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Solidification/stabilization of hazardous waste sludge obtained from a chemical industry

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Abstract In this study, treatment sludge obtained from a chemical-metal finishing industry, which contained potentially toxic heavy metals and organics, was characterized, and the performance of the solidification/stabilization (S/S) of the sludge was discussed. The hazard characteristics of the waste were determined by means of extraction procedure toxicity test and DIN 38414-S4 Test, as defined in both Turkish and USEPA regulations. S/S studies were conducted using Portland cement to solidify the sludge containing high concentrations of total organic carbon, Cr, Cu, Hg, Ni, Pb, and Zn. The waste/binder ratios of 36 sludge specimens were kept between 0/100 and 40/100. The specimens were cured at room temperature for 7, 28, and 90 days. The compressive strengths of the specimens were measured to determine the feasibility of using solidified waste sludge as construction materials. The compressive strength values indicated that specimens could be potentially used as construction materials. The heavy metal and organic contents of the extracts of each specimen were detected in concentrations which were lower than the standard concentrations in EPTox and DIN 38414-S4 leaching procedures for the most part.

Keywords Solidification/stabilization · Hazardous waste treatment · Compressive strength · Portland cement · Leaching

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Introduction

Solidification/stabilization (S/S) processes have become widely used technologies as the final treatment step for the treatment of industrial hazardous waste before disposal (Cioffi et al. 2002; Poon and Lio 1997). S/S technologies are potentially useful for improving chemical and physical properties of hazardous wastes to an extent that they are suitable for less expensive disposal or utilization with safety environmental impact (Stegemann and Cote 1996).

Stabilization refers to reducing the hazardous behavior of a waste by means of chemical reactions, and solidification refers to generating a monolithic solid of high structural integrity (Coz et al. 2004). When a specific waste is stabilized by means of a specific binding matrix, a study is necessary to assess its potential to be used as building material and therefore, assess the reuse of the waste instead of safe disposal (Cioffi et al. 2002). Different processes exhibit different setting and curing reactions. However, most of the commercial inorganic S/S systems were thoroughly in connection with the Portland cement technology used in concrete making. S/S processes develop a very wide variety of strength and durability values depending on many factors: waste type, water content, reagent type, reagent addition ratio (mix ratio), curing time, and temperature. Many processes can adjust the final strength and durability values by changing reagent mix ratios (Conner and Hoeffner 1998).

Although there is no ideal laboratory test to evaluate the stabilization efficiency for every waste, it is possible to select proper tests based on the aim of the stabilization program and chemical composition of the waste (La Grega et al. 1994). Performance of S/S systems is assessed traditionally using three parameters: leaching behavior, permeability, and structural integrity (Cohen et al. 1997).

In the literature, several physical and chemical testing procedures have been identified. The most common physical tests are compressive strength, permeability, bulk density, and index property tests. Leaching tests are used to study the leaching behavior of solidified wastes. The leaching test is used by regulatory agencies for classifying hazardous waste as well as by waste disposal contractors to compare the leaching performance of different waste forms (Poon and Lio 1997). The commonly used leaching tests are categorized as: (a) extraction tests, (b) leach tests, and (c) column leach tests. Two established extraction tests are toxicity characteristics leaching procedure and extraction procedure (EP) toxicity test (Chan et al. 2000).

During the last decade, the literature concerning the application of cement-based S/S for industrial sludge has spread into hazardous waste management. Examples can be listed as follows: evaluation of the effectiveness of the stabilization and solidification of heavy metals from an electroplating industry sludge (Silva et al. 2007), affectivity of cement in immobilizing heavy metals in the steel processing plant sludge (Malviya and Chaudhary 2006), the potential for utilization of municipal solid waste incineration fly ash as solidification binder to treat heavy metals-bearing industrial waste sludge (Qian et al. 2006), S/S of an industrial copper sludge by ordinary Portland cement and pulverized fly ash and evaluation of the speciation, binding mechanisms and leaching behaviors of heavy metals (Li et al. 2001), the leaching behavior of arsenic from a solidified/stabilized waste (Singh and Pant 2006), evaluation of leaching behavior of inorganic constituents from stabilized/solidified refinery oily sludge and ash produced from incineration of oily sludge with cement (Karamalidis and Voudrias 2007), and observations on the transient leaching behaviour of chromium, cadmium, and aluminum that were solidified/stabilized by pozzolanic fly ash (Camacho and Munson-McGee 2006).

Many studies have been reported concerning the leaching of metals during S/S. However, most of these studies were conducted by preparing concrete samples in the laboratories. The application of S/S technologies for commercially used high-strength concrete (e.g., C35 class) is yet to be investigated thoroughly. The main objectives of this study were to implement the S/S technology on the treatment sludge of a chemical industry and to investigate the compatibility with disposal and reuse. The present study was undertaken to help solidify and stabilize the treatment sludge in different compositions in a C35 class of concrete which contained commercial additive. The stabilization characteristics of the waste were evaluated from leaching tests (EP Toxicity Test and DIN 38414-S4 Test) after each S/S process. The waste/binder (W/B) ratios on the performance of the S/S were also determined according to compressive strength of the solidified materials. Satisfactory results were obtained in the study.

Materials and methods

During experimental studies, the S/S technology was applied to an industrial treatment sludge which originated from a chemical industry located in Gebze (in the northwest of Turkey). This industrial facility produces chemical materials to be used for metal finishing and surface treatment processes, as well as other chemicals for many different purposes such as aerospace technologies and fine chemistry.

The sludge was analyzed by means of eluate and extract procedures as defined, and the hazard characteristics of the waste were determined based on both Turkish and USEPA regulations by means of leaching test methods (DIN 38414-S4 Test and Extraction Procedure Toxicity Test). The sludge was considered a hazardous waste.

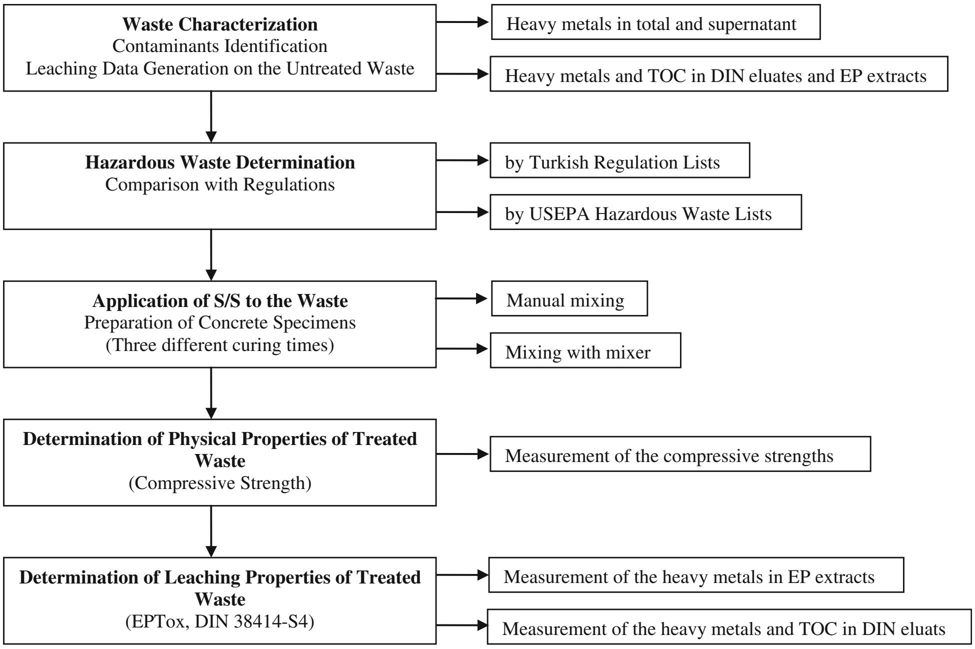
Portland cement PC-42,5 (CEM I 42,5 R) was used as a binder. It was mixed with the sludge to solidify the waste. Fine aggregate, course aggregate, and water were also used for concrete specimen preparation. For cement-based S/S studies, cement was used with Rheobuild® 1000 as concrete additive. Rheobuild® 1000 is a strength-enhancing, super-plasticizing, high-range, water-reducing admixture formulated to produce rheoplastic concrete. The addition of Rheobuild® 1000 allows mixing water to be reduced considerably and concrete strength to be accelerated significantly, particularly at the early stages.

Compressive strength tests and leaching tests were conducted to evaluate the S/S efficiency for solidified materials, as shown in Fig. 1. Figure 1 shows a work flow for experimental studies. Compressive strength tests were used for determination of the physical properties of treated waste; EP Toxicity Test and DIN 38414-S4 Test were used for determination of the leaching properties of treated waste as it was also shown in the experimental work flow. Total organic carbon (TOC) and the heavy metal concentrations in EP extracts, eluates, and digested solutions were determined.

Characterization of hazardous waste

A waste sludge sample was collected before the dewatering processes, and Hach Digesdahl Digestion Apparatus was used to determine the total amount of metal in the sample. The samples were digested by heating in the presence of sulfuric acid and hydrogen peroxide. The water was decanted in order to analyze the soluble part of the sludge. Sludge water was digested by heating in the presence of nitric acid (nitric acid digestion) (APHA, AWWA, WPCF,

Fig. 1 Experimental work flow



1998). In order to measure the total amount of metal and the amount of soluble metals, atomic absorption spectrometer (AAS) was used.

Two procedures were used to specify leaching properties of untreated waste. One of them is DIN 38 414 S4 Test which is a German Standard Method (Institut fur Normung 1984) and the other is USEPA EP Toxicity Test Method (USEPA 2002). The DIN 38414-S4 test is generally enforced in the Turkish Regulation on Hazardous Waste Control (Ministry of Environment 1995). DIN eluate and EP extract were prepared according to these two procedures. At this stage of the study, the treatment sludge was dried before use. Eluate and extract were digested according to Standard Methods (APHA, AWWA, WPCF 1998) in order to make metal measurements by AAS. The amount of metals which change into liquid phase (in other words, the amount of soluble metals) could be determined in eluate and extract.

The composition of the raw waste determined by these processes, and the leaching concentrations of untreated waste are given in Table 1. Leaching concentrations were compared to three different regulations to decide whether the samples could be experimentally determined as hazardous waste. Maximum concentrations of metals (Ag, As, Ba, Cd, Cr, Hg, Se, and Pb) for characteristic EP toxicity; in other words, EPTox Regulatory Limits for certain metals are given in Table 1. DIN-leaching levels for metals (As, Cd, Cr, Cu, Hg, Ni, Pb, and Zn) are also given in Table 1. "-" in the table means "there was no limit for those metals" in leaching procedures. Leaching concentrations of metals having specified standard were identified.

As lead, mercury, nickel, zinc, and chromium concentrations of the first leachate were all above these standards, the waste could be determined as hazardous waste. As it can be seen according to extraction procedure toxicity test (USEPA 2002), the untreated waste used in this study is EP toxic. In Table 1, the information on TS EN 12457-4 leaching levels for waste characterization, before landfilling in recent Turkish Hazardous Waste Control Regulations of both 1995 (Ministry of Environment 1995) and 2005 (Ministry of Environment 2005), are presented.

TOC concentration was measured for the characterization of the sludge TOC instrument, according to standard methods (APHA, AWWA, WPCF, 1998). TOC of DIN eluate as organic content of untreated waste is 1,030 mg L^{-1}.

As a result, waste in sludge form from a chemical industry was determined to be hazardous waste according to the hazard criteria: toxicity/ecotoxicity lists of the hazardous waste.

S/S and evaluation tests

Portland Cement as a binder was mixed with the sludge to solidify the waste and immobilize the pollutants. Control and other specimens were prepared with dried sludge, cement, fine aggregate, course aggregate, and water, and Rheobuild® 1000 as C 35 class of concrete according to standard "TS EN 206/1" (Institute of Turkish Standards 2002). In order to prepare C 35 class of concrete, waste was added instead of aggregate, and Portland cement was used as a binder. Five different mixing ratios of waste to binder (W/B) were changed from 1/100 to 40/100. Mixing

Table 1 Heavy metal content of the waste (raw sludge)

Parameters	Total (mg kg^{-1})	Soluble[a] (mg L^{-1})	Leaching[b] (mg L^{-1})				
			EPTox	EPTox regulatory limit	DIN	DIN leaching level	TS EN 12457-4 leaching level[c]
Ag	58	0.1	0.013	5	–	–	–
As	3.7	0.01	0.009	5	0.002	0.2	0.2
Ba	4.0	0.53	0.130	100	–	–	10
Cd	25.1	0.02	0.009	1	0.003	0.1	0.1
Cr	4700	0.13	0.010	5	0.320	0.1	1 (total)
Cu	1640	1.18	–	–	0.490	2	5
Hg	40	2.33	1.200	0.2	0.060	0.02	0.02
Se	20.1	0.03	0.061	1	–	–	0.05
Ni	806	71.9	–	–	4.745	0.4	1
Pb	45	3.49	0.085	5	0.950	0.4	1
Zn	65813	2490	–	–	1246	2	5

[a] Soluble heavy metal values represent the measurements in supernatant of the raw sludge

[b] Leaching levels represent the measurements in extracts obtained from leaching procedure for certain amount of raw sludge. EPTox leaching levels are stipulated by USEPA. DIN 38414-S4 leaching levels are stipulated for waste characterization before landfilling in the Turkish Hazardous Waste Control Regulations of 1995

[c] TS EN 12457-4 leaching levels are stipulated for waste characterization before landfilling in the Turkish Hazardous Waste Control Regulations of 2005

processes continued until concrete had a uniform appearance. Specimens were mixed manually initially and afterward the procedures were repeated by mixers. Water/cement ratio of specimens for each mixture was kept to be 0.49–0.68, and it was adjusted to between 170 and 200 mm for the slump test. Cubic specimens with dimension of 15 × 15 × 15 cm were molded and immersed in a cure bath at 20 ± 2 °C room temperature for three different curing times.

The molded specimens were examined for the change in compressive strength at the end of 7, 28, 90 days curing time by compression test press. Three different curing times were applied to specimens to compare the compressive strength of the different curing time periods.

The compliance test used for heavy metal leaching was the EP Toxicity Test (USEPA 2002) which was performed using an acetic acid solution with a liquid/solid proportion of 16 for 24 h. In this test, concrete specimen was crushed to pass a 9.5 mm sieve. A 0.04 M acetic acid ($pH = 5 \pm 0.2$) leaching solution was used (La Grega et al. 1994; USEPA 2002). The extraction liquid was a mixture of deionized water and acetic acid; so the specified pH was achieved (La Grega et al. 1994). The pH value of the extraction liquid was adjusted to 5.0. The filtered extract was analyzed using AAS with graphite furnace (Perkin Elmer, SIMAA 6000 Model) for metal concentrations. DIN 38414-S4 leaching procedure was also used (Institut fur Normung 1984). This test involved the addition of distilled water to the sample in the ratio of 1 L water to 100 g dry material. After 24 h of shaking, the sample was filtered and analyzed. In the DIN 38414-S4 test leachates, the components were selected as a function of origin and qualitative composition of sludge.

Results and discussion

Mechanical properties

The compressive strength of all specimens in various ratios and control was measured for 7, 28 and 90 days of curing periods. Compressive strength values of the specimens

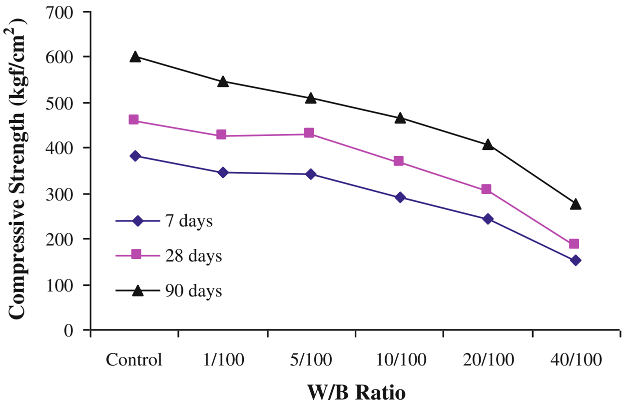


Fig. 2 Compressive strength as a function of W/B for different curing times

Table 2 Leaching concentrations of specimens

Waste/Binder (waste/cement) ratio (W/B)	Metal parameter	Leaching concentrations (mg L^{-1})						
		EP extracts			EPTox regulatory limit	DIN eluates		DIN leaching level
		7 days	28 days	90 days		28 days	90 days	
0 (control specimen)	Chromium	NM[a]	NM[a]	NM[a]	5	0.102	0.137	0.1
	Lead	0.040	0.014	0.007	5	0.102	0.031	0.4
	Mercury	0	0.481	0.001	0.2	0.053	0.006	0.02
	Copper	–	–	–	–	0	0	2
	Nickel	–	–	–	–	0.107	0	0.4
	Zinc	–	–	–	–	1.695	2.112	2
1/100	Chromium	NM[a]	NM[a]	NM[a]	5	0.166	0.190	0.1
	Lead	0.026	0.009	0.006	5	0.011	0.058	0.4
	Mercury	0.001	0.053	0	0.2	0.057	0.0001	0.02
	Copper	–	–	–	–	0	0	2
	Nickel	–	–	–	–	0.066	0.08	0.4
	Zinc	–	–	–	–	1.335	1.02	2
5/100	Chromium	NM[a]	NM[a]	NM[a]	5	0.154	0.095	0.1
	Lead	0.023	0.012	0.016	5	0.024	0.010	0.4
	Mercury	0.041	0.195	0.016	0.2	0.002	0.002	0.02
	Copper	–	–	–	–	0	0	2
	Nickel	–	–	–	–	0.069	0.075	0.4
	Zinc	–	–	–	–	3.139	2.842	2
10/100	Chromium	NM[a]	NM[a]	NM[a]	5	0.195	0.045	0.1
	Lead	0.019	0.016	0.018	5	0.029	0.009	0.4
	Mercury	0.011	0.060	0.008	0.2	0.059	0	0.02
	Copper	–	–	–	–	0	0	2
	Nickel	–	–	–	–	0.057	0.007	0.4
	Zinc	–	–	–	–	7.182	5.966	2
20/100	Chromium	NM[a]	NM[a]	NM[a]	5	0.109	0.14	0.1
	Lead	0.011	0.015	0.015	5	0.013	0.044	0.4
	Mercury	0.005	0.013	0.002	0.2	0.043	0.003	0.02
	Copper	–	–	–	–	0.0001	0	2
	Nickel	–	–	–	–	0.003	0.029	0.4
	Zinc	–	–	–	–	11.331	9.861	2
40/100	Chromium	NM[a]	NM[a]	NM[a]	5	0.099	0.159	0.1
	Lead	0.011	0.032	0.052	5	0.065	0.057	0.4
	Mercury	0.01	0.105	0	0.2	0.059	0.007	0.02
	Copper	–	–	–	–	0	0	2
	Nickel	–	–	–	–	0.217	0.1	0.4
	Zinc	–	–	–	–	20.668	2.615	2

[a] Not Measured since leaching concentration of untreated waste was lower than standard leaching concentration

were compared to an average compressive strength of concrete in control samples and the standard (TS EN 206/1) (Institute of Turkish Standards 2002). It was shown that the higher the W/B ratio, the lower the compressive strength for this solidified sludge. Maximum and minimum compressive strengths (426, 182 kgf cm^{-2}) were obtained at 1/100 and 40/100 ratios of W/B, respectively for 28 days curing period. Figure 2 shows the changes of compressive strength as a function of W/B ratios for different curing times. Compressive strengths of concrete specimens show that the longer the curing time, the higher the compressive strength for this solidified sludge. There were significant differences between compressive strength values for 28 and 90 days cured specimens. Better performance of S/S of treatment sludge for 90 day of curing period was observed. If the compressive strength values were considered solely, then it could be assessed that the solidified sludge by cement, at W/B ratio of <20/100, met the strength criterion for C20–C25 class of concrete (250–300 kgf cm^{-2}) (10/100 W/B ratio could be used for making C30 class of

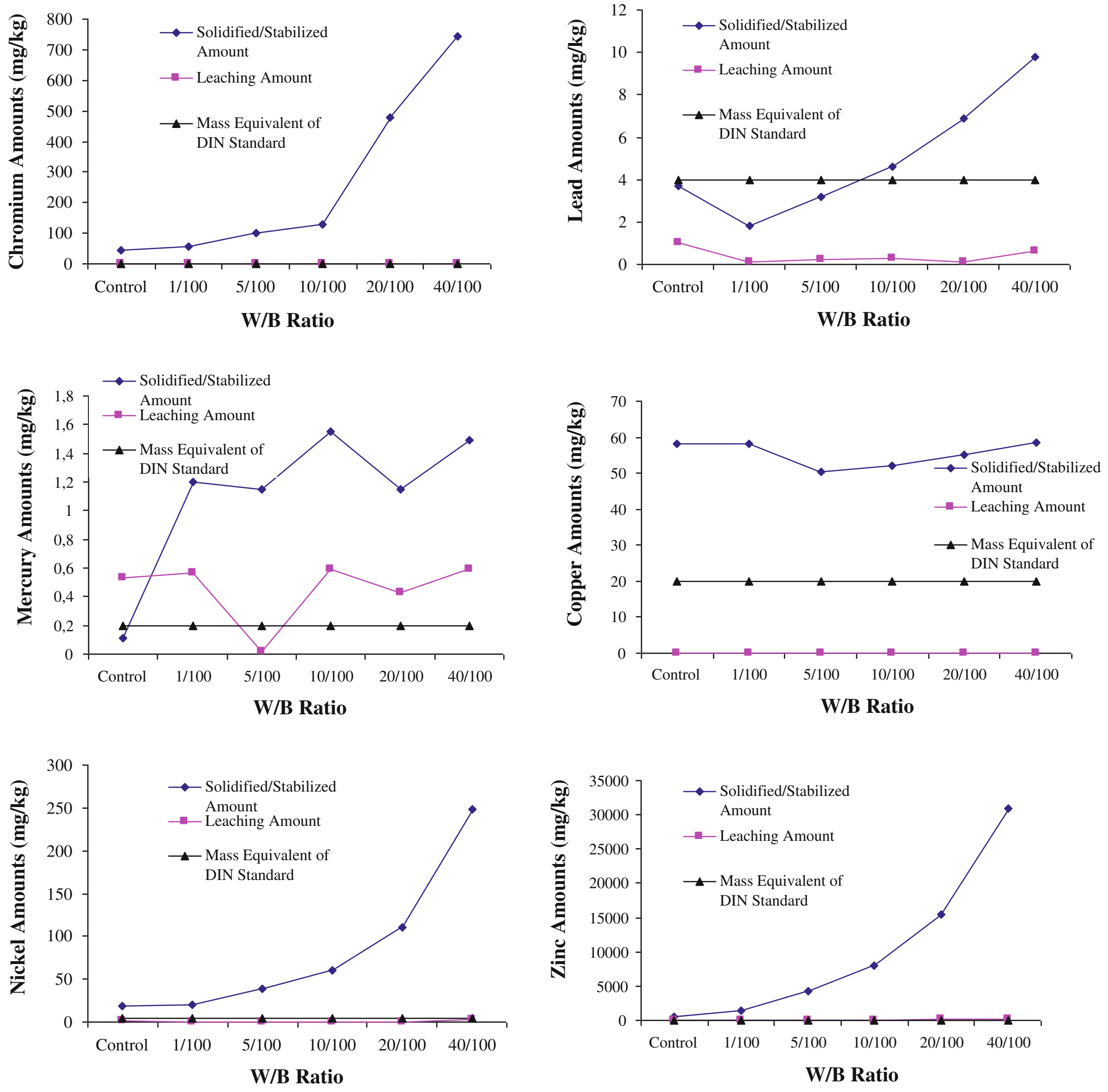


Fig. 3 Metal (Cr, Pb, Hg, Cu, Ni, and Zn) amounts as a function of W/B for 28 days

concrete and 20/100 W/B ratio could be used as C20–C25 class of concrete), and it could be ultimately disposed in a landfill area in ratio of 40/100. The high compressive strength for metal sludge (426 kgf cm^{-2}) showed that it could be used as a desired construction material as a reuse of hazardous wastes.

The compressive strength values of the solid matrix form are highly dependent on the water/cement ratio and the degree of hydration of the cement. The cement is sufficiently hydrated at a water/cement ratio of 0.48 by weight, leaving some free pore water, gel water, and air voids (Chan et al. 2000).

Water/cement ratios for each manual-mixed mixture were 0.49–0.68, and it was balanced with 200 mm of slump test value. Water/cement ratios for each mixer-used mixture were 0.53–0.68, and it was balanced with 170–180 mm of slump test values. As a result, changing concrete sample preparation conditions (decreasing slump, increasing water/cement ratios, and changing curing conditions) caused lower compressive strengths.

Leaching properties

Leaching tests were run for concrete specimens. Heavy metal concentrations in leachates of specimens are shown in Table 2.

While total concentrations of heavy metals in raw waste are very high, they were near zero or less than minimal detectable limits in leachates for most of the leached specimens. The leaching test results by DIN 38414-S4 and EPTox showed that heavy metal leaching from the specimens was lower than leaching from untreated waste before

S/S. The results showed that there was a slight amount of heavy metal leaching. The leaching test results also showed that the leaching metal amounts were comparatively less than the metal amounts in concrete specimens. It can be seen that the high metal concentrations could be stabilized by S/S at these ratios. For example, lead content of the WWTS sludge (untreated waste) is 0.95 mg L^{-1} in DIN eluate, while lead concentrations in DIN eluates of specimens have been measured between 0.011 and 0.102 mg L^{-1}. While maximal W/B ratio for S/S was 40/100, stabilized heavy metal concentrations in DIN eluates for 28 days of curing were 0 mg L^{-1} for copper, 0.099 mg L^{-1} for chromium, 0.217 mg L^{-1} for nickel, 0.065 mg L^{-1} for lead, 0.059 mg L^{-1} for mercury, and 20.668 mg L^{-1} for zinc. While maximum W/B ratio for S/S is 40/100, stabilized heavy metal concentrations in EP extracts for 28 days of curing are 0.032 mg L^{-1} for lead and 0.105 mg L^{-1} for mercury. Leaching test results by DIN 38414-S4 and EPTox showed that leaching of heavy metals from the specimens was less than leaching from untreated waste before S/S. The results showed that there was a slight amount of heavy metal leaching.

In addition, heavy metal (Cr, Pb, Hg, Cu, Ni, and Zn) amounts in concrete specimens kept for 28 days as a function of W/B ratio are shown in Fig. 3. In Figure 3, the concentration variations for each metal are given in six different figures. Metal amounts in DIN eluates for 28 day of curing period and mass equivalents of DIN standards for each metal are also shown in figures for the purpose of comparison. It could be seen that the leaching metal amounts were comparatively less than the metal amounts in concrete specimens. As could be seen from the figures, the higher the W/B ratio, the higher the metal amounts in concrete specimens. However, significant changes with W/B ratio for leaching amounts were not observed. Although the chromium-leaching amounts were similar to mass equivalents of DIN standard, they were lower than the solidified/stabilized chromium amounts in concrete specimens. The lead-leaching amounts and the solidified/stabilized lead amounts in concrete specimens were both lower than the mass equivalents of DIN standard. In addition, the lead-leaching amounts were quite less than solidified/stabilized lead amounts in concrete specimens. Although the mercury-leaching amounts were generally higher than the mass equivalents of DIN standard, it can be concluded that the high mercury amounts could be stabilized by S/S because, as it can be noted, the mercury-leaching amounts were decreased after the S/S process. The copper-leaching amounts were much lower than the mass equivalents of DIN standard and the solidified/stabilized copper amounts in concrete specimens at these ratios. As can be seen, for copper, the S/S process was efficient. Although the nickel and zinc-leaching amounts were similar to mass equivalents of DIN standards, they were lower than the solidified/stabilized amounts in concrete specimens. As can be seen, nickel and zinc showed similar leaching behavior as chromium. All these results indicated that even high metal concentrations could be stabilized by S/S at these ratios.

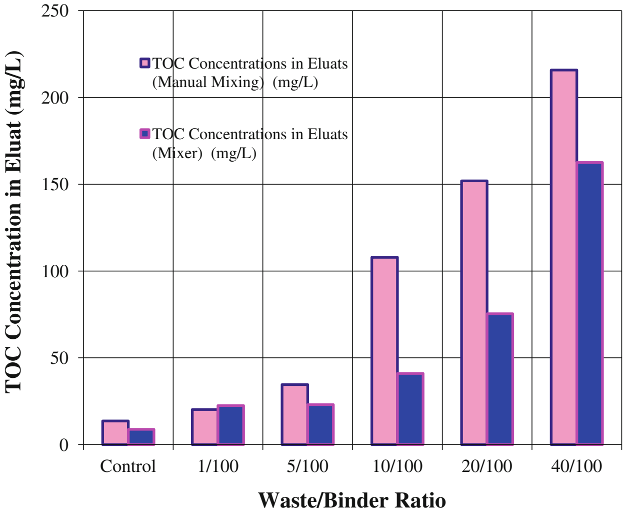


Fig. 4 TOC concentrations in DIN eluates as a function of W/B for concrete specimens

Figure 4 shows TOC concentrations in DIN eluates for 28 days of curing period. It was shown that the higher the W/B ratio, the higher the TOC concentration for this solidified sludge. It was reported that the S/S performance can be reduced by the presence of organic matters in the solidified waste; it has an influence on the cement hydration/hardened process and structural consequences on the formed materials (Vipulandan and Krishnan 1990; Tiruta-Barna et al. 2006). In this study, TOC-leaching results showed that S/S process was not inhibited by high organic content of the untreated waste (1,030 mg L^{-1} TOC). TOC-leaching concentrations of waste were less than TOC-leaching concentrations of all the concrete specimens. TOC-leaching results showed that organic material was successfully bounded and TOC removal efficiency was between 81 and 99.6 %.

It was observed that hazardous compounds in the hazardous waste were stabilized by S/S. It was also observed that the waste was no longer EP toxic after S/S, which was suitable for land disposal, while the untreated waste was EP toxic.

Conclusions

This study demonstrated the stabilization of the hazardous compounds of the industrial treatment sludge by S/S technology and helps to solve problems on the management alternatives of these hazardous wastes. Specific conclusions were as follows:

- Compressive strength measurements could be used to determine the performance of the S/S process for this sludge. Compressive strengths of concrete specimens show that the higher the W/B ratio W/B, the lower the compressive strength for this solidified sludge. It was assumed that 90 days cured specimens could be potentially used as construction materials.
- The compressive strengths of the concrete specimens were less than the 28-day characteristic compressive strength of C 35 class of concrete (450 kgf cm^{-2}) (TS EN 206-1) (Institute of Turkish Standards 2002). In order to be able to treat and reuse hazardous wastes, high class concrete should be produced, but compressive strength for low class concrete should be aimed at. As the results of compressive strengths will not decrease in time, this does not cause a problem.
- It was assessed that 10/100 W/B ratio could be used for making C30 class of concrete (370 kgf cm^{-2}), and 20/100 W/B ratio could be used as C20–C25 class of concrete (250–300 kgf cm^{-2}). 40/100 W/B ratio meets the strength criterion for land disposal.
- Hazardous waste management alternatives could be used on the produced specimens as construction material or concrete. Their direct disposal to landfill depended on W/B ratio.
- S/S process was not inhibited by high organic content of the untreated waste (1,030 mg L^{-1} TOC). TOC-leaching results showed that organic material was successfully bounded and TOC removal efficiency was between 81 and 99.6 %.
- The leaching test results by DIN 38414-S4 and EPTox showed that heavy metal leaching from the specimens was lower than leaching from untreated waste before S/S. It could be concluded that the high metal amounts could be stabilized by S/S at these five W/B ratios.
- When the leaching concentrations were examined according to the W/B ratios, sometimes, higher or lower concentrations than the expected values were observed. However, these changes could be neglected since the leaching levels were much lower than the standards. The changes in the leaching concentrations could be explained by the differences between concrete's particles sizes. For this reason, keeping the particulate size constant in future studies is of great importance.

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