



Leaching behavior of pollutants in ferrochrome arc furnace dust and its stabilization/solidification using ferrous sulphate and Portland cement

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ABSTRACT

In this study, dissolution properties under different conditions and pollution potential by toxicity characteristic leaching procedure (TCLP) of arc furnace dust generated in the production of ferrochrome were examined and some stabilization/solidification (S/S) techniques were applied to the dust depending on contaminants determined. Dissolution properties and pollution potentials of all the materials stabilized/solidified were also studied under the similar conditions. It was determined that the metallic components concentrations dissolved from the ferrochrome arc furnace dust (FAFD) except for chromium and zinc were below the detection limits. The chromium concentration dissolved from the FAFD by TCLP was found to be 9.8 mg/l. Portland cement (PC), PC–FeSO₄ and PC–sand–FeSO₄ mixtures for S/S of the FAFD were tested. Although metal ions in the cationic form were stabilized when the PC was only used, Cr(VI) in the sample was not changed depending on PC amount and remained in the soluble chromate form. The stabilization efficiency of Cr(VI) increased by the increasing amounts of PC and FeSO₄. The best S/S of the FAFD was accomplished when the 5 stoichiometric amounts of FeSO₄, 30% PC and 16% sand mixture were used. TCLP leaching results of the samples obtained under the optimum conditions were below the EPA landfilling limits.

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1. Introduction

Ferrochromium is an iron and chromium alloy, containing 45–80% Cr and various amounts of Fe, C and other elements. Ferrochromium is produced pyrometallurgically by carbothermic reduction of chromite ore (FeO·Cr₂O₃) [1]. Slag and arc furnace dusts are generated as waste material in enormous quantities during the ferrochrome production. While small percentage of the slag has been used as construction materials and aggregate [2–4], the majority of it is held in dumps. Slags can contain high levels of toxic elements [5,6].

Fine solid particles (dusts) released into environment during the ferrochrome making process are also an important pollution source due to its high levels of extractable toxic elements content which could pose environmental pollution. It has been reported that the most abundant heavy metals in these dusts are Cr, Zn, Pb, Ni and Cd [7–9]. Particularly, it has been indicated that the filter dust or its sludge produced during the ferrochromium production contain high levels of soluble hexavalent chromium [10–12]. Arc furnace dusts have been categorized as hazardous waste by USEPA, EU and a

lot of country due to their chemical, physical and leachability properties [13]. Therefore, it is not allowed to disposing of at landfills without treatment by regulations.

Stabilization/solidification (S/S) technologies are widely applied for treatment of hazardous wastes such as sludges, slags and ashes containing heavy metals. Main purposes in the S/S processes are to reduce the hazard of a waste by converting the contaminants into less soluble, mobile or toxic forms and to encapsulate the waste in a monolithic solid of high structural integrity by using some additives such as metal stabilization additives and binding materials [14]. Since electric arc furnace dusts are considered as hazardous pollutants, several researchers have focused on the stabilization of them [15–18]. For this purpose, Salihoglu et al. have studied properties of steel foundry electric arc furnace dust solidified/stabilized with Portland cement. In the study, leachability of lead and zinc from the dust solidified/stabilized has been evaluated by TCLP and SPLP and it has been found that the concentrations of the lead and zinc released exceeded the EPA landfilling limits [13]. Solidification/stabilization of electric arc furnace dust using coal fly ash was investigated by Pereira et al. [19]. The solidification/stabilization mechanism and leaching behaviors of the Pb, Zn, Cd and total Cr were determined depending on pH, TCLP and DIN-38414 S4. They have reported that the final pH of the leachate must be within a range of values corresponding to the minimum solubility of the

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Table 1
Chemical composition of FAFD

Constituents	%
Mg	17.18
Cr	13.90
Si	10.13
Fe	5.19
Al	2.83
Zn	1.50
Ca	0.99
Cu	0.03
Mn	0.18
Ni	0.13
Pb	0.02
Ti	0.07

metals in the leaching medium, which is achieved in the pH 8.0–11.3 interval for TCLP leachates. Similar investigations by using cement-based additives for stabilization/solidification of wastes containing heavy metal have also been made by Andres et al. [20], Valls and Vázquez [21], Janusa et al. [22], Diet et al. [23], Ruiz and Irabien [24], Islam et al. [25], Zain et al. [26], Viguri et al. [27].

Arc furnace dusts contain zinc, nickel, lead and cadmium and trivalent and hexavalent chromium. Oxidation stage of the chromium in the some researches related to stabilization/solidification and leaching behavior of chromium in these wastes has not been taken into account. Although S/S of the heavy metals having cationic form in the wastes could be achieved, it has been reported that Cr(VI) in the wastes stabilized/solidified released into leaching solution [28–30]. Cohen and Petrie were studied dissolution of chromium and zinc in ferrochromium flue dusts by cement-based solidification. They also taken into account chromium species together with other metal contaminants in their study and found that the alkaline nature of the cementitious product reduces the solubility of Cr(III) and zinc, but Cr(VI) in the solidified waste is mobile.

Chromium contamination is a significant problem since hexavalent form of chromium is highly mobile, toxic and carcinogenic to living organisms [31,32]. The trivalent chromium is quite immobile in the water environment because of limited solubility of its hydroxide which is readily formed in the natural pH range of waters. Therefore, detoxification and immobilization processes of Cr(VI) is based on its reduction to Cr(III). For this purpose, it is needed to use some additives having reducing potential for Cr(VI). Kindness et al. used blast furnace slag in order to fix trivalent and hexavalent chromium ions in the solid waste. They have reported that Cr(VI) can reduced to Cr(III) in the presence of blast furnace slag [33].

Point of view the knowledge mentioned above, in this study, dissolution properties under different conditions and pollution potential by TCLP of arc furnace dust generated in the ferrochrome production were examined and some S/S techniques were applied to the dust depending on contaminants determined. Dissolution properties and pollution potentials of all the samples stabilized/solidified obtained were also studied under the similar conditions and results were compared.

2. Materials and methods

2.1. Materials

Ferrochrome arc furnace dust (FAFD) used in the study was collected from Eti Krom AS located near Elazığ, Türkiye. The chemical composition of the sample is presented in Table 1. As seen in the table, sample contains 13.9% Cr, 17.18% Mg, 10.13% Si, 5.19% Fe, 2.83% Al and 1.5% Zn as main components. The FAFD sample mean particle size of which is about 105 μm was used in the experiments.

Ordinary Portland cement (PC), sand and ferrous sulphate (Merck-1.03965) were used as S/S reagents. The working solutions were prepared by analytical reagent grade chemicals and distilled water.

2.2. Experimental procedure

2.2.1. Leaching tests

Batch leaching experiments and TCLP test method were used to evaluate the leaching and pollution potentials of pollutants in untreated and treated samples. Effects of the pH, contact time and liquid/solid ratio on leaching behaviors of the pollutants in the FAFD were investigated in the batch leaching experiments. These experiments were carried out in the batch reactors (250 ml erlenmeyer) containing various amounts of sample and 100 ml of solutions having different pH. The batch reactors were shaken at 150 rev/min by using a flask shaker (Gallenkamp) for different contact times. All of the experiments were carried out at 25 °C.

Batch leaching experiments applied to the samples stabilized/solidified were carried out at a constant contact time and liquid/solid rate depending on pH. At the end of each experiment, the mixtures were filtered and then the final pH of the filtrates was measured by a pH meter. The filtrates were acidified with 1 ml of HNO₃ solution to prevent the metal ion precipitation and then they were analyzed.

In order to determine pollution potentials of the FAFD and its products stabilized, the toxicity characteristics leaching procedure (TCLP) was also applied to them. For this purpose, 100 g of samples were placed separately in a plastic bottle together with 2000 ml of leach solution, sodium acetate/acetic acid buffer solution. The mixtures were then agitated at 30 \pm 2 rpm and 22 \pm 3 °C for 18 \pm 2 h. The mixture was filtered through a 0.45 μm glass fiber filter and the filtrates were analyzed.

The experiments were performed in duplicate and mean values were taken into account.

2.2.2. Stabilization/solidification experiments

In order to stabilize/solidify to the FAFD, mortars having different compositions were prepared by mixing various amounts of PC, ferrous sulphate and sand and 0.3 ml of distilled water/g solid mixture. Amount of the water used was determined to be 0.3 ml per gram of solid. This amount does not cause ooze and can turn the mixture into mud. The mortars were then poured into cylindrical polyethylene moulds (50 mm diameter and 150 mm high) and compacted. The samples were cured for 28 days in a controlled moisture chamber (90% RH). After 28 days, they were crushed to 2.36 mm (8 mesh), and subjected to the leaching experiments.

2.3. Methods of analysis

The concentration of the metal ions in the solutions was determined by atomic absorption spectrophotometer (Ati-Unicam 929). In order to determine the hexavalent chromium content of the extracts, they were analyzed colorimetrically with 1,5-diphenyl carbazide method by using Jenway 6105 Spectrophotometer [34].

Standard solutions were prepared by using analytical chemicals. All dilutions were made by distilled water.

3. Results and discussions

3.1. Leaching behavior of FAFD

Wastes can be affected from the some environmental conditions in landfilling or stored area. For example; concentration of the contaminants released can change depending on pH, contact time and

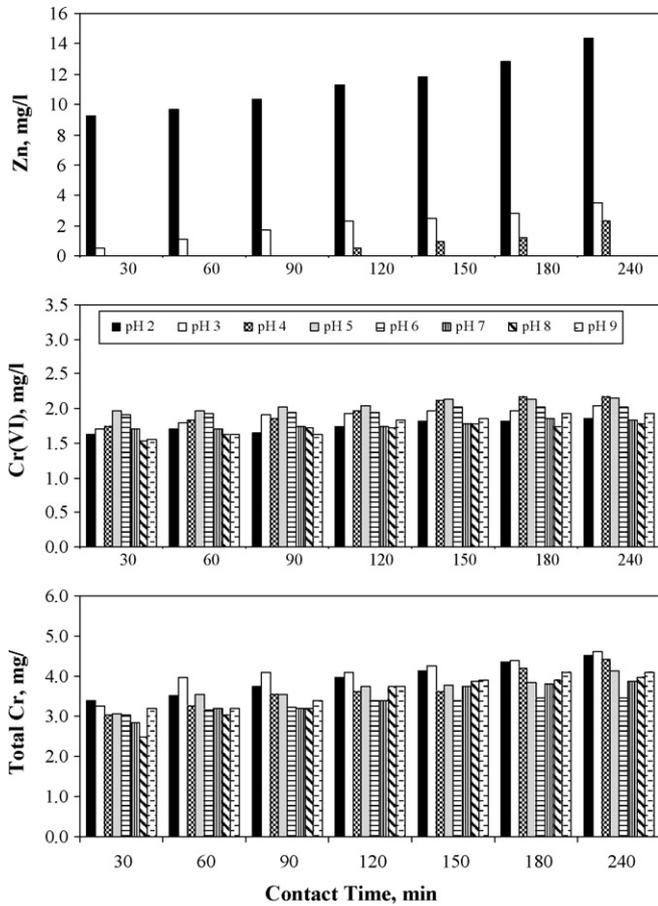


Fig. 1. The variations of total chromium, Cr(VI) and Zn concentrations released from the FAFD depending on pH [liquid/solid: 20].

amount of rains (liquid/solid ratio) or water contacted. This situation has been taken into account in the standard test methods determining the pollution potentials of the wastes [35–38]. Starting from this point, effects of pH, contact time and liquid/solid ratio on the dissolution of metal constituents in the FAFD were investigated. The obtained results are presented in Figs. 1 and 2. Although the FAFD contains different heavy metals constituents such as Cr, Fe, Al, Zn, Cu, Mn, Ni, Pb and Ti, concentrations of the metal ions released from the dust except for Cr and Zn were below the detection limits probably due to final pH of the solution determined in the range of 7.24–9.92. Therefore, concentrations of the zinc, total and hexavalent chromium have only been given in the figures.

The concentrations of the zinc and total chromium in the leaching solutions increased with the increasing contact time and the decreasing pH. However, it has been determined that the Cr(VI) in the dust sample can dissolve in the first 30 min and amounts of the dissolved do not depend on pH and contact time (Fig. 1). Under the investigated conditions, concentrations of the hexavalent chromium, total chromium and zinc dissolved from the sample were varied in the concentration ranges of 1.5–2.2; 3.5–4.7 and 9.4–14.5 mg/l, respectively.

In order to determine effect of the liquid/solid ratio on the metal dissolution from the dust, the solutions having different initial pH in the range of 2–9 were contacted with the FAFD which has 5–100 range of liquid/solid ratio at temperature of 25 and contact time of 60 min. As seen from Fig. 2, the metal concentrations released into the solution decreased with increasing liquid/solid ratio. It has been determined that Cr(VI) and total chrome concentrations (close to

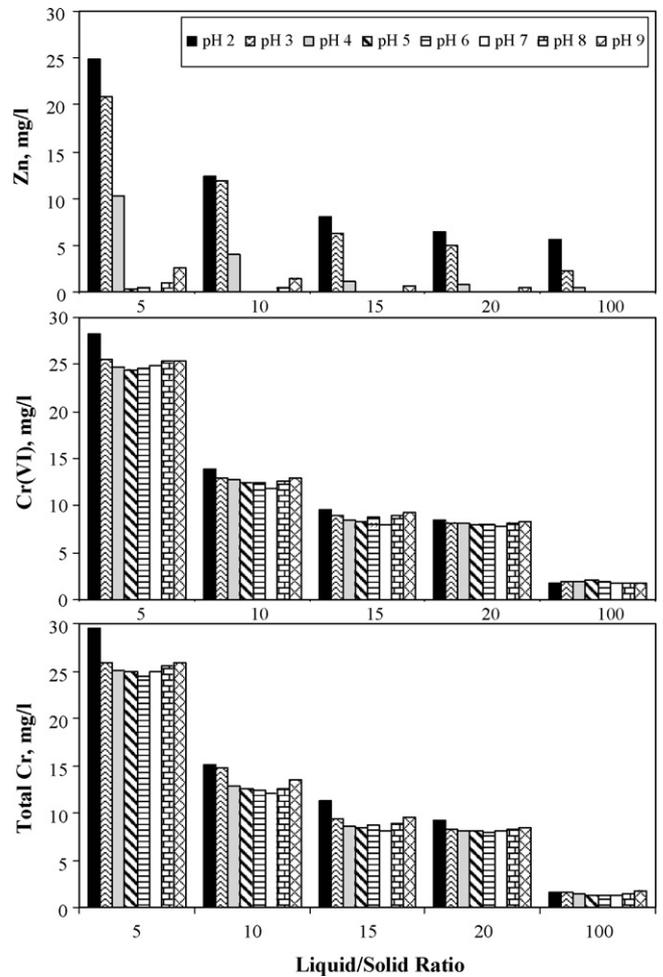


Fig. 2. The variations of total chromium, Cr(VI) and Zn concentrations released from the FAFD with liquid/solid ratio depending on pH [contact time: 60 min].

other) did not change with the increasing pH. However, zinc dissolved in different concentrations with the increasing pH due to its amphoteric properties.

Solid wastes are classified as hazardous or harmless depending on their toxic constituent contents and dissolved concentration levels determined by applying TCLP test. Liquid/solid ratio in TCLP is 20. In order to compare the results obtained with TCLP results, it can be said that the concentration of metal ions released into solution at the liquid/solid ratio of 20 is more meaningful than the other. When these concentration values are compared, it can be seen that the chromium levels released from the FAFD are higher about twofold than the limit given as 5 mg/l for TCLP. Therefore, FAFD is a hazardous material for environment.

3.2. Stabilization/solidification (S/S) of FAFD

The leaching experiments have showed that the FAFD is a hazardous material depending on concentration value of chromium released. In order to prevent metal dissolution from the FAFD, some S/S procedures were tested. Since PC is a most widely used as a binder and stabilization material in the S/S processes, in the first group experiments, mortars having different compositions were prepared by mixing of the FAFD and various amounts of PC in the range of 10–50%. The stabilized/solidified samples obtained after 28 days of curing time were subjected to the leaching experiments. Fig. 3 shows the variations of total chromium, Cr(VI) and zinc

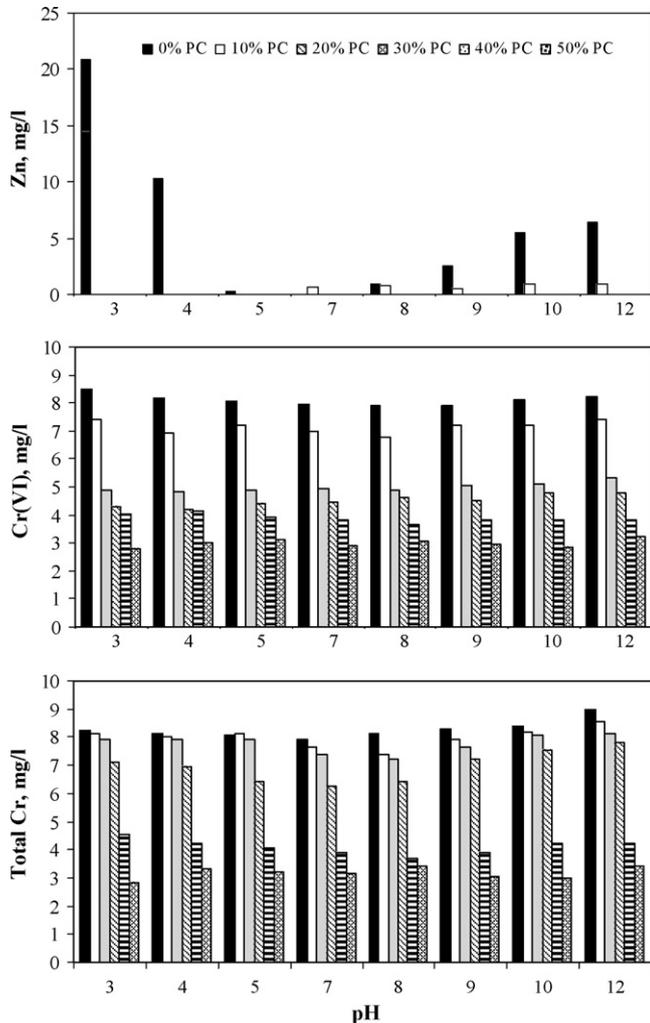


Fig. 3. The variations of total chromium, Cr(VI) and Zn concentrations released from the FAFD with pH depending on amount of Portland cement [liquid/solid: 20; contact time: 60 min].

concentrations released from stabilized and solidified samples with PC as a function of pH.

The concentrations of metal ions released from the samples were decreased with the increasing amount of PC. However, Cr(VI) did not change with the amount of PC. While total chromium concentrations were determined as near that of the Cr(VI), concentrations of other metal ions were below the detection limits. Final pH of the solutions was determined in the range of 8.84–12.25. These results show that the PC can stabilize the metal ions in the cationic form in the FAFD by precipitation in this pH range. Similar results have been reported in some researches that the S/S of metal ions in the form of cationic has been studied [20–25]. It was observed that the waste in a monolithic solid of high structural integrity could not be obtained. That is, PC could not solidify the FAFD. This situation can be attributed to the nonpuzzolonic character of the FAFD.

When the hazardous constituents dissolved from the FAFD are taken into account, Cr(VI) must be considered as the most important pollutant and to be stabilized constituent. In order to stabilize the Cr(VI) in the form of CrO_4^{2-} in the stabilized/solidified waste, a stabilization additive for Cr(VI)-binding must be added into the mixture. Since S/S binders are highly alkaline, precipitation is a major reaction in immobilization by PC. Cationic metal species in the mobile phase convert to metal hydroxides [39]. One of the main

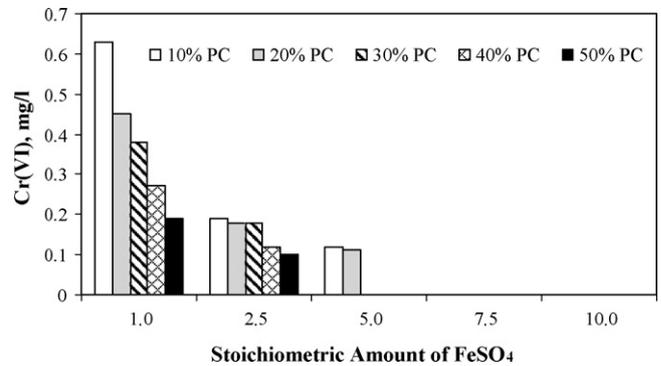


Fig. 4. Leachability of Cr(VI) in the S/S products depending on amounts of Portland cement and FeSO_4 [liquid/solid: 20; contact time: 60 min].

purposes in stabilization process is to obtain a metal compound having low solubility. Cr(VI) compounds having low solubility are barium, silver and lead chromate [40]. In order to form this compound in the waste, their soluble salts must be used. But, they can definitely not be used as a S/S material due to their highly toxic properties. In this situation, reduction of chromium from hexavalent to trivalent is unique solution method.

In the stabilization of toxic pollutants, various reactions such as neutralization, precipitation, redox, adsorption, ion exchange and complex forming may occur [39,41]. Among these reactions, redox reactions have important role on the Cr(VI) mobility. Trivalent chromium is more likely to be precipitated or sorbed than hexavalent chromium under most conditions. Cements generally provide a moderately oxidizing environment, but the addition of blast furnace slag can produce reducing conditions by releasing sulfide and other reduced sulfur compounds [42,43]. Furthermore, reductants such as ferrous iron can be added to promote reduction of contaminants for immobilization [39,41]. In light of the foregoing, ferrous sulphate, is the most commonly used reagent for reduction of hexavalent chromium [44,45], was used as an immobilization reagent for Cr(VI) in the FAFD. For this purpose, firstly, stoichiometric amount of ferrous sulphate required for reduction of whole Cr(VI) in the FAFD was calculated according to Eq. (1). Then, FAFD-PC mixtures were prepared with ferrous sulphate solutions (0.3 ml/g mixture) containing 1; 2.5; 5; 7.5 and 10 stoichiometric amount of FeSO_4 . The mortars prepared were moulded, cured for 28 days and crushed. The samples stabilized/solidified were subjected to the leaching experiments. The obtained results are presented in Fig. 4.



Important amount of the chromium could be immobilized with the increasing amount of FeSO_4 . Cr(VI) in the samples stabilized/solidified with the least PC and FeSO_4 released in the lower concentrations. When the concentrations of Cr(VI) given in Figs. 3 and 4 are compared, it can be seen that the amount of the Cr(VI) released into leaching solution decreased about 92% by S/S using the 10% of PC and 1 stoichiometric amount of FeSO_4 .

The concentrations of Cr(VI) released from the samples stabilized/solidified by stoichiometric amount of $\text{FeSO}_4 \geq 5$ and amount of $\text{PC} \geq 30\%$ were below the detection limit of 0.05 mg/l (Fig. 4). Therefore, it can be stated that the 5 stoichiometric amount of FeSO_4 and 30% PC are optimum dosages for S/S of the FAFD. Although S/S of the FAFD could be achieved under these conditions, it was observed that a monolithic solid waste of high structural integrity could not also be obtained and the samples were broken into small pieces by friction and stroke effects. In order to eliminate this disadvantage, sand was added to the mixtures and different S/S experiments were

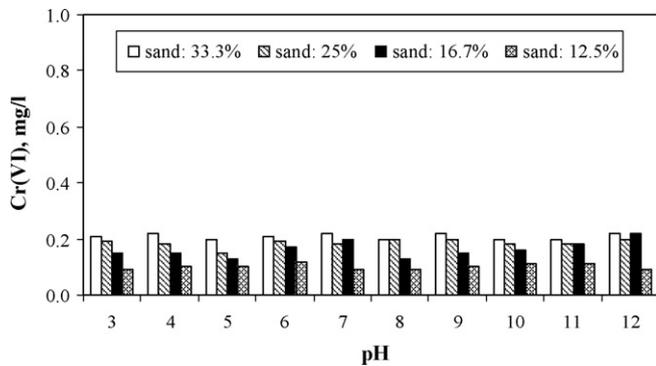


Fig. 5. The variation Cr(VI) concentrations released from the S/S products with pH depending on amount of the sand [FeSO_4 amount: 5 stoichiometric; liquid/solid: 20; contact time: 60 min].

carried out with sand in the range of 12.5–33.3%, FAFD, 30% PC and 5 stoichiometric amount of FeSO_4 . The results are given in Fig. 5.

It was observed that the samples prepared by using different amounts of sand had more solidity than the sample not containing sand. Zinc concentration values released from all the samples stabilized/solidified were found to be below the detection limits. But, Cr(VI) dissolved in the concentration range of 0.1–0.2 mg/l (Fig. 5).

Consequently, it has been determined that the FeSO_4 has an important role in the Cr(VI) stabilization in FAFD and a pozzolanic solidification additive such as sand to obtain a monolithic solid waste is necessary.

3.3. TCLP test results

In order to determine if the stabilized/solidified products can be disposed of at a landfill site with domestic waste or at a segregated landfill, TCLP test was subjected to the all samples. The results are shown in Table 2.

TCLP test show that the concentration of the chromium dissolved from FAFD is 9.81 mg/l. Since this concentration value is above the allowable limit of 5 mg/l, FAFD must be considered a hazardous pollutant and must not be discharged unless it is stabilized and solidified. Similar result has been found by Laforest and Duchesne [46] who are studied characterization and leachability of electric arc furnace dust (EAFD) made from remelting of stainless steel. It has been determined that the EAFD contain 10.9% Cr, 4.1% Ni, 1.4% Pb and 5.2% Zn and the total chromium and Cr(VI) concentrations (9.7 and 6.1 mg/l, respectively) in the test solution

Table 2
TCLP tests results of FAFD and stabilization/solidification products

Solid sample	Dissolved metal concentration (mg/l)	
	Cr	Zn
FAFD	9.81	103.85
10% PC + FAFD	6.56	27.84
20% PC + FAFD	4.84	0.37
30% PC + FAFD	4.10	UDL ^a
40% PC + FAFD	1.8	UDL ^a
50% PC + FAFD	1.3	UDL ^a
10% PC + 1 stoichiometric FeSO_4 + FAFD	0.16	56.4
20% PC + 2.5 stoichiometric FeSO_4 + FAFD	0.04	1.61
30% PC + 5 stoichiometric FeSO_4 + FAFD	0.12	1.57
40% PC + 7.5 stoichiometric FeSO_4 + FAFD	UDL ^a	0.19
50% PC + 7.5 stoichiometric FeSO_4 + FAFD	UDL ^a	UDL ^a
33.3% sand containing S/S product	1.03	UDL ^a
25% sand containing S/S product	0.69	UDL ^a
16.7% sand containing S/S product	0.12	UDL ^a
12.5% sand containing S/S product	UDL ^a	UDL ^a

^a UDL: under the detection limits.

exceeded the toxicity characteristic regulatory level. In our study, TCLP test results showed that the concentrations of the chromium released from the all S/S samples (except for 10% PC) were below the 5 mg/l. Therefore, the S/S products obtained under the investigated conditions can be considered as harmless. But, in the leaching experiments, it has been determined that the chromium in the lots of samples partly dissolved and some of the samples do not have a monolithic solid waste of high structural integrity. Among these S/S samples, the sample obtained by using 30% PC, 12.5% sand and 5 stoichiometric amount of FeSO_4 is more stable and has a high structural integrity than the others.

4. Conclusions

In this study, dissolution properties under different conditions and pollution potential by TCLP of the FAFD were examined and some S/S techniques were applied to the dust depending on contaminants determined. Dissolution properties and pollution potentials of all the stabilized/solidified materials prepared were also studied under the similar conditions.

In the dissolution experiments, it was determined that except for chromium and zinc, metallic components concentrations dissolved from the FAFD were below the detection limits. However, chromium and zinc in different concentrations released into the solution. The chromium concentration dissolved from the FAFD by TCLP was found to be 9.8 mg/l, is higher than the maximum allowable concentration of 5 mg/l. According to TCLP test result, it has been concluded that the FAFD has an important pollution potential, it is a hazardous waste.

For the stabilization/solidification of the FAFD, PC, PC– FeSO_4 and PC–sand– FeSO_4 mixtures were tested and it was found the following results.

When the Portland cement was only used, although metal ions in cationic form were stabilized, Cr(VI) in the sample was not changed depending on Portland cement addition and remained in the soluble chromate form. Also, weak structural integrity was observed due to non-pozzolanic properties of the FAFD.

In the stabilization/solidification experiments carried out with PC– FeSO_4 mixture, the stabilization efficiency of Cr(VI) increased by the increasing amounts of PC and FeSO_4 . Optimum PC and FeSO_4 dosage was determined to be 30% and 5 times of stoichiometric amount calculated from Cr(VI) reduction stoichiometry by ferrous iron with respect to Cr(VI) content of the FAFD, respectively. Even though effective stabilization of the FAFD under the conditions investigated was established, monolithic solid residue could not be obtained.

In order to both stabilize and solidify to the FAFD, PC–sand– FeSO_4 mixtures were also tested. The best S/S of the FAFD could be accomplished when the 5 stoichiometric amount of FeSO_4 , 30% Portland cement and 16% sand mixture was used. TCLP test results applied to the samples stabilized/solidified showed that the samples could be disposed of a landfill.

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