very few quantitative data are available concerning the effects of potential compounds upon particular S/S processes. This study was undertaken to help fill the void in these data. The main objectives of the study were to investigate the effects of interfering chemicals (Cu, Pb, Zn, NaOH and sulfate) on solidified heavy metal sludge and physical and containment properties of different binder systems (cement, lime–fly ash and cement–fly ash). The reason for selecting these binders mixture are twofold: (1) it is a mixture commonly used in practice, (2) there are differences in the hydration reactions and physical properties of Portland cement, fly ash and lime which may affect the solidification process.

2. Materials and methods

2.1. Synthetic toxic sludge preparation

A synthetic, heavy metal sludge was prepared by adding soluble nitrates of chromium, nickel, cadmium and mercury to water. Calcium hydroxide was added to raise the pH to precipitate the metals in the form of insoluble hydroxides. The synthetic metal solution was prepared by dissolving 23.1 g/l of $Cr(NO_3)\cdot 9\ H_2O$, 14.9 g/l of $Ni(NO_3)\cdot 6\ H_2O$, 1.6 g/l of $Cd(NO_3)\cdot 4\ H_2O$ and 0.02 g/l $Hg(NO_3)\cdot H_2O$ in water. Calculations revealed that a calcium hydroxide dosage of 10.6 g/l provided the stochiometric amount of hydroxide required to precipitate the metals in the synthetic metal solution. Lime dosages of 10, 12, 14, 16, 18 and 20 g/l were evaluated during the precipitation of metals. A lime dosage of 20 g/l produced sludge with optimal settling characteristics and a supernatant with the lowest turbidity. The additional lime required could be attributed to water hardness, the acidity of the metal solution and the impurities in the lime. A lime dosage of 20 g/l was used for sludge preparation throughout this study. The metal sludge was dewatered to approximately 9% solids and had a density of 1.08 g/ml. The final pH of the supernatant was 11.5.

2.2. Binders

Three different kinds of binder materials—30% Portland cement (CEM) only, 20% cement and 50% fly ash (CFA), and 30% lime and 50% fly ash (LFA)—were used to solidify a synthetic metal plating waste sludge. The concentration (ratio) of the binders was taken on the basis to achieve the required compressive strength of the molded samples. The fly ash was obtained from a coal power plant and its major composition is presented in Table 1. Heavy metal concentrations from the binders were found to be negligible.

2.3. Interfering substances

The interfering substances used to evaluate their effect on the solidified synthetic metal plating sludge included three metals—copper, lead and zinc—and two sodium salts—sodium hydroxide and sodium sulfate. Each of these substances was mixed in metal sludge in three different concentrations (2%, 5% and 8%) individually.

2.4. Solidification

After the metal sludge was produced, binding materials were mixed individually with synthetic metal sludge to

Table 1
Fly ash composition (% w/w)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Ig.loss
59.1	16.35	6.25	2.25	1.08	8.1

solidify the wastes and immobilize the contaminants. Prior to set, this sludge/binder mixer was divided into four portions. Select interfering chemicals were added with three of the sludge/binder portions at three different concentrations (2%, 5% and 8%). The sludge to binder (CEM, CFA and LFA) ratio was kept 1:0.3 (30% cement only), 1:0.8 (30% lime and 50% fly ash) and 1:0.7 (20% cement and 50% fly ash), respectively, and water to binder ratio was kept 1:2.5. The mixtures of three-interference/sludge/binder and control without interference (sludge and binders only) were poured into 5-cm^3 molds for 24-h hardening and 28-day curing in a damp chamber at room temperature. These molded specimens with and without interference were examined for visual changes, changes in bulk density and changes in compressive strength. Each experiment is performed in triplicate and the average values of bulk density and compressive strength are reported.

3. Results and discussion

The results of the effect of interfering substances on the solidification of heavy metal sludge have been shown in Figs. 1–6 and are discussed in the following sections.

3.1. Visual observations

During the process of curing, molded specimens were observed visually for any change prior to testing. Very little changes were observed between the controls and the samples containing lead. In contrast, significant changes were observed for NaOH, Zn, Cu and sulphate interference at all the concentrations studied. For all the three binder systems, the samples with added NaOH appeared to absorb more water than the other samples. The CEM and CFA binders with as little as 5% Zn produced samples that were very soft and difficult to remove from the molds, even after 11 days of curing, while the same condition was not observed in case of LFA binder added with zinc. The CEM and CFA samples with 5% and 8% Cu additions were noted to have developed about a 0.25-in. crust layer on the surface of the samples. This layer was very hard, but beneath the crust the sample appeared to be soft. No observations of the crust were recorded for the LFA-Cu samples and were elastic even after the 28 days of curing. Surface cracks were observed in the CFA samples with 2% to 5% sulfate additions as early as 11 days of curing. No cracks were observed in 8% sulphate samples for the LFA binder. These samples appeared to be elastic in the early curing process. Only slight surface scaling was observed for the CEM-sulfate samples. Changes in colour and shape were almost negligible in all the samples containing metals and sodium salts as interfering materials for all the three binder systems at 1 and 28 days of curing.

3.2. Effect on bulk density

Bulk densities of all the controls and samples added with interference were determined at 1 and 28 days of curing. The average bulk density of the control samples (15 specimens for each binder system) for the different binders CEM, CFA and LFA were 1.293, 1.399 and 1.538 g/cm^3, respectively. The coefficient of variation (CoV) observed in bulk density was consistently between 6% and 8% for all three binder systems. The waste products solidified with the CEM had bulk densities 7% to 16% lower than those solidified with CFA or LFA, while the highest bulk density was with LFA binder.

The average bulk densities (mean of three specimens of each interferent concentration and binder system) of the solidified products with metals (Pb, Zn and Cu) with three binders studied are plotted in Fig. 1 at 1-day curing. A perusal of Fig. 1 shows that the bulk density of the solidified product varies between binders and with the addition of different interference. The bulk densities of the samples containing metal interference increased proportionally with the amount of metal added. It is observed that the waste stabilized with CEM and CFA shows increase in bulk density to a greater extent due to addition of metals. The metals had a smaller effect on the bulk density of the more dense LFA solidified samples, increasing them only 6% to 8%. Fig. 1 also showed that the average bulk density of solidified samples with CEM binder varied from 1.421 to 1.529 g/cm^3 with increase in metal concentration from 2% to 8% and the same for CFA and LFA binders varies from 1.467 to 1.574 g/cm^3 and 1.584 to 1.606 g/cm^3, respectively at 1-day of curing.

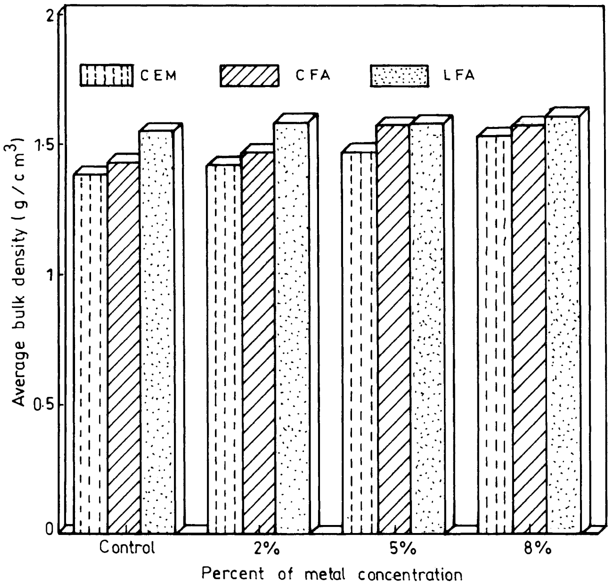

Fig. 1. Average bulk density of solidified product with metals at 1-day curing.

Addition of sodium salts (sodium hydroxide and sodium sulfate) increased the bulk density of the final product by 10% at highest added concentrations (8%). The average bulk density of solidified samples added with sodium salts with the CEM binder varies from 1.402 to 1.502 g/cm^3 with increase in interference concentration from 2% to 8% and the same for CFA and LFA binders varies from 1.540 to 1.640 g/cm^3 and 1.577 to 1.633 g/cm^3, respectively at 1-day of curing.

The bulk densities of metals and sodium salts containing samples at 28-day curing are almost close to the values obtained at 1-day curing and most of the samples retaining between 90% and 100% of their originals bulk densities. Differences observed in bulk densities would be due to loss of water through evaporation or by change in the volume of the final product during curing.

The overall average bulk density of the solidified products with all added metals and sodium salts along with all the three binders is shown in Table 2 with the average bulk density of control at 1 and 28 days of curing. Table 2 shows that the average bulk densities increase with increase in concentration of added metals and sodium salts from 2% to 8%.

3.3. Effect on compressive strength

The compressive strength of all the controls and samples added with metals and sodium salts was analysed on 7–11 and 28 days of curing. Compressive strength values for controls showed that the CEM mixes were designed to give at least 10 MPa for 5 cm^3 after 28 days. The average compressive strength of control samples (15 specimens of each binder system) with CEM, CFA and LFA binders was 8.4, 19.4 and 24.9 MPa at 7–11 days of curing and the same was 11.2, 62.4 and 59.1 MPa at 28-day curing. These results showed that the compressive strength of cement mixes was about 75% of their 28-day compressive strength after 7–11 days of curing. To produce samples with compressive strengths in this low range, the proportions of the reagents and mixing parameters of the batch are quite critical. Although great care was taken to reduce variability associated with the batch preparation steps and reagent additions were carefully controlled, substantial variability was observed in the control samples. The variability within a specific batch or mix was low. However, variability between mixes was appreciable. The effect of all the interfering substances on the cured samples was compared only with their corresponding control samples.

Table 2
Average bulk density of solidified products with metals and sodium salts

Materials	Days	Average bulk density (g/cm^3)			
		Control	2%	5%	8%
Metals	1	1.443	1.483	1.522	1.562
	28	1.314	1.398	1.526	1.562
Sodium salts	1	1.495	1.507	1.546	1.592
	28	1.423	1.478	1.511	1.545

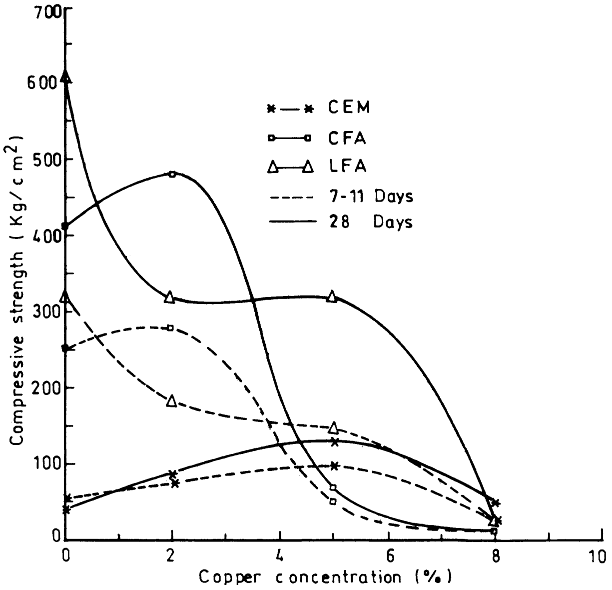

Fig. 2. Compressive strength as a function of added copper concentrations.

Figs. 2–6 show the variation of compressive strength (mean of three specimen of each interferent concentration and binder system) with the addition of interference at 7–11 and 28 days of curing using three binders for solidification. Figs. 2–4 show that all the three metals have pronounced effects on the rate of attainment of strength and the final 28-day compressive strength. Fig. 2 indicates that copper produced different effects on all the three binder systems depending upon its concentration. Addition of 2% to 5% Cu increased the compressive strength throughout the curing period for the CEM binder. The Compressive 28-day strength for 2% Cu in CEM samples was twice that of control and for 5%, three times of the control. However, addition of 8% Cu delayed the onset of the setting reactions. At 28 days of curing, the compressive strength of the 8% Cu addition samples has surpassed that of the control. The samples with CFA binders lessen the increase in compressive strength for the 2% copper additions and caused a consistent loss of compressive strength for the 5% and 8% additions. An addition of 8% Cu practically eliminates the development of compressive strength in these samples. The LFA samples are consistently weakened by the addition of any of the Cu concentrations. Copper interferes with the setting of all the mixes containing fly ash, as evidenced by the reduction of compressive strength.

Fig. 3 shows that Zn has the largest effect on all the three binder systems, lowering the strength of all samples at all the days and concentrations except the 2% Zn in cement. In CEM samples at 2% Zn, a consistent strength increase in compressive strength is observed and a 28-day compressive strengths of four times the compressive strength of the control is observed. At 5%, Zn limited the 28-day compress-

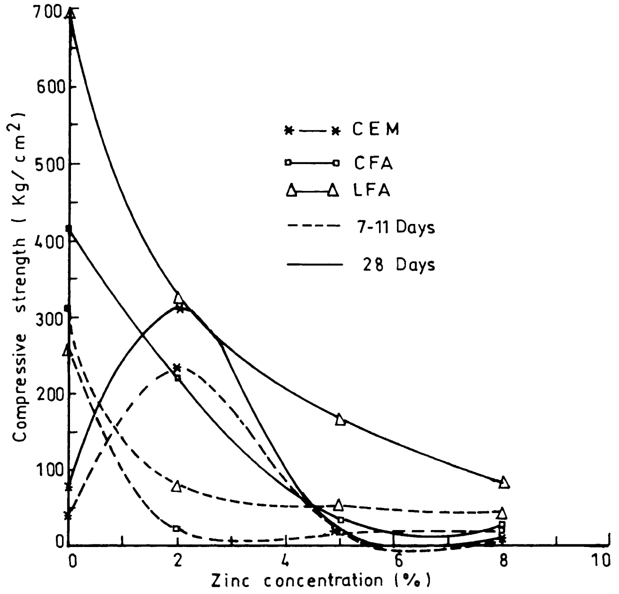

Fig. 3. Compressive strength as a function of added zinc concentrations.

ive strength to less than 30% of that of the control, and at 8% Zn, to less than 15% of the controls. Even at 2%, Zn lowers the 28-day compressive strength to about 50% of the control for both CFA and LFA binders.

Fig. 4 indicates that addition of lead to both LFA and CFA samples lowers the compressive strength to levels comparable to those of zinc. The compressive strength of these samples was inversely proportional to the added percentage of lead. However, at all the concentrations added, Pb had only minor effect on the compressive strength on the cement sample and values remained almost constant at all the times and concentrations studied.

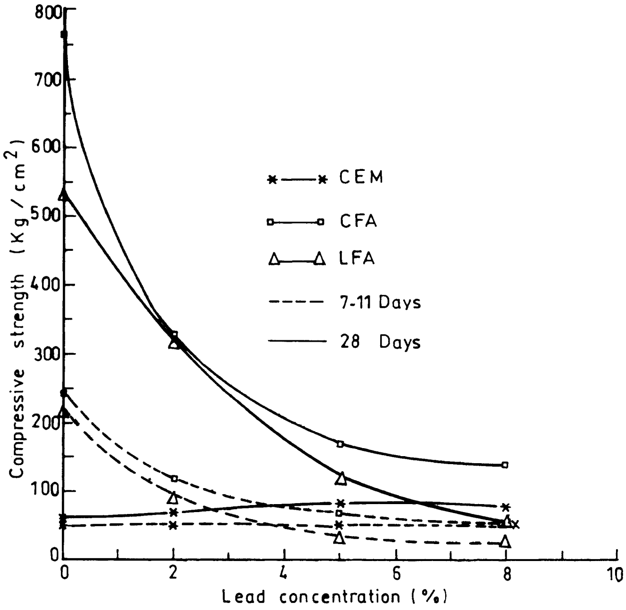

Fig. 4. Compressive strength as a function of added lead concentrations.

The effect of sodium hydroxide addition to solidified samples has been shown in Fig. 5. A perusal of Fig. 5 shows that addition of NaOH generally promotes rapid early strength development, especially at lower concentrations. However, results vary depending upon its concentration and the binder. Addition of 2% NaOH is responsible for a higher compressive strength in CFA and LFA binders at all the days. Only CEM samples with 5% and 8% NaOH addition have consistently lower compressive strength throughout the 28 days of curing. The LFA and CFA samples are affected to a greater extent with all concentrations of NaOH, showing a rapid set and very high early compressive strength values. LFA is especially affected. However, after 28 days of curing, the samples with 5% and 8% additions have about the same or lower compressive strength values than the controls. If these trends continue, the controls may be expected to continue to have higher compressive strength after periods longer than 28 days.

The effect of sodium sulfate addition on solidified samples is shown in Fig. 6, which showed that the strength development varied greatly for the three different binder systems. For CEM samples, the compressive strength decreases slightly or remains almost constant all the time with the increase in sulfate concentration from 2% to 8% throughout the 28-day curing period. LFA samples show rapid set and high early strength development with both 2% and 5% sulfate additions. However, after 28 days, 8% sulfate reduces the compressive strength of the LFA samples to about 20% of the controls. Effects on the CFA samples were intermediate between those on the other two binders. Fig. 6 also shows that LFA samples has a more rapid rate of cure at low sulfate concentration and an inhibition of strength formation at the highest sulfate level.

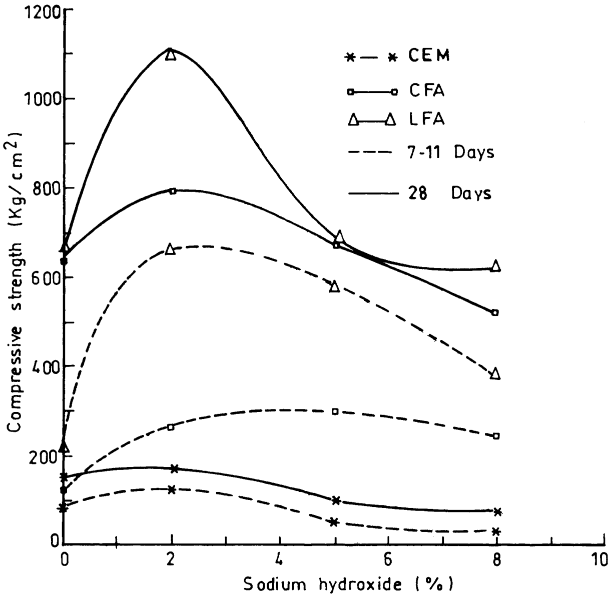

Fig. 5. Compressive strength as a function of added sodium hydroxide concentrations.

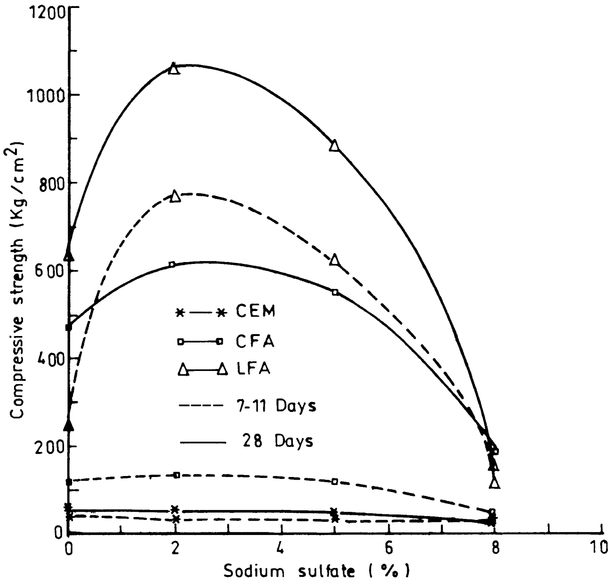

Fig. 6. Compressive strength as a function of added sodium sulfate concentrations.

In the present study, the results show that the metal ions with CFA and LFA binders had detrimental effect on cementing reactions, and inhibited the rate and extent of hydration. The possible reason for it may be that the degree of hydration in the sample containing metal ions with CFA or LFA is low as compared to the control. This results in decrease of compressive strength due to nonhydration of the calcium silicate and absence of calcium hydroxide. The absence of calcium hydroxide could be due to one of the following [15]:

(i) a lower degree of hydration of calcium silicates could be producing a smaller amount of calcium hydroxide (which is entirely consumed by the fly ash) than in CFA/LFA binder; or
(ii) hydroxyl ions from calcium hydroxide combine with heavy metals to form complexes.

The behavior of binders (fly ash/lime) in solidification/stabilization processes should be better understood since these are widely used in commercial processes [15,17]. It can chemically react with the waste and binder. Fly ash reacted with Portland cement component to form a variety of reaction products including ettringite and straetlingite which reduced the chances of alkali–aggregate attack by entrapping K and Na. Tashiro et al. [11] also reported that hydration of cement materials was retarded in the presence of Zn, Cu and Pb. The inhibitory effects of these metal ions to hydration were related to the formation of double salts, which were formed between these heavy metals and calcium ions dissolved from cement.

The results of alkali addition (sodium hydroxide and sodium sulfate) as interferents may be explained on the basis of earlier studies [6–8], where alkalies accelerate early hydration, resulting in increasing the initial strength of the samples, but decreasing compressive strength later. The results of present study are in good agreement with the earlier studies. Singh et al. [9] also reported the similar results where alkali accelerated the hydration, causing decreased porosity in the hardened structure, and increased compressive strength.

4. Conclusions

The observations of the present study revealed the effect of added interferents (heavy metal ions and alkalies) on the bulk density and the compressive strength of the solidified products. Each interference produced a strong, moderate or slight positive or negative effect on each of the measured parameters for each binder system. It can be concluded that the metals are generally deleterious to the strength and durability of all binder system. The effect of the interference generally increased with the increase in its concentrations. All the metals and sodium salts added increased the average bulk density of the final product with increase in concentration (2% to 8%) for all the binder systems. The samples containing copper decreased the compressive strength at all the concentrations added with CFA and LFA binders, while increase in the early strength was observed with CEM binder. Zn had the largest effect on all the three binder systems, lowering the strength of the samples at all the days and concentrations except the 2% Zn with CEM binder. The compressive strengths of lead containing samples were inversely proportional to the added concentration of lead with CFA and LFA binders. However, at all the concentrations added, Pb had only minor effect on the compressive strength with CEM binder and values remained almost constant at all times and concentrations studied. Sodium hydroxide and sodium sulfate had mixed effects, increasing early strength at lower concentration (2%), but decreasing early strength at higher concentration (8%). Sodium salts addition caused a very slight decrease in compressive strength with increase in concentration in CEM samples. The results of the present study confirmed the need for specific studies of waste and binder prior to the selection of a chemical solidification/stabilization process for the treatment of hazardous wastes. Further studies are also needed to determine the effect of sludge variability on macroscopic leaching or mechanical behavior during solidification processes.

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