PERFORMANCE OF pH STATIC LEACHING TEST FOR EVALUATING LEACHABILITY OF SOLIDIFIED WASTE FORMS

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Abstract: The pH static leaching procedure (PSLP) is useful for measurement of total potential leachability in accordance with the continuing addition of acidic leachant up to the point of the maximum breakdown of the cement-based solidified waste forms (SWF). A rate of dissolution of calcite-additional Pb-doped SWF (KC5P10) some 4-5 times slower than Pb-doped SWF (KP10) is apparent. The release of calcium in KC5P10 was slightly lower than that in KP10, and that of lead in KC5P10 was dramatically lower than that in KP10. The difference in remarkedly less reduction in weight of KC5P10 is also indicative of the markedly higher resistance of KC5P10 to acid attack compared to that of KP10. In the PSLP, the leaching of Pb from Pb-doped SWF is proposed to be dependent on the amount of initial Pb concentration as well as the meq CH₃COOH added/g dry solidified waste during the PSLP. A maximum release of lead from Pb-doped SWF is found at the point at which a maximum amount of meq CH₃COOH is added, and a plot of the leaching concentration (mg/L) and meq CH₃COOH added/g dry solidified waste reveals that there is a quadratic relationship rather than a linear relationship between the two quantities.

Key Words: Cement, calcite, Lead, pH, pH Static Leaching Test

INTRODUCTION

The quantity of hazardous waste, radioactive waste and contaminated land has increased with the development of industrial activities. Solidification/stabilization is a recognized technique for disposing waste materials with common methods including cement and lime based techniques, glassification, thermoplastic techniques, and encapsulation. The cement-based solidification/stabilization (S/S) processes have widely been used in waste disposal due to its relatively low cost, an extensively documented history of use and an accessibility of technology. The cement-

based S/S processes produce solids to be non-hazardous or less hazardous than the original waste, and to prevent pollutants from migrating into the environment.²⁾

In recent years leaching methodologies to assess solidified waste materials have been discussed for managing the undesired release of contaminants into the environment. Actually, the release of contaminants from waste is influenced by a large number of physical (e.g. particle size, temperature, porosity) and chemical (e.g. pH, redox, reaction kinetics) parameters. A variety of test procedures are available to characterize materials with respect to their leaching behavior.

Portlandite and calcium-silicate-hydrate (C-S-H) (and sometimes CaCO₃), which are major hydrated components constituting the acid

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neutralizing capacity (ANC), dominate the solution chemistry of cement-water systems to be alkaline conditions. The alkalinity of cementitious materials rectifies a low pH of a leachate (normally below pH 5.0 in most leaching test) to be over 12.0 for a couple of hrs. The rapid change of pH produces the remarked variation of solubility of hazardous substances, particularly heavy metals.

The high variation of solubility by the change of pH makes some limitations for measuring the precision of release of contaminants in many methodologies, even in the US-environmental protection agency (EPA) toxicity characteristic leaching procedure (TCLP) undoubtedly recognized as one of the most common leach tests. The precision of leachate concentration determined by the TCLP was found to be poor with five replicates. These results are entirely consistent with the work of Stegemann and Cote.⁵⁾ They investigated the precision of four replicates as well as the number of replicates required to achieve a desired precision of ×/÷ 1.5 (i.e., 50%). The precision of each four replicates of different contaminants varied from \times/\div 1.2 to 3.8 except lead. The precision of lead was \times/\div 19. The number of replicates for a desired precision of \times/\div 1.5 ranged from 4 to 19. They suggest that more replicates may be required to increase the precision for the regulatory purposes intended by the EPA.

The purpose of this study is to perform a new leaching method, the pH static leaching procedure(PSLP), for assessing a quantitative measurement of total potential leachability with overcoming the remarked variations of solubility by the change of pH at the attack of acid. The PSLP was implemented in accordance with the continuing addition of acidic leachant up to the point of no change of the leachate pH produced at the maximum breakdown of the cement-based solidified form. In addition, attention was particularly given to the effects of calcite in the S/S of lead.

EXPERIMENTS

Materials

Ordinary Portland cement was purchased from Sangyong Cement in Korea and used in all studies. The cement has the following mineralogical composition: (i) 41.7% of tricalcium silicate (Ca₃SiO₅: C₃S), (ii) 27.9% of dicalcium silicate (Ca₂SiO₄: C₂S), (iii) 12.7% of tricalcium aluminate (Ca₃Al₂O₆: C₃A), (iv) 9.4% of calcium aluminoferrite (Ca₄Al₂Fe₂O₁₀: C₄AF), and (v) 2.9% of calcium sulfate (CaSO₄ \cdot 2H₂O).

Calcite used was of high grade crystalline form (\geq 95.0% CaCO₃) with \leq 4.0% MgCO₃ and \leq 1.5% acid insolubles. Its specific gravity was 2.7 and its BET specific surface area was 5.2 m²/g. It was ground such that it exhibited an average diameter (D₅₀) of 1.8µm, maximum particle size of 8 µm, and residue on 20 µm screen of no more than 0.005%. The addition of up to 5% calcite to cement clinker has been recommended by the American Society for Testing and Materials (ASTM), the Canadian Standards, and many countries in Europe. 6) Calcite addition to cement (i) acts as a partial substitute for gypsum as a set controller, (ii) results in some reduction in the energy costs for grinding clinker, and (iii) results in improvements in some characteristics of the hardened concrete.

The wastes were prepared from lead nitrate which precipitated to form hydroxide sludge on adjusting the pH to 8.5 with 6.0 N sodium hydroxide and then dried at 104°C for 24 hours. The major crystalline phases present were identified by X-ray diffraction to be lead nitrate hydroxide, [Pb₂(NO₃)(OH)₃] and lead oxide nitrate hydroxide [Pb₆O₃(NO₃)₂(OH)₄]. These XRD data were consistent with the results of Asavapisit *et al.*⁷⁾ and Gress *et al.*⁸⁾

Dry Pb waste and cement were mixed with water at a water/solid (cement, calcite and Pb wastes) ratio (W/S) of 0.3. After thorough mixing, the samples were introduced into polyethylene cylindrical moulds measuring 20mm diameter \times 40 mm height and were removed

after 24 hours. The curing was carried out in humid air at 20°C for 28 days. The samples are denoted KCiPj where i is the calcite content, j is the Pb-doped waste content. In this study, samples were prepared in triplicate and are denoted KP10 (OPC + 10% lead wastes) and KC5P10 (OPC + 10% lead wastes + 5% calcite). The samples were cured for 28 days, and then dried at 104°C for 24 hours for leaching test.

Methods

The pH static leaching procedure(PSLP) was performed at the ratio of water/solid(W/S) = 20under pH-controlled condition (pH 5.0 ± 0.1) using automatic pH control equipment with addition of 0.5 N CH₃COOH. The metal analysis of leachate in accordance with extraction time (16 times sampling for 60 hrs) and meq CH3COOH added/g dry solidified waste (up to addition of 25.0 meg CH₃COOH) was carried out by ICP-AES using a Perkin Elmer 3030 spectrometer. A half of maximum concentration of leachate was generally investigated around 6~8 meq CH₃COOH added/g dry solidified waste and 10-18 hrs extraction time. In addition, the PSLP could provide the maximum acid neutralization capacity (ANC) of solidified waste samples for measuring the potentially maximum release of wastes.

RESULTS AND DISCUSSION

The pH Static Leaching Procedure

In the pH static leaching procedure (PSLP) used here, the pH of the reaction mixture is maintained at 5.0 + 0.1 using automatic pH-controlled acetic acid addition. The PSLP is performed particularly for measurement of total potential leachability in accordance with the continuing addition of acidic leachant up to the point of no change of the leachate pH, which is the maximum breakdown of the cement-based solidified forms through empirical investigation of only a few days. Therefore, in this study, the meq CH₃COOH added (per 1 g dry waste solidified material) is an important factor for the assessment of the potential hazards in alkaline cement-based solidified materials.

The lead and calcium release of the leachate as a function of both the extraction time and meq CH₃COOH added/g dry solidified waste is shown in Figure 1. The release of calcium in lead contaminated materials (KP10) leachate increased relatively rapidly in the first 10 hours to around 4,000 mg/L and then proceeded to increase more slowly to 6,000 mg/L at which it plateaued after about 25 hours. Calcium is mainly released in the first leach by the breakdown of portlandite (Ca(OH)₂) which produces 0.2-0.3 g per 1.0 g cement corresponding to an ANC of 8 meq/g dry cement.9) The lead concentration in the leachate increased to around 1,000 mg/L in the first 25-hours after which very little additional lead was released.

On the other hand, on addition of calcite (KC5P10) the release rate of calcium dropped

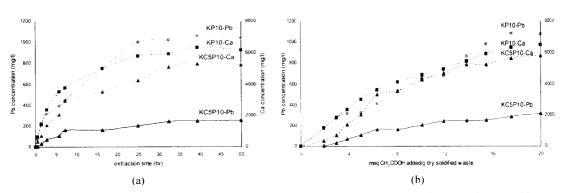


Figure 1. Pb concentration and Ca concentration of leachate by the PSLP in accordance with (a) extraction time (b) meg CH₃COOH added/g dry solidified waste.

slightly but was reasonably similar to that for KP10. In comparison, the rate and extent of release of lead from calcite-additional SWF (KC5P10) was markedly lower than that from KP10. The lead concentration was relatively stable at approximately 200 mg/L after 8 hrs and thereafter.

It has been investigated, in our earlier studies using scanning electron microscopy (SEM)/ electron dispersive spectroscopy (EDS) and X-ray diffraction (XRD), 10,111 that lead species form an extremely insoluble coating on the cement clinker in the hydration of cement. These coatings appear to involve formation of lead precipitates with hydroxide, carbonate and sulfate anions such as lead carbonate sulfate hydroxide [Pb₄SO₄(CO₃)₂(OH)₂, JCPDS file No. 38-354] and lead carbonate hydroxide hydrate [3PbCO₃.2Pb(OH)₂.H₂O, JCPDS file No. 9-356] by XRD investigations. 10,12) The effect of lead precipitates significantly slows the development of hydrated products (with a marked reduction in the S/S properties of the SWF). In the initial leaching, the rate of fast release of lead will be dominated by the dissolution of these lead precipitates.

On the other hand, the addition of calcite markedly accelerates the rate of cement hydration in the SWF. In the early stage of hydration, X-ray diffraction investigation 10,12) confirmed the presence of similar lead precipitates found in KP10, however these solids had essentially disappeared after 14 days of curing with the subsequent development of portlandite and C-S-H. In addition, with the onset of hydration, new peaks of calcium lead silicate (sulfate) hydrates (C-Pb-S-H) appeared. These calcium lead silicate sulfate hydrates are apparently forms in which lead is incorporated in the

silicate matrix on dissolution of the lead hydroxide/carbonate/sulfate solids. Microscopic and spectroscopic analyses by SEM/EDS found the morphology of the calcium lead silicate hydrate to be "coral-like" crystalline structure.¹¹⁾

It is expected that the calcium lead silicate sulfate hvdrate in calcite-additional **SWF** (KC5P10) will be more resistant to acid attack than lead solids such as Pb(OH)2(s) and PbCO₃(s). Dutre and Vandecasteele¹³⁾ have found that lead silicate crystalline material is 10 to 10⁴ times more insoluble than lead hydroxide and lead carbonate at pH 8 in equilibrium with water at 25°C and latm. Consequently, the addition of calcite, which encourages hydration of clinker components with the release of silica and the resultant formation of silica-rich gels containing lead in the cement solution, could provide an effective method of "fixing" lead against acid attack. Indeed, as can be seen in Figure 1(a), a rate of release of lead in KC5P10 is some 4-5 times slower than KP10 is apparent.

From Figure 1(b), we see that the releases of calcium and lead were reasonably proportionate to the meq CH₃COOH added/g dry solidified waste. As found in the time-dependent plots, the release of calcium in KC5P10 was slightly lower than that in KP10, and that of lead in KC5P10 was dramatically lower than that in KP10. Interestingly, the difference in reduction in weight of KP10 and KC5P10, 62.7% (for 50 hrs extraction) and 48.0% (for 75 hrs extraction) respectively (Table 1), is also indicative of the markedly higher resistance of KC5P10 to acid attack.

Comparison of the leached concentration of each element at 8 meq and 20 meq CH₃COOH added/g dry solids is shown in Table 2. For 8

Table 1. A remaining weight of solidified sample in the pH static leaching procedure

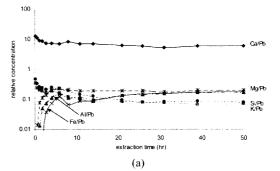
	Monitoring time (hr)	meq CH ₃ COOH added/g dry solidified waste	Initial weight (W _i)(g)	final weight (W _f)(g)	$(W_i - W_f)/W_i$
KP10	50	24.8	60.0	22.4	0.63
KC5P10	75	20.0	60.0	31.2	0.48

		Pb	Ca	Si	Al	Fe	Mg	K
	KP10 (A)	671	4670	60	70	58	129	87
8 meg	KC5P10 (B)	243	3490	42	36	31	139	72
•	B/A	0.36	0.75	0.70	0.51	0.53	1.08	0.83
	KP10 (C)	992	6490	81	166	167	188	69
20 meg	KC5P10 (D)	318	5820	47	77	78	174	60
•	D/C	0.32	0.90	0.58	0.46	0.47	0.93	0.87
/20	KP10 (A/C)	0.67	0.72	0.74	0.42	0.35	0.69	1.26
meq/20 meq	KC5P10 (B/D)	0.76	0.60	0.89	0.47	0.40	0.80	1.20

Table 2. Concentration (mg/L) of each element at 8 meq and 20 meq CH₃COOH added/g

meq/g dry solidified waste, about three times the concentration of lead was leached from KP10 compared to KC5P10 while for aluminum and iron, double the concentration was leached from the calcite-free wastes. At least 30% more calcium and silicate were leached from KP10 compared to KC5P10 for 8 meq CH3COOH added/g dry solids. In the case of 20 meq CH₃COOH added/g dry solidified waste for lead, aluminum, iron, magnesium, and potassium, similar ratios compared to that found with 8 meg/g dry solidified waste were observed in relative release rate from KC5P10 and KP10. Only the ratio of calcium was higher, and that of silicate was reduced. In KP10, lead, calcium and silicate at 8 meg CH₃COOH added were released to approximately 70% of those at 20 meq CH3COOH added. In KC5P10, lead, calcium and silicate at 8 meg were released to 76%, 60%, and 89% of those at 20 meg. Aluminum and iron at 8 meq were released to around 40% of those at 20 meg in both KP10 and KC5P10.

The ratios of the release concentrations for an investigation of correlation of lead and other elements in the SWF are shown in Figure 2(a). The ratio of Ca/Pb in the KP10 leachate gradually decreased in the first 8 hrs of leaching but, subsequently, was relatively invariable. The ratio of Si/Pb and K/Pb was observed to continually decrease through the leaching process. The ratio of Mg/Pb, Al/Pb, and Fe/Pb increased sharply in the first 6 hrs but the Mg/Pb ratio was essentially constant thereafter while Al/Pb and Fe/Pb ratios slightly increased for the remainder of the leaching process.



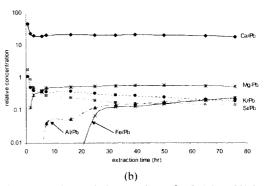


Figure 2. The relative ratio of Ca/Pb, Si/Pb, Al/Pb, Fe/Pb, Mg/Pb, and K/Pb by the PSLP in accordance with extraction time for (a) KP10 (b) KC5P10.

The ratios for KC5P10 showed similar trends albeit at slightly higher values than in KP10. The ratio of Al/Pb and Fe/Pb began to increase at a substantially later time than in KP10 as Al and Fe were not released from this SWF in the early stages of the leach process. As seen from the results of linear regression analysis shown in Table 3, the correlation of Ca-Pb and Mg-Pb was very high (above R²=0.96) for both KP10 and KC5P10. In comparison, the correlation of Si-Pb and K-Pb was very poor. Even though the

	KP10		KC5P10			
Ca-Pb	y = 5.6544x + 503.08	R2 = 0.9652	y = 19.582x + 134.99	R2 = 0.9894		
Si-Pb	y = 0.0585x + 21.601	R2 = 0.7941	y = 0.0988x + 19.092	R2 = 0.6156		
Al-Pb	y = 0.1668x - 17.195	R2 = 0.9513	y = 0.2189x - 12.340	R2 = 0.8219		
Fe-Pb	y = 0.1756x - 26.543	R2 = 0.9168	y = 0.2053x - 13.506	R2 = 0.7156		
Mg-Pb	y = 0.2059x - 8.8672	R2 = 0.9881	y = 0.6025x - 12.037	R2 = 0.9865		
K-Pb	y = 0.0473x + 37.834	R2 = 0.3938	y = 0.2126x + 12.589	R2 = 0.8223		

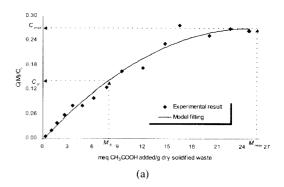
Table 3. Correlation between each element and Pb in the concentration of release by the PSLP

correlation of Al-Pb and Fe-Pb is higher for KP10 than for KC5P10, neither correlation in either case is particularly strong. These results again support the conclusion that the leaching of lead is closely associated with that of calcium and magnesium.

Leaching Model

A mathematical model to predict the amount of toxic ions leached from a solidified cement matrix by the toxicity characteristic leaching procedure(TCLP) and the American Nuclear Society's ANS 16.1 test (a dynamic leaching test lasting 90 days, performed on monolithic cylindrical specimens) was proposed by Vipulanandan. He found that a plot of C(t) versus t follows a linear relationship, where t = leaching time and C(t) = contaminant concentration.

In alkaline conditions, Portlandite and C-S-H (and sometimes CaCO₃), which are major hydrated components constituting the acid neutralizing capacity (ANC), dominate the solution chemistry of cement-water system. In the PSLP, the leaching of Pb from Pb-doped SWF is proposed to be dependent on the concentration of Pb initially present in the SWF as well as the meq CH3COOH added/g dry solidified waste during the PSLP. As shown in Figure 3, a maximum release of lead from Pb-doped SWF is found at the point at which a maximum amount of meq CH₃COOH is added, and a plot of C(M) and M reveals that there is a quadratic relationship rather than a linear relationship between the two quantities, where C(M) = the leaching concentration, mg/L, M = meq CH₃COOH added/g dry solidified waste. An appropriate mathematical equation takes the form as:



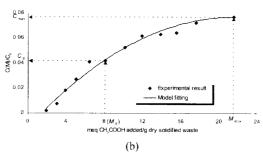


Figure 3. Leaching model of lead for (a) KP10 and (b) KC5P10.

$$\frac{C(M)}{C_o} = \alpha M^2 + \beta M + \gamma \tag{1}$$

where C_o = the initial concentration, mg/L, α , β and γ = leaching parameters independent of

various SWF. As
$$\frac{d(C(M)/C_o)}{dM} = 2\alpha M + \beta = 0$$
, M_{max} (= $-\beta/2\alpha$) is the maximum meq CH₃COOH added per g dry solidified waste for the maximum release and C_{max} at $M = -\beta/2\alpha$

Table 4 shows M_{max} , C_{max} , α , β and γ for Eq. (1). In addition, the table presents values for C_N which represents the C(M)/Co value where 8 meq CH₃COOH added/g dry solidified waste

is the maximum release of contaminant.

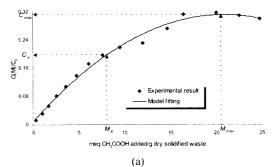
Table	4.	Summary	of	M_{max} ,	C_{max} ,	M8,	C(8),	and
		all parame	eters	for 1	Eq. (1))		

	KP10	KC5P10
The release of lead		
α	-1.67	-0.76
eta	86.03	32.35
γ	-14.10	-43.78
Coefficient of correlation	0.99	0.99
C_{x}	0.135	0.041
M_{max}	25.7	21.4
C_{max}	0.270	0.077
The release of calcium		
α	-14.54	-18.83
β	594.62	716.13
γ	158.73	-987.38
Coefficient of correlation	1.00	1.00
C_{8}	0.204	0.172
M_{max}	20.4	19.0
C_{max}	0.305	0.287

(*M₈*). In normal Portland cement, approximately 30% of the solid by weight is composed of portlandite (Ca(OH)₂), which represents the main alkaline hydrated product and corresponds to an ANC of 8 meq/g of dry. Cheng¹⁵⁾ showed that the cement-based waste form totally requires 16 meq CH₃COOH per gram of dry solid to reduce the pH from 12.4 to 9.0 and 24.0 meg/g to 5.0.

In the PSLP, the measured ANC of about 24 meq/g dry solidified waste at pH 5.0, is consistent with the experimental result of Cheng. The prediction made using Eq. (1) is in good agreement with the experimental data as shown in Table 4. At the M_{max} , the C_{max} of lead for KP10 and KC5P10 is 27.0 % and 7.7 % respectively as shown in Figure 3.

The C₈ is almost a half of the C_{max} value for KP10 and KC5P10. Eq. (1) could be used to predict the potential of leachability of lead from a cement-based SWF. It is suggested that the C₈, corresponding to an ANC of portlandite, could be simply used for leachability comparison of different SWFs. The leaching model for calcium release is shown in Figure 4. The M_{max} of calcium is lower than that of lead around 20 meq/g. The C_{max} of calcium approaches about 30%. The prediction of calcium made using Eq. (1) also makes a good agreement with the experimental data as shown in Figure 4. The C₈



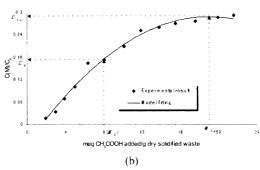


Figure 4. Leaching model of calcium for (a) KP10 and (b) KC5P10.

was approximately 60 % of the C_{max} in the leaching of calcium.

CONCLUSION

The PSLP is carried out for overcoming the difficulties associated with the pH dependence in solubility of SWF components with performance of the extraction at a fixed pH. A rate of dissolution of calcite-additional SWF (KC5P10) some 4-5 times slower than lead contaminated materials (KP10) is apparent. The release of calcium in KC5P10 was slightly lower than that in KP10, and that of lead in KC5P10 was dramatically lower than that in KP10. The difference in remarkedly less reduction in weight of KC5P10 is also indicative of the markedly higher resistance of KC5P10 to acid attack compared to that of KP10.

In a mathematical leaching model of the PSLP, the leaching of Pb from Pb-doped SWF is proposed to be dependent on the amount of initial Pb concentration as well as the meq

CH₃COOH added/g dry solidified waste during the PSLP. A maximum release of lead from Pb-doped SWF is found at the point at which a maximum amount of meq CH₃COOH is added, and a plot of the leaching concentration (mg/L) and meq CH₃COOH added/g dry solidified waste reveals that there is a quadratic relationship rather than a linear relationship between the two quantities.

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