Comparisons of operating envelopes for contaminated soil stabilised/solidified with different cementitious binders

Reginald B. Kogbara[1,2]*, Abir Al-Tabbaa[1], Julia A. Stegemann[3]

[1]Department of Engineering, University of Cambridge, Trumpington Street, Cambridge CB2 1PZ, UK.

[2]Present Address: Mechanical Engineering Program, Texas A&M University at Qatar, P O Box 23874, Education City, Doha, Qatar.

[3]Department of Civil, Environmental and Geomatic Engineering, Chadwick Building, University College London, London WC1E 6BT, UK.

*Corresponding author email: regkogbara@cantab.net; Tel: +974 4423 0289.

Abstract

This work initiated the development of operating envelopes for stabilised/solidified contaminated soils. The operating envelopes define the range of operating variables for acceptable performance of the treated soils. The study employed a soil spiked with 3000 mg/kg each of Cd, Cu, Pb, Ni and Zn, and 10,000 mg/kg of diesel. The binders used for treatment involved Portland cement (CEMI), pulverised fuel ash (PFA), ground granulated blast furnace slag (GGBS) and hydrated lime (hlime). The specific binder formulations were CEMI, CEMI:PFA=1:4, CEMI:GGBS=1:9 and hlime:GGBS=1:4. The water contents employed ranged from 13% to 21% (dry weight), while binder dosages ranged from 5% to 20% (w/w). We monitored the stabilised/solidified soils for up to 84 days using different performance tests. The tests include unconfined compressive strength (UCS), hydraulic conductivity, acid neutralisation capacity (ANC) and pH-dependent leachability of contaminants. The water content range resulted in adequate workability of the mixes but had no significant effect on leachability of contaminants. We produced design charts, representing operating envelopes, from the results generated. The charts establish relationships between water content, binder dosage and UCS; and binder dosage, leachant pH and leachability of contaminants. The work also highlights the strengths and weaknesses of the different binder formulations.

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Cambridge CB2 1PZ, UK.

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P O Box 23874, Education City, Doha, Qatar.

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Keywords: binder dosage; leaching; metals; stabilization/solidification; water content.

1. **Introduction**

Stabilisation/solidification (S/S) is an established remediation technology that employs cementitious binders for treatment of contaminated soils and other hazardous wastes. The technology involves chemical fixation and physical encapsulation of contaminants in cementitious matrices. S/S is primarily used for the immobilisation of metals and, to a lesser extent for organic contaminants as organics interfere with cement hydration and structural formation (LaGrega et al. 2001; Spence and Shi 2005). S/S treatment design usually satisfies certain criteria, depending on the end use of the treated material. Contaminant leachability in the granular leaching test is the most important performance criteria. This is because the test simulates contaminant migration from the stabilised/solidified soil in the event of its eventual breakdown. Two other key performance parameters commonly used in assessing S/S treatment efficiency are unconfined compressive strength (UCS) and hydraulic conductivity. The UCS is an indication of the ability of a monolithic S/S material to resist mechanical stresses. It relates to the progress of hydration reactions in an S/S product, as well as the durability of a monolithic S/S material. The hydraulic conductivity determines the potential for the transport of leachate bearing contaminants to move through the S/S treated material into underlying strata and eventually into groundwater (Bone et al. 2004; Perera et al. 2005).

Several variables govern the performance of stabilised/solidified contaminated soils. These include soil type and properties, contaminant type, speciation and concentration, curing environment, binder type and dosage, etc. The multiplicity of variables involved complicates the optimisation of S/S treatment process design. In chemical processing, a range of operating policies is determined during the design stage, which ensures plant performance meets certain targets. An operating envelope is the union of such operating policies. The aim of operating envelopes is to understand how variability in any of the operating variables affects the overall process (Samsatli et al. 2001). Similarly, since S/S treatment processes involves significant variability, it is important to develop operating envelopes for stabilised/solidified contaminated soils. Such operating envelopes define the limits of operating variables that result in acceptable performance. This would enhance the applicability of S/S technology for treatment of a wide range of contaminated soils. The observation that specific waste types exhibit systematic properties that allow discernment of distinct trends for different waste types provides the impetus for this work. For instance, leaching trends in widely different classes of waste materials demonstrated similar characteristics (van der Sloot et al. 1996). The leachability of particular contaminants were controlled by a limited number of parameters, namely, pH, redox potential and complexation (van der Sloot et al. 1996).

The water and binder proportions added to the contaminated soil during S/S treatment are the primary operating variables (Stegemann and Zhou 2008; Kogbara et al. 2011). However, acidic influences in the environment reduce the initial highly alkaline pH of the stabilised/solidified soil over time. Such acidic influences include rainwater (pH around 5.6), landfill leachate, acid sulphate soils, etc. Thus, the pH of the stabilised/solidified soil is reduced to levels that favour metal solubility and hence, leaching of metals. Therefore, the environment of a stabilised/solidified contaminated soil determines the leachability of contaminants. Consequently, this work identifies the pH of the leaching fluid (leachant) as a (secondary) operating variable. This is because the leachant simulates acidic water conditions in the environment.

In the light of the above, this work attempted to develop operating envelopes for treatment of a mixed contaminated soil. It is thought that operating envelopes developed for a given soil type will be applicable to other soils with similar characteristics. Hence, different cementitious binders incorporating generic S/S binder materials were utilised in this study. Such materials include Portland cement (CEMI), pulverised fuel ash (PFA), ground granulated blast furnace slag (GGBS) and hydrated lime (hlime). The different binder materials bring about certain advantages to blended cements. For example, PFA grouts lead to reduced hydraulic conductivity, increasing compressive strength and durability (UKQAA 2006). GGBS provides enhanced durability, high resistance to chloride penetration and resistance to sulphate attack as well as improved sustainability (Higgins 2005). While lime-based S/S processes are able to accommodate large quantities of organics as well as common inorganic sludges (Conner and Hoeffner 1998).

There is a paucity of literature on studies deploying different binder blends for S/S treatment of a mixed contaminated soil. Thus, information on granular leachability of soils treated by different binders systems under different pH conditions is rare in the literature. Furthermore, very few studies too have compared the strength and hydraulic conductivity of contaminated soils treated by blends of the afore-mentioned generic binder materials. These are necessary for informed decisions on recycling options (e.g. as sub-base course in road pavement) for contaminated soil treated by one binder or another. Therefore, this work seeks to fill in such gaps in the literature. The aim of the study was to compare the effectiveness, or the strengths and weaknesses, of different binder blends for S/S treatment of a mixed contaminated soil. Specifically, the study evaluates the range of binder dosage and water content that leads to acceptable strength, hydraulic conductivity and leaching performance of different binder systems.

2. **Materials and methods**

2.1 *Contaminated soil*

It was the aim of the study to utilise a real site soil contaminated with metals and organics. This is because many contaminated soils are characterised by the concomitant presence of metals and organics. However, the site soil found, contained very low levels of metals and total petroleum hydrocarbons (TPH). The source site of the soil was a service station in Birmingham, UK. The soil was clayey silty sandy gravel comprising 65% gravel, 29% sand, 2.8% silt and 3.2% clay. It had a natural water content of ~ 12% and very low (0.22%, dry weight) organic carbon content. The pH of the spiked contaminated soil (9.83) was very alkaline. Such high pH in soil is probably caused by the association between sodium and carbonate species in the soil (Brautigan 2010). It is important to point out that such a high soil pH would influence the leachability of contaminants. However, since one third of the world's soils are alkaline (Guerinot 2007), it is necessary to understand the leaching behaviour of such soils.

In the light of the above, we spiked the soil with five metals (Cd, Cu, Pb, Ni and Zn) and diesel. We employed small batches of ~3 kg for the spiking. The aim was to increase the contaminant levels to relatively high values for monitoring during the course of the study. The concentration of each metal spiked was 3000 mg/kg, while the concentration of diesel used was 10,000 mg/kg. The metals used were reagent grade chemical compounds, all supplied by Fisher Scientific. The contaminant levels were chosen to represent the upper range of concentrations found in contaminated soils (Kabata-Pendias and Mukherjee 2007).

Table 1 shows the total concentration of contaminants in the spiked soil. We understand that in reality, a contaminated site soil may not contain many metals at such high concentrations as used here. Most contaminated sites may have two to three of the metals at similar concentrations and others at much lower concentrations. However, we employed high concentrations of the metals to represent some sort of 'worst-case scenario' of elevated metal concentrations in contaminated soil.

Table 1. Amount of contaminants in the spiked contaminated soil

Contaminant	Compound	Contaminant concentration (mg/kg)
Cadmium	$Cd(NO_3)_2.4H_2O$	3500 ± 150*
Copper	$CuSO_4.5H_2O$	3200 ± 230
Lead	$PbNO_3$	3700 ± 210
Nickel	$Ni(NO_3)_2.6H_2O$	3600 ± 150
Zinc	$ZnCl_2$	4200 ± 290
TPH	Diesel	6300 ± 1500

*Results represent mean ± standard deviation of three replicates

2.2 *Cementitious binders*

The binders used for S/S treatment of the contaminated soil involved mixtures of CEMI, PFA, GGBS and hlime. Lafarge, UK supplied the CEMI, while UK Quality Ash Association (UKQAA) supplied the PFA. UK Cementitious Slag Makers Association (UKCSMA) and Tarmac Buxton Lime and Cement (UK), supplied the GGBS and hlime, respectively. Table 2 shows the physico-chemical properties of the binder materials. The specific binder formulations used were: CEMI, CEMI:PFA=1:4, CEMI:GGBS=1:9 and hlime:GGBS=1:4. CEMI is the most commonly used binder in S/S treatment. Hence, it served as a control for assessment of the relative performance of the other binders. The binder mix proportions were the same as those used in parallel studies on S/S of metal filter cakes (Stegemann and Zhou 2008). The screening and optimisation stage in the parallel studies showed good leachability results for the blends. The mix proportions were also consistent with relevant literature (Wild et al. 1996; Arora and Aydilek 2005; Oner and Akyuz 2007). Further, the choice of higher replacement levels of PFA and GGBS would ensure maximum re-use of industrial by-products for economic considerations.

Table 2. Physico-chemical properties of binder materials (from Materials data sheets)

Constituent / Parameter	CEMI	PFA	GGBS	hlime
Bulk density (kg/m^3)	1,300 – 1,450	1,100 – 1,700	1,200	470 – 520
Specific gravity	3.15	1.80 – 2.40	2.90	2.30 – 2.40
Specific surface area (m^2/kg)	400	3,430	350	1,529
Granulometry/mean particle size (μm)	-	< 600	5 – 30	63 – 125
Colour	grey	grey	off-white	white
pH*	12.80	10.22	11.79	12.85
CaO (%)	63.6	1 – 5	40	-
$Ca(OH)_2$ (%)	-	-	-	96.9
SiO_2 (%)	13.9	45 – 51	35	-
MgO (%)	0.6	1 – 4	8	-
$Mg(OH)_2$ (%)	-	-	-	0.5
Al_2O_3 (%)	10.2	27 – 32	13	-
$CaCO_3$ (%)	-	-	-	1.4
$CaSO_4$ (%)	-	-	-	0.03
Fe_2O_3 (%)	2.7	7 – 11	-	-
K_2O (%)	0.9	1 – 5	-	-
TiO_2 (%)	0.1	0.8 – 1.1	-	-
SO_3 (%)	6.9	0.3 – 1.3	-	-
Cl (%)	0.02	0.05 – 0.15	-	-
L.O.I	2.15	NS	-	-

*The pH was determined in a 1:10 binder material: deionised water suspension, L.O.I: loss on ignition, NS: not specified

2.3 *Preparation of stabilised/solidified soil samples*

The spiked contaminated soil was prepared by first mixing the soil with diesel thoroughly. Addition of solutions of the metallic compounds made with de-ionised water followed thereafter. Further mixing continued until the mix was homogenous. The spiked contaminated soil was then stored in a sealed container for about 2 hours. Analyses of leachability of contaminants from soil samples followed thereafter. We then mixed binder materials together in the appropriate proportions, and de-ionised water added to form a paste. The binders were then applied to the contaminated soil in 5%, 10% and 20% dosages (dry weight), and mixed thoroughly.

Stabilised/solidified products were prepared using the density-moisture content relationship determined in the standard (2.5 kg rammer) Proctor compaction test (BSI BS1377: Part 4 1990). A few trials showed the range of water contents that would be amenable to compaction. Thereafter, the contaminated soil-binder mixes were compacted at 4 – 5 different water contents ranging from 13% – 21% (dry weight). The determination of water content did not include the liquid content due to diesel. Hence, the optimum water/solid (w/s) ratio and the optimum moisture content (OMC) are synonymous in this work. The compacted mix was broken up after each stage and cast into cylindrical moulds, 50 mm diameter and 100 mm high. The samples were stored at 95% relative humidity and 20°C.

2.4 *Testing methodologies*

Assessment of S/S treatment efficiency involved UCS, hydraulic conductivity, and acid neutralisation capacity (ANC) and pH-dependent granular leachability. The tests followed the procedures described in relevant literature (Stegemann and Côté 1991; ASTM D1633-00 2000; ASTM D5084-03 2003). The ANC test was determined using 0, 1 and 2 meq/g HNO_3 additions. The choice of only three acid additions was for consistency with parallel studies on S/S of metal filter cakes (Stegemann and Zhou 2008). The ANC test on the untreated contaminated soil also included two more acid and base (NaOH) additions. This was meant to cover the full pH range and help assess metal immobilisation in a given pH zone.

Curing of the stabilised/solidified soils lasted 7, 28, 49 or 84 days before testing. Testing started with low (5%) binder dosage mixes with assessment of contaminant leachability until most granular leaching criteria were satisfied. Hence, the highest (20%) binder dosage employed had fewer testing. Details of the testing methods are contained in previous related works (Kogbara and Al-Tabbaa 2011; Kogbara et al. 2011; Kogbara et al. 2012; Kogbara et al. 2013).

2.5 *Statistical analysis and contour map plotting*

Normality of data was determined using the Kolmogorov-Smirnov test. One and two-way ANOVA were then used to test for statistically significant differences in performance parameters due to differences in operating variables.

We plotted contour maps from the experimental results using Origin 8.6 software (OriginLab Corporation, Northampton, USA). The contour maps are in the form of design charts and do represent operating envelopes for selected performance parameters. The contour plots for UCS were generated from matrices based on moulding water content, binder dosage and 28-day UCS data, for a given binder system. While contour plots for leachability / leachate pH were generated using binder dosage, leachant pH and contaminant leachability / leachate pH data from different water contents and curing ages. The contour matrices were determined using the software's gridding method (mainly kriging correlation), which best fits the data points.

3. **Results and discussions**

This section compares the effectiveness and the operating envelopes of the four different binders employed. The initial testing employed 7-day and 28-day old samples of mixes treated with 5% and 10% dosage of the different binders. These indicated the best mechanical and leaching performance around the OMC. Consequently, testing at 49 and 84 days employed only OMC mixes. Moreover, further testing continued with only the OMC mix for samples with 20% binder dosage. Where applicable, we compared the mechanical performance in terms of w/s ratio and binder dosage on 3-D plots. In such plots, one can read the performance parameters by following the vertical lines to the base of their respective axes.

3.1 Compaction behaviour

Figure 1 shows the compaction characteristics of the different contaminated soil-grout mixes. Generally, water contents ranging from 13% to 21% (dry weight) resulted in adequate workability of the soil-binder systems. The compaction characteristics of the four binder systems were similar. The OMC of most of the soil-binder mixes varied within a 2% range (i.e. water contents of about 15% to 17%) depending on the binder dosage. The maximum dry density ranged from 1.73 Mg/m^3 to 1.87 Mg/m^3, depending on the binder dosage. The results demonstrate that soil contamination and binder addition up to 20% dosage does not alter the compaction behaviour significantly.

3.2 UCS comparison

3.2.1. *Strength behaviour of the binders*

Figure 2a compares the different mixes in terms of their 28-day UCS on a 3-D plot. Figure 2b shows the strength development of OMC mixes over time. Generally, the 28-day UCS behaviour of CEMI-PFA, CEMI-GGBS and hlime-GGBS mixes was similar. While that of CEMI-treated soil was markedly different as its performance was superior. CEMI-PFA mixes generally showed the lowest strengths as the strength of CEMI-PFA binder largely depends on the cement content. Moreover, the presence of the hydrocarbon contaminant generally leads to decreased strength in S/S treated soils (Trussell and Spence 1994). The strength gain over time of all binders except

CEMI was similar (p = 0.16) in 5% dosage mixes. However, they were significantly different (p = 0.04) in 10% dosage mixes (Figure 2b).

CEMI-GGBS and hlime-GGBS mixes showed appreciable strength gain over time, especially with 10% binder dosage. The UCS of GGBS-based binders increases over time and could even be higher than that of CEMI at the same binder dosage over time, depending on the mix formulation (Higgins 2005; Oner and Akyuz 2007). This is because the pozzolanic reaction involved in GGBS-based binders is slow and the formation of calcium hydroxide requires time (Oner and Akyuz 2007). However, the results in Figure 2b differed from the above position, especially for lime-GGBS mixes for which the mix formulation for optimum strength (Higgins 2005) was used. CEMI-treated contaminated soil showed much higher strengths than soils treated with the GGBS-based binders throughout the 84-day study period (Figure 2b). This difference in strength behaviour is probably due to the interactive effects of contaminants, especially in the presence of large amounts of hydrocabons. The results corroborate Jegandan (2010) on superior performance of CEMI in soil with high organic content.

Table 3 shows the 49-day UCS before and after immersion in water of the OMC mixes of the four binders. The GGBS-based binders were least affected by immersion in water. There was just 3% to 4% reduction in UCS of hlime-GGBS mixes. Moreover, the UCS of 5% CEMI-GGBS dosage mixes increased after immersion in water. The transformation of the amorphous aluminosilicate network structure of the GGBS-based binders into a crystalline one may account for this behaviour. Some of the crystals have a faujasite structure, which appears to act as reinforcement in the binder matrix (Shi and Fernandez-Jimenez 2006). Thus, the results support previous reports that alkali-activated cements have a much better resistance to corrosive environments (e.g. water, and acidic and sulphate solutions) than CEMI (Shi and Fernandez-Jimenez 2006).

Table 3. 49-day UCS of the different soil-binder mixes before and after immersion in water

Binder	Binder dosage (%)	UCS before immersion (kPa)	UCS after immersion (kPa)	UCS reduction (%)
CEMI	5	330	280	15
	10	1,990	990	50
CEMI:PFA = 1:4	5	n.d.	n.d.	n.d.
	10	110	66	40
hlime:GGBS = 1:4	5	142	137	4
	10	412	400	3
CEMI:GGBS = 1:9	5	134	184	-27 (increase)
	10	760	647	15

n.d.: not determined as samples crumbled due to insufficient hydration

3.2.2. *Design chart representing operating envelopes for UCS*

Figure 3 shows design charts based on contour plots illustrating operating envelopes for 28-day UCS of the different mixes. Each graph consists of the contour plots for two binders, grouped according to the 'base materials' – CEMI and GGBS. This facilitates direct comparisons between the binders, while ensuring that the graphs are not too congested. Solid, dash or dotted black and gray lines distinguish the UCS levels of the different binders on the plots. There are also constructions in the graphs to show how the charts work.

One common performance threshold for defining the operating envelope(s) for 28-day UCS is the 1 MPa criterion for landfill disposal (Environment Agency 2006). Another performance threshold is the Environment Canada 440 kPa criterion for controlled utilisation (Stegemann and Côté 1996). The construction with solid black lines in Figure 3a shows how to use the chart. Assuming we want an S/S product with a UCS of 1 MPa from a similar type of contaminated soil. The minimum CEMI dosage that would be required is ~ 7.5%. The w/s ratio would range from 0.17 to 0.19 (Figure 3a). If a low w/s ratio is used, the CEMI dosage required will significantly increase. Figure 3 indicates that greater than 20% dosage is required for the other three binders to achieve the aforementioned threshold. However, all soil-binder systems satisfied the 440-kPa UCS criterion with ≥ 10% dosage. Nevertheless, this depended on the choice of water contents. Generally, water contents at the OMC and 2% – 4% wet of OMC was required to satisfy the criterion. For instance, 10% CEMI-PFA dosage can achieve the criterion with a w/s of ~ 0.2 (i.e. OMC+4) (Figure 3a). Similarly, 10% CEMI-GGBS dosage can achieve it with a w/s of ~ 0.17 (i.e. OMC) (Figure 3b).

The above implies that except CEMI, none of the other binders would meet strength requirements for sub-base course in road pavements with up to 20% dosage. One recommended minimum UCS requirement for sub-base course in rigid pavements is 200 psi (i.e. 1.38 MPa) at 7 days for CEMI and 28 days for the other binder materials (UFC 2004). It is common knowledge that UCS increases with binder dosage. Hence, higher dosages of the different binders can make the treated soils recyclable for construction applications.

Furthermore, the relatively low water contents used here was adequate for workability of the soil-binder mixtures under laboratory conditions. However, it is likely that they may not work in the field. Moreover, field scenario would involve weathered contamination as opposed to fresh contamination used here. Soils with weathered petroleum hydrocarbons are more likely to have higher UCS than soils with fresh hydrocarbons (Al-Sanad and Ismael 1997). Hence, these results provide a conservative estimate of the UCS values under usual field conditions.

3.3 **Hydraulic conductivity**

Figure 4 shows the hydraulic conductivity of the different mixes at 28 days. The figure also shows the hydraulic conductivity of OMC mixes at 28 and 84 days. The 5% dosage CEMI-PFA and CEMI-GGBS OMC mixes crumbled over time due to insufficient hydration. Hence, the hydraulic conductivity of the mixes was not tested. Thus, Figure 4 does not show data on the said mixes. The hydraulic conductivity of the three CEMI-containing binders was generally similar. However, the hydraulic conductivity of hlime-GGBS mixes was markedly different from those of the CEMI-containing binders. It was generally higher and increased with increasing binder dosage (Figure 4a).

The increase in hydraulic conductivity with binder dosage in hlime-GGBS mixes may be associated with the presence of lime. However, increasing steel slag content, which is similar to GGBS, was reported to increase the permeability of lateritic soil and it was linked with the free lime content of the slag (Akinwumi et al. 2012). The reaction of lime with soil particles, especially clays, leads to agglomeration and flocculation of clay particles with a consequent reduction in the plasticity. The resultant soil structure becomes an open matrix. This in turn leads to increase in hydraulic conductivity. However, the above differs from a previous observation that lime addition reduced the hydraulic conductivity of poorly graded river sand but increased that of sandy silty clay (El-Rawi and Awad 1981). The exact reason for this behaviour is unclear as there is little information on the effect of lime-GGBS addition on sandy and gravelly soils. Thus, more work is required in this area to elucidate the effect of the binder on hydraulic conductivity in different soil types.

Hydraulic conductivity values were generally at the minimum around the OMC. As expected, increase in binder dosage generally led to decrease in hydraulic conductivity except for hlime-GGBS mixes. The hydraulic conductivity of all mixes except CEMI-PFA mixes slightly increased above their 28-day values at 84 days (Figure 4b). A similar observation has been reported (Al-Tabbaa and Evans 2000). The different behaviour of the 10% CEMI-PFA mix was probably due to reduction in interconnectivity of the pores by products of on-going pozzolanic reactions (Kogbara et al. 2013).

There is no design chart for hydraulic conductivity due to paucity of data points. Nevertheless, the most common performance threshold for hydraulic conductivity is the 10^{-9} m/s limit for in-ground treatment. Another performance threshold is the 10^{-8} m/s Environment Canada proposed limit for landfill disposal scenarios (Stegemann and Côté 1996). None of the mixes satisfied the 10^{-9} m/s criterion with 20% binder dosage. Thus, higher binder dosages would be required to meet that criterion. The binders satisfied the 10^{-8} m/s criterion for landfill disposal with up to 20% dosage at certain water contents. However, hlime-GGBS mixes were an exception to this (Figure 4).

3.4 Granular leachability of contaminants

3.4.1 *ANC and leaching trends*

This section focuses on comparisons between the different soil-binder systems. Discussion on the leaching behaviour and chemistry of the contaminants is phenomenological. This is because they were essentially the same as described in previous related works (Kogbara and Al-Tabbaa 2011; Kogbara et al. 2011) for all soil-binder systems. Table 4 compares the ANC or buffering capacity of the four binders. The buffering capacity is the ability to neutralise acidic influences and maintain chemical durability. It was in the order, hlime-GGBS > CEMI > CEMI-GGBS > CEMI-PFA. The order of the buffering capacities is due to the natural pH of the binder materials (see Table 2).

Table 4. ANC of the different binders and leachant pH at 0, 1 and 2 meq/g HNO_3 addition

Binder/Leachant	pH			Difference in pH units between 0 and 2 meq/g
	0 meq/g	1 meq/g	2 meq/g	
CEMI	12.80	12.66	12.53	0.27
CEMI:PFA = 1:4	12.78	12.40	10.95	1.83
hlime:GGBS = 1:4	12.94	12.71	12.59	0.35
CEMI:GGBS = 1:9	12.61	11.51	11.02	1.59
Leachant	7.20	1.20	0.85	not applicable

Figures 5 – 7 show the 28-day leachability of the six contaminants, in OMC mixes of different soil-binder mixes. The figures also show the initial leachability of the untreated spiked contaminated soil at different pH values. Each stabilised/solidified product has three points on the graphs. From right to left, these represent the leachate pH values at 0, 1 and 2 meq/g HNO_3

additions. The smooth curves in the metal leachability graphs are the theoretical solubility profiles of the metal hydroxides (Stegemann 2005). The solubility profiles come from data in the MINTEQ database – a chemical equilibrium model for predicting metal speciation and solubility. The range of water contents considered had no significant effect on leachability of contaminants. Hence, Figures 5 – 7 shows the contaminant leachability in only OMC mixes of different soil-binder systems.

The leaching behaviour of all five metals was largely pH-controlled in all binders. Metal leachability depended on the leachate pH attained by a given mix. The leachate pH in turn depended on the binder dosage used. This corroborates van der Sloot et al. (1996) on similarity of leaching trends in different classes of materials. Generally, metal leachability in all binders increased with decreasing pH and decreased with increasing binder dosage. However, there were some exceptions. These occurred when the pH fell in the zone for increased leachability of some metals above their minimum solubility values. This is obvious for Cu in CEMI S/S soil (Figure 5b). The same goes for Pb and Zn in CEMI and hlime-GGBS S/S soil (Figures 6a and 7a). This corroborates Akhter et al. (1990) on problems encountered with CEMI for Pb stabilisation. As opposed to CEMI, which presents problems with Pb stabilisation, CEMI-PFA tends to widen the immobilisation pH range. This is due to its fly ash content, which forms pozzolanic products that either adsorb Pb on to fresh surfaces or incorporate Pb by means of chemical inclusions. Additional pozzolanic product formation with increasing curing age further increases the amount of non-extractable Pb (Dermatas et al. 2006). Generally, CEMI and hlime-GGBS binders showed better immobilisation capacities for Cd, Ni and Zn. CEMI-PFA and CEMI-GGBS binders were better for Cu and Pb immobilisation.

The speciation of the metals was similar in all binder systems. The leaching behaviour of Cd, Cu, Pb and Zn generally followed the hydroxide profile of the metals in all binder systems. The leaching behaviour of Ni suggests the existence of the metal in more soluble phases other than the hydroxide. The leachabiity of the metal exceeded $Ni(OH)_2$ solubility limits in most cases (Figure 6b). It is likely that Ni existed as carbonate-complexes in the cementitious systems (Christensen et al. 1996). Compared to the untreated soil, the 20% dosage mix of all binder systems showed the potential for chemical immobilisation of the metals. In some cases, the leachability of the metals was lower in such mixes than in the untreated soil even at the same pH.

TPH leachability was similar in all binder systems. Binder dosage had no significant effect on it (Figure 7b). Schifano et al. (2005) made a similar observation for contaminated soil treated with lime. All the same, S/S treatment with the binders reduced TPH leachability. Generally, the untreated soil leached out higher TPH concentrations than S/S treated soils. The lowest TPH concentrations occurred in hlime-GGBS mixes (Figure 7b). It is likely that the presence of lime contributed to the marginally better performance of hlime-GGBS mixes. Lime has been shown to be very effective for macroencapsulation of hydrocarbon-contaminated soils (Adams 2004). Similarly, there was no observable effect of pH on TPH leachability. Besides being soluble in one another, petroleum hydrocarbons are generally characterised by insolubility under different conditions in single solvents. Hence, the pH of the leachant does not govern TPH leachability. Nevertheless, increasing acidity apparently influenced TPH leachability in the treated soils as compared to the untreated soil. Higher TPH concentrations were generally leached out at 1 and 2 meq/g acid addition than with no acid addition (Figure 7b). In many cases, the solubility of an organic contaminant depends on the pH of the environment in which it is present (Bone et al. 2004).

We did not include the leachability results in the different soil-binder systems over time due to manuscript length. These are contained in related publications (Kogbara and Al-Tabbaa 2011; Kogbara et al. 2012; Kogbara et al. 2013). The leaching behaviour observed over time is summarised as follows. The leachability of the metals in the mixes was either stable or decreased by over half an order of magnitude between 28 and 84 days of treatment. CEMI-GGBS mixes showed a unique leachability trend between 49 and 84 days. The leachate concentrations of all metals decreased between both curing ages, and this corroborated the strength increase observed during the said period (Kogbara and Al-Tabbaa 2011). There was a general increase in TPH leachability over the 28-day values at 84 days in all binder systems. However, the increase was quite significant in CEMI-PFA mixes especially under acidic influence, probably due to the hydrocarbon origin of PFA. Hence, CEMI-PFA appears to be the least suitable binder for reducing TPH leachability.

3.4.2 *Design charts for leachate pH and leachability of contaminants*

The effects of water content and curing age on contaminant leachability were not statistically significant. Hence, we combined contaminant leachability and leachate pH data from soil-binder mixes with different water contents and curing ages. We used the said data to produce the design charts here (Figures 8 – 14). The design charts compare the leachate pH and leachability of the six contaminants studied, in the four soil-binder systems. The contour plots here have two graphs for each parameter/contaminant similar to those in section 3.2.2. The binders are distinguished with the same lines used previously. Binder dosage and the leachate pH attained during leaching mainly determined contaminant leachability. The binder dosage and leachant pH controls the leachate pH. Hence, both parameters are the independent operating variables for the contaminant leachability design charts. The constructions in Figures 8b and 9b show how the charts work. One can use the leachate pH and contaminant leachability charts together.

Given that, a natural leachant with pH around 5.6 continuously gets in contact with a 9% CEMI-GGBS dosage stabilised/solidified product. Figure 8b (construction with solid gray lines) shows that its pH will decrease to around 10.2. Based on the same information, Figure 9b shows that the Cd leachability would range from 0.01 mg/kg to 10 mg/kg. Similarly, Figure 8b also shows the effect of a leachant pH of 3.8 on an 8% hlime-GGBS stabilised/solidified product. Its leachate pH drops to around 9.4 (Figure 8b, construction in dotted gray lines). The Cd leachability of the 8% hlime-GGBS stabilised/solidified product would be around 10 mg/kg (Figure 9b). The situation in the second example is similar to the effects of progressive carbonation on stabilised/solidified materials. Absorption of CO_2 causes decrease in pH of such materials to ~ 9 (Arickx et al. 2010). The aforementioned leachant pH is similar to the pH (3 – 4) of carbonic acid, which is involved in carbonation. It is noteworthy that these examples relate to leaching from granular forms of the stabilised/solidified materials. This could occur in a worst-case scenario over a long time when the stabilised/solidified soil breaks down.

There are no established performance thresholds for contaminant leachability at specific pH values. Performance thresholds for contaminant leachability only exist for leachability at zero acid addition. These are contained in a related publication (Kogbara and Al-Tabbaa 2011). The design charts are also useful for this purpose as a leachant pH ~7 corresponds to zero acid addition. For instance, an applicable performance threshold is the granular leachability waste acceptance criteria (WAC). The WAC for Cd in stable non-reactive hazardous waste in non-hazardous landfill is 1.0 mg/kg. We can deduce from Figure 9a that 5% CEMI dosage can easily satisfy the criterion. While ≥ 10% dosage of the other three binders would be required to satisfy the criterion.

Naturally, the acceptable binder dosage limit for leachability is the threshold at which leaching criteria for all metals are satisfied. Consequently, about 20% dosage is required for all binders to satisfy the most stringent leaching criteria (see Figures 9 – 14). The said criteria are the environmental quality standard (EQS) for inland surface waters and the inert waste landfill WAC. The available EQS in mg/kg are 0.04 for Cd, 72 for Pb and 0.2 for Ni. The following are the WAC for inert waste landfill. The WAC for Cd, Cu, Pb, Ni and Zn are 0.04, 2, 0.5, 0.4 and 4 mg/kg, respectively (Kogbara and Al-Tabbaa 2011). However, Pb content presents problems in binders like CEMI and hlime-GGBS. This is because low binder dosage may satisfy certain leaching criteria but higher dosage may not. This occurs where the pH attained by higher dosages of both binders corresponded to the zone for increased Pb leachability. Thus, both binders may not be very suitable for similar soils with high Pb concentrations. It is noteworthy that soils with weathered contamination are more likely to have lower contaminant leachability than freshly contaminated soils. Hence, the leaching results here may be higher estimates (perhaps the worst-case scenario) of those under typical field conditions.

Design charts for soils would depend on the properties of a given soil. All the same, the charts here can also provide likely estimates of contaminant leachability in other stabilised/solidified soils. The following examples comparing results from similar situations in other studies illustrate this. Yilmaz et al. (2003) used 10% CEMI binder in treating fine mining waste soil (27% sand, 18% clay, 55% silt). The initial concentrations of Cu (3640 mg/kg) and Pb (4380 mg/kg) were close to those here. The leachant pH used was 4.93 in toxicity characteristic leaching procedure (TCLP) tests. The 28-day leachability of Cu and Pb were 6.2 and 7.8 mg/kg, respectively. These are similar to Cu and Pb leachability around 4 and 10 mg/kg, respectively, for CEMI in Figures 10a and 11a. Secondly, Moon et al. (2010) used 20% CEMI-PFA binder to treat Zn-contaminated soil (55% sand, 33.8% silt, 10.3% clay; natural pH 8.31). The binder formulation used (CEMI:PFA=1:3) is close to that used here. The initial Zn concentration was 4973 mg/kg. The 28-day Zn leachability recorded in TCLP tests with leachant pH, 2.88±0.05, was 260 mg/kg. This is close to the Zn leachability range (~ 300 mg/kg) for CEMI-PFA in Figure 13a.

4. **Conclusions**

This work considered the initial development of operating envelopes for S/S treatment of contaminated soil with different binders. It culminated in the production of design charts that establish relationships between operating variables and strength and leaching performance. The study showed that with freshly contaminated soils containing large amounts of hydrocarbons, stabilisation with CEMI results in a much higher UCS than CEMI-PFA, CEMI-GGBS and hlime-GGBS. With up to 20% binder dosage, CEMI satisfies the strength criteria required for different management scenarios. However, depending on the extent of contamination, greater than 20% dosage of the other three binders would be required to satisfy strength criteria for most management scenarios. Thus, economic considerations on the amount of binder materials required to satisfy set criteria would influence the choice of one binder over another. Generally, water contents at the OMC and 2% – 4% wet of OMC resulted in the highest UCS values.

Stabilisation with the cement-containing binders gave similar hydraulic conductivity values. However, hlime-GGBS stabilisation led to much higher hydraulic conductivity. The hydraulic conductivity increased with increasing binder dosage. Hence, the binder may not be suitable for recycling applications requiring low hydraulic conductivity. Hydraulic conductivity values were generally at the minimum around the OMC. Greater than 20% dosages of all four binders are required to give hydraulic conductivities $< 10^{-9}$ m/s. The cement-containing binders would satisfy reuse applications requiring 10^{-8} m/s hydraulic conductivity with up to 20% dosage but hlime-GGBS mixes would not.

Water contents ranging from 13% to 21% (dry weight) had no significant effect on leachability of contaminants. About 20% dosage of all four binders is required to satisfy the most stringent leaching criteria. However, this does not apply to Pb stabilisation with CEMI and hlime-GGBS as the binders present problems with the contaminant. CEMI and hlime-GGBS binders showed better immobilisation capacities for Cd, Ni and Zn. CEMI-PFA and CEMI-GGBS binders were better for Cu and Pb immobilisation. All four binders showed similar performance for TPH immobilisation, although hlime-GGBS gave a marginally better performance.

The operating envelopes for the different parameters considered in this work largely depend on the properties of the soil used. Nevertheless, comparison with results from similar situations in other studies shows that the design charts may be applicable to other stabilised/solidified soils with similar characteristics. Especially, the charts can provide likely estimates of contaminant leachability in other stabilised/solidified soils. The data utilised for the design charts were limited, which may reduce their reliability for certain values of operating variables. Further studies may improve the reliability of the design charts. Such studies may consider data from different soil types, contaminant concentrations, ranges of operating variables, and other performance parameters.

Acknowledgments

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Figure captions

Figure 1. Compaction behaviour of different soil-binder mixes

Figure 2. UCS of different soil-binder mixes (a) at 28 days
(b) in OMC mixes at different curing ages

— CEMI, ----- CEMI-PFA, — CEMI-GGBS, ….. hlime-GGBS

Figure 3. (a) Design chart for 28-day UCS (b) example of how the chart works

Figure 4. Hydraulic conductivity of different soil-binder mixes (a) at 28 days
(b) in OMC mixes at different curing ages

Figure 5. Leachability in the treated soils at 28 days for (a) Cd and (b) Cu

Figure 6. Leachability in the treated soils at 28 days for (a) Pb and (b) Ni

Figure 7. Leachability in the treated soils at 28 days for (a) Zn and (b) TPH

— CEMI, ----- CEMI-PFA, — CEMI-GGBS, ….. hlime-GGBS

Figure 8. Leachate pH design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 9. Cd leachability design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 10. Cu leachability design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 11. Pb leachability design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 12. Ni leachability design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 13. Zn leachability design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 14. TPH leachability design chart for mixes based on (a) CEMI, and (b) GGBS

Figure 1

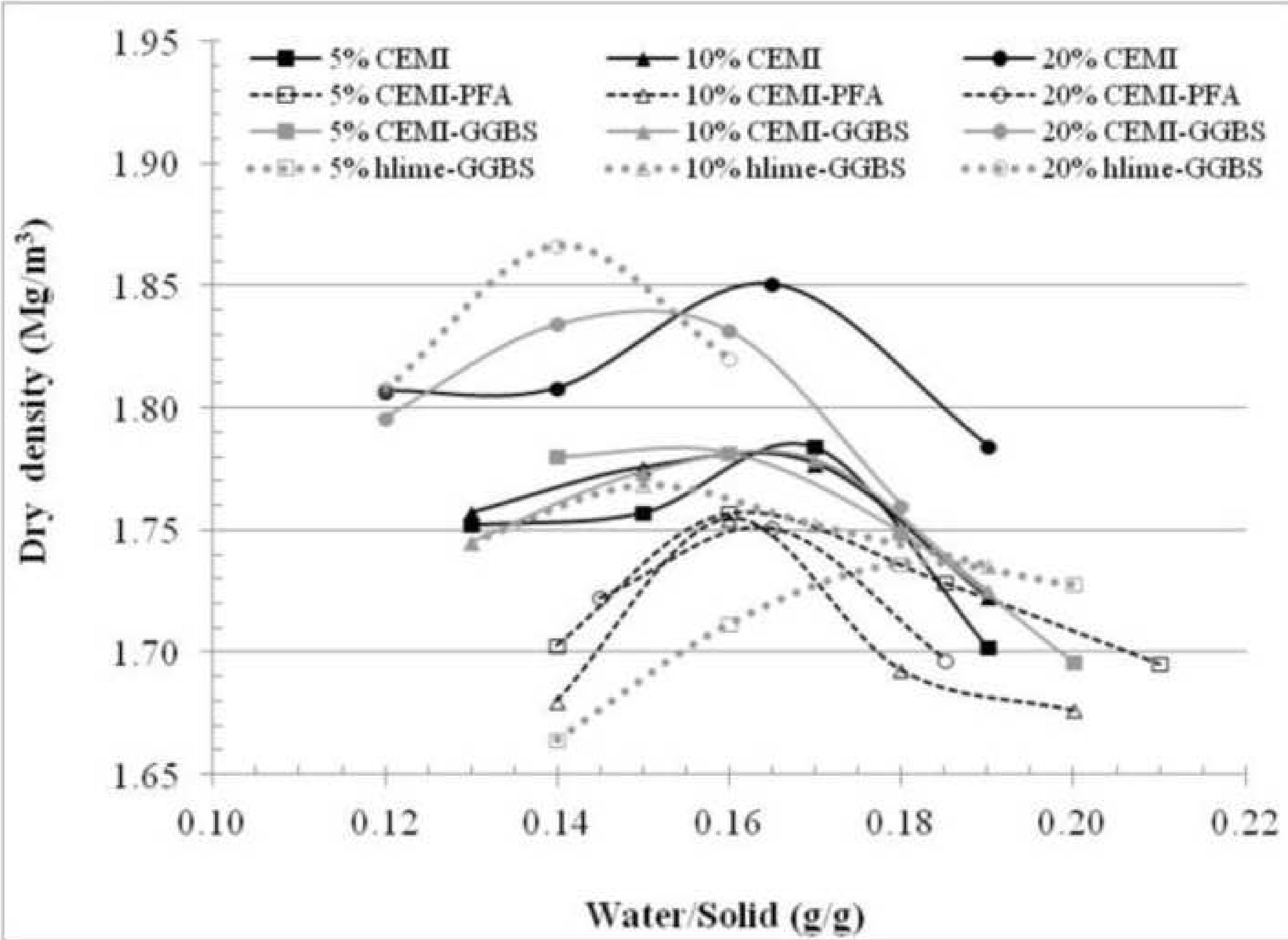
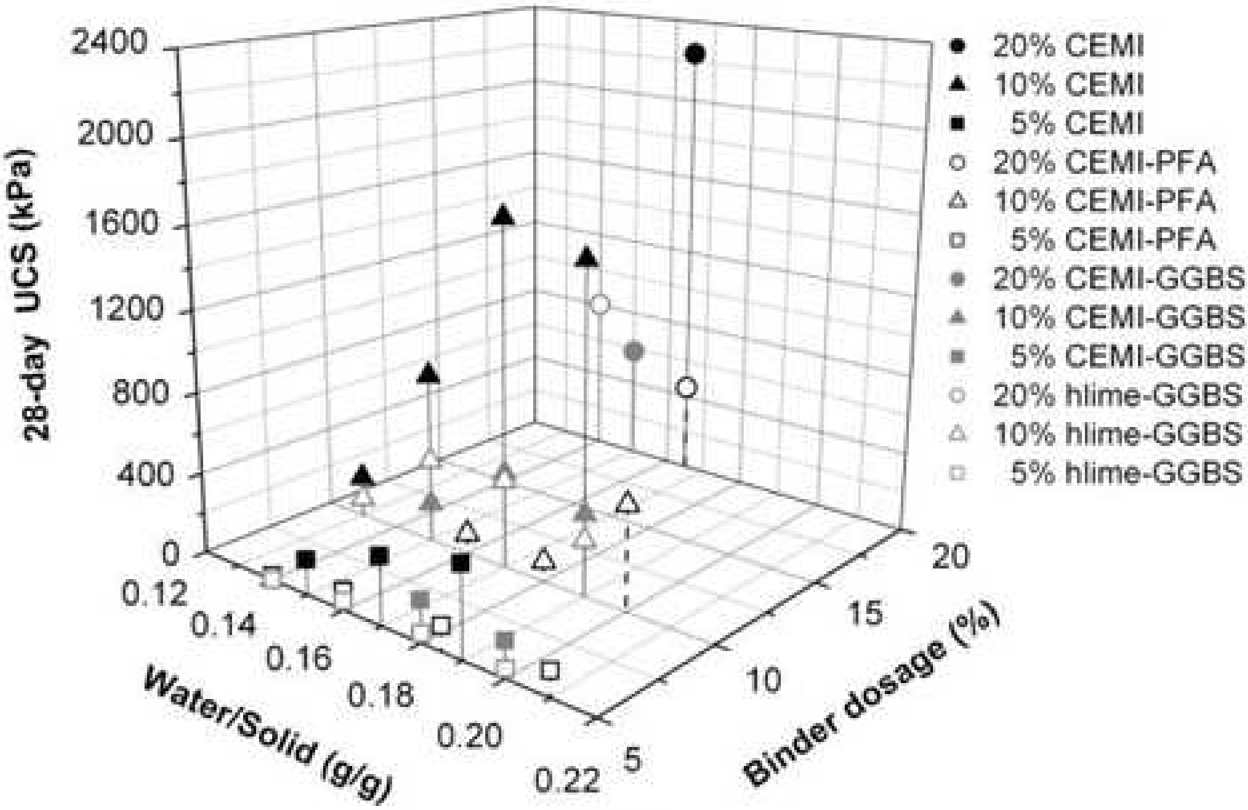
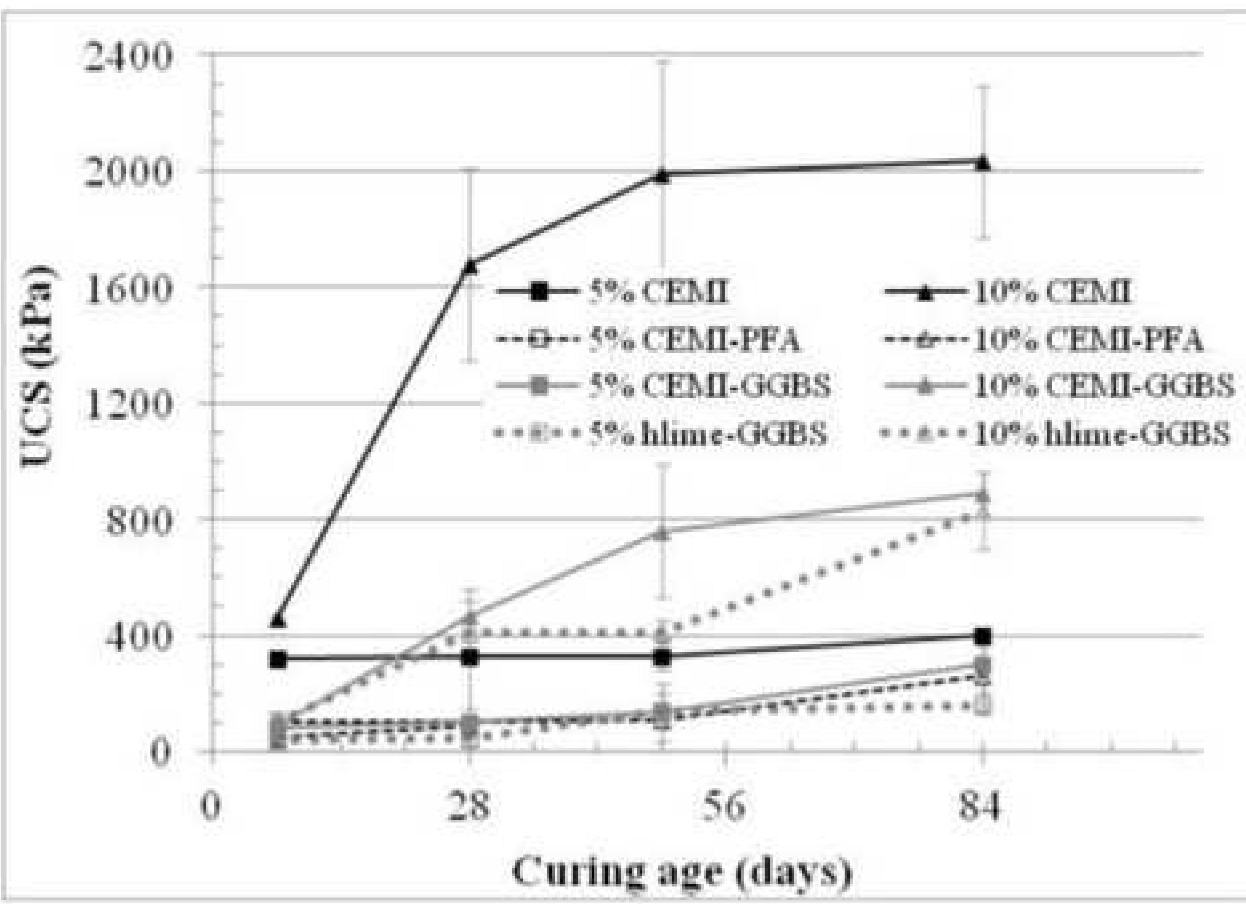

5% CEMI
10% CEMI
20% CEMI
5% CEMI-PFA
10% CEMI-PFA
20% CEMI-PFA
5% CEMI-GGBS
10% CEMI-GGBS
20% CEMI-GGBS
5% hlime-GGBS
10% hlime-GGBS
20% hlime-GGBS
Dry density (Mg/m^3)
1.95
1.90
1.85
1.80
1.75
1.70
1.65
0.10
0.12
0.14
0.16
0.18
0.20
0.22
Water/Solid (g/g)

Figure 2



28-day UCS (kPa)
2400
2000
1600
1200
800
400
0
0.12
0.14
0.16
0.18
0.20
0.22
Water/Solid (g/g)
5
10
15
20
Binder dosage (%)
20% CEMI
10% CEMI
5% CEMI
20% CEMI-PFA
10% CEMI-PFA
5% CEMI-PFA
20% CEMI-GGBS
10% CEMI-GGBS
5% CEMI-GGBS
20% hlime-GGBS
10% hlime-GGBS
5% hlime-GGBS

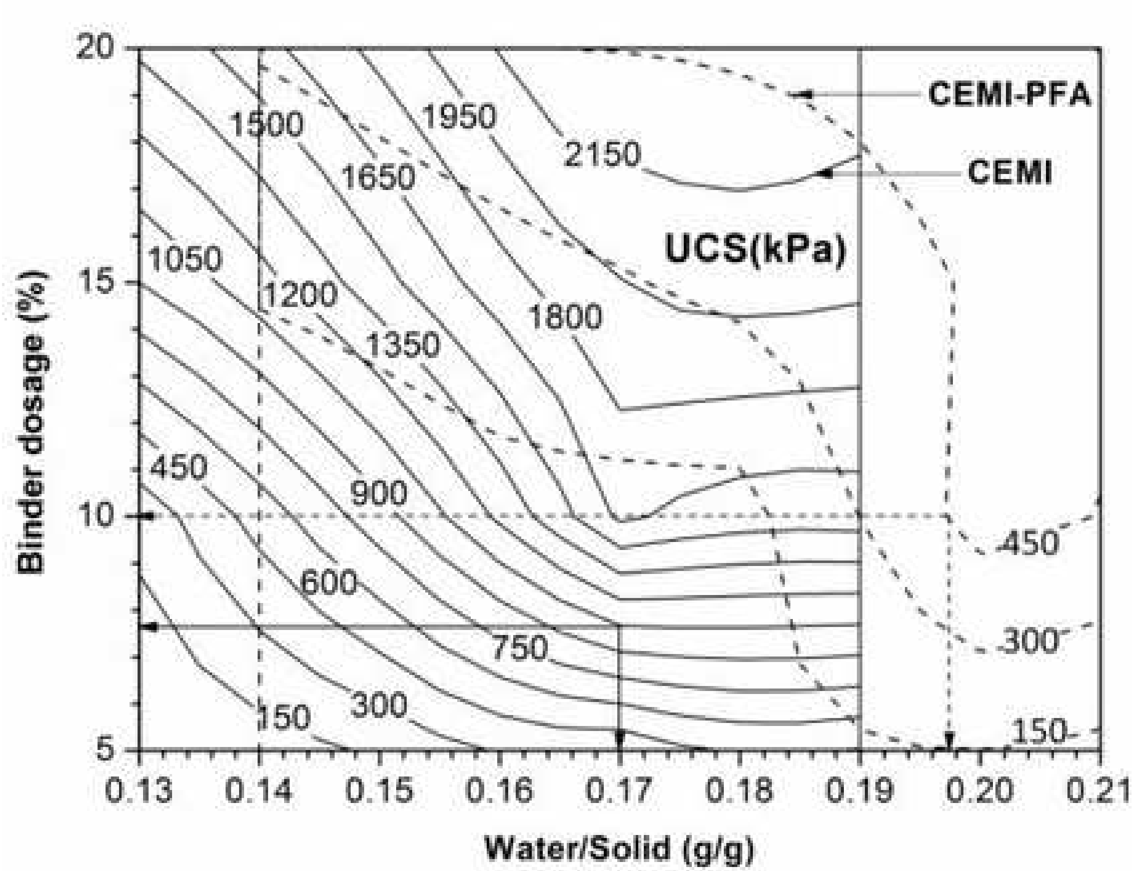
(a)



UCS (kPa)
2400
2000
1600
1200
800
400
0
0
28
56
84
Curing age (days)
5% CEMI
10% CEMI
5% CEMI-PFA
10% CEMI-PFA
5% CEMI-GGBS
10% CEMI-GGBS
5% hlime-GGBS
10% hlime-GGBS

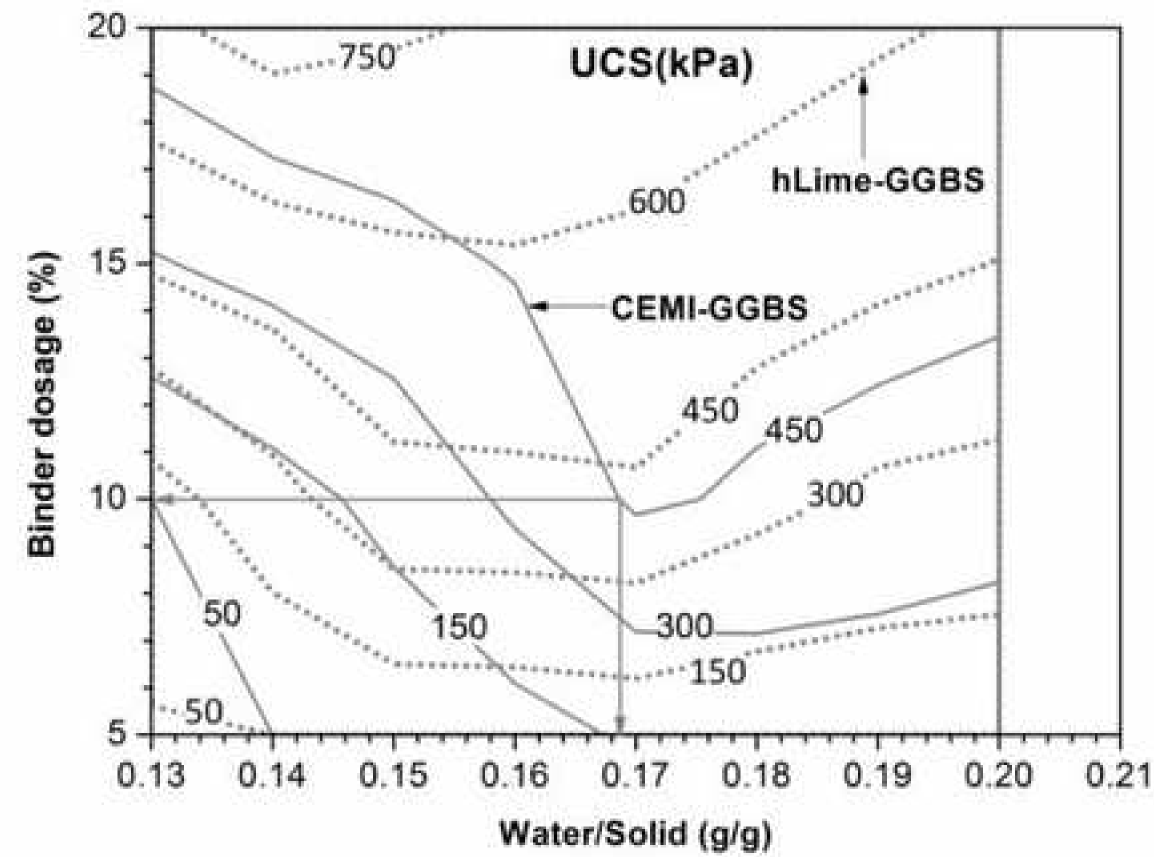
(b)

Figure 3



UCS(kPa)
CEMI-PFA
CEMI
1500
1950
2150
1650
1050
1200
1800
1350
450
900
600
750
300
150
450
300
150
Binder dosage (%)
Water/Solid (g/g)

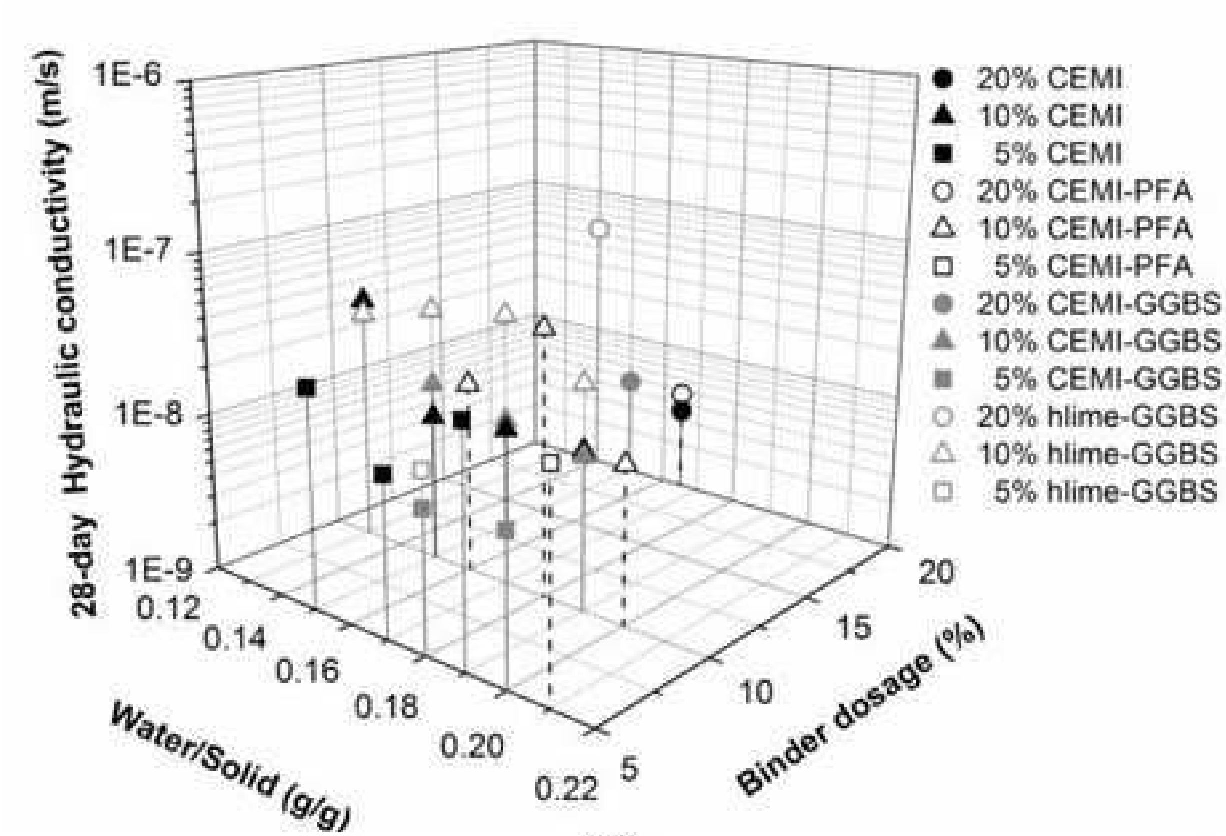
(a)



UCS(kPa)
750
600
hLime-GGBS
CEMI-GGBS
450
450
300
50
150
300
150
50
Binder dosage (%)
Water/Solid (g/g)

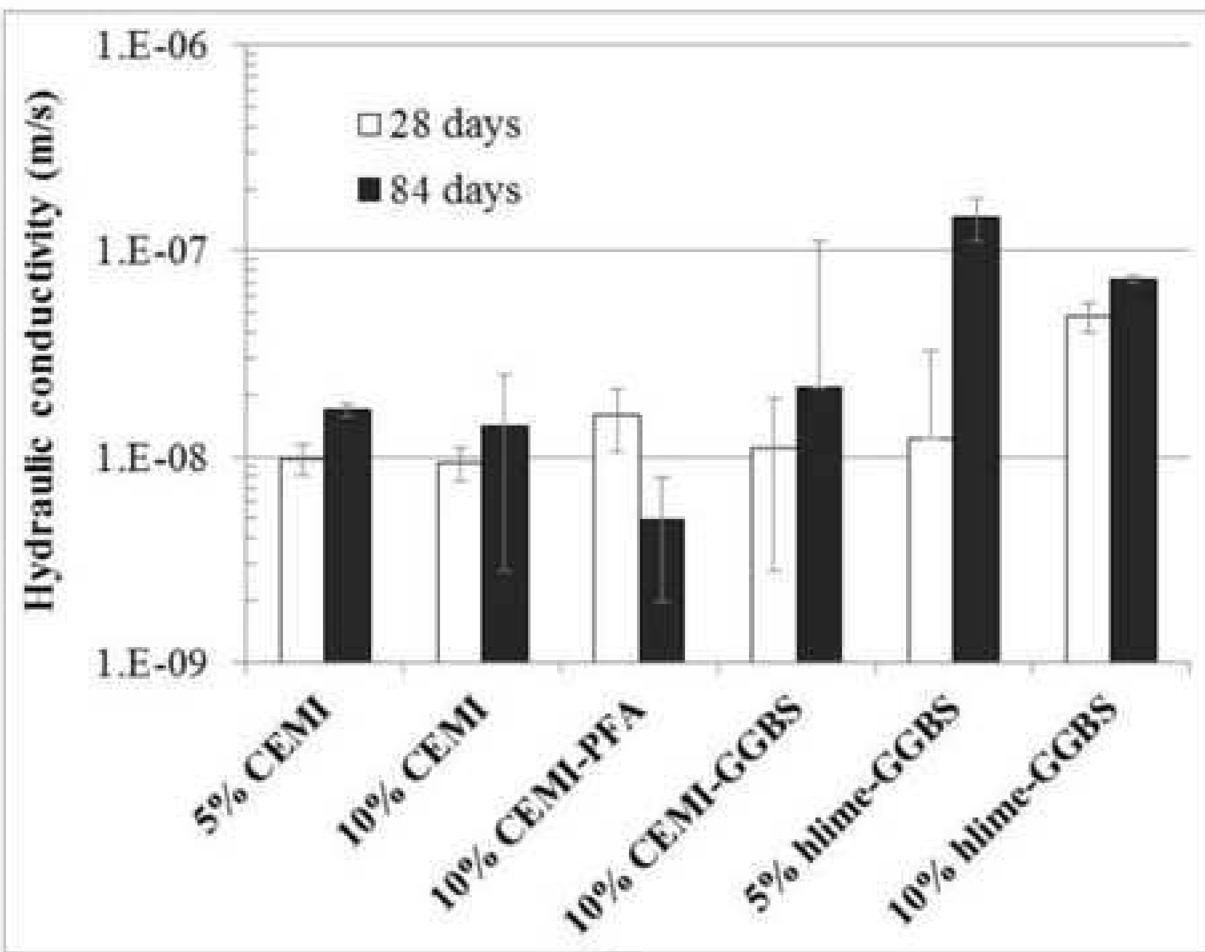
(b)

Figure 4



28-day Hydraulic conductivity (m/s)
1E-6
1E-7
1E-8
1E-9
0.12
0.14
0.16
0.18
0.20
0.22
Water/Solid (g/g)
5
10
15
20
Binder dosage (%)
20% CEMI
10% CEMI
5% CEMI
20% CEMI-PFA
10% CEMI-PFA
5% CEMI-PFA
20% CEMI-GGBS
10% CEMI-GGBS
5% CEMI-GGBS
20% hlime-GGBS
10% hlime-GGBS
5% hlime-GGBS

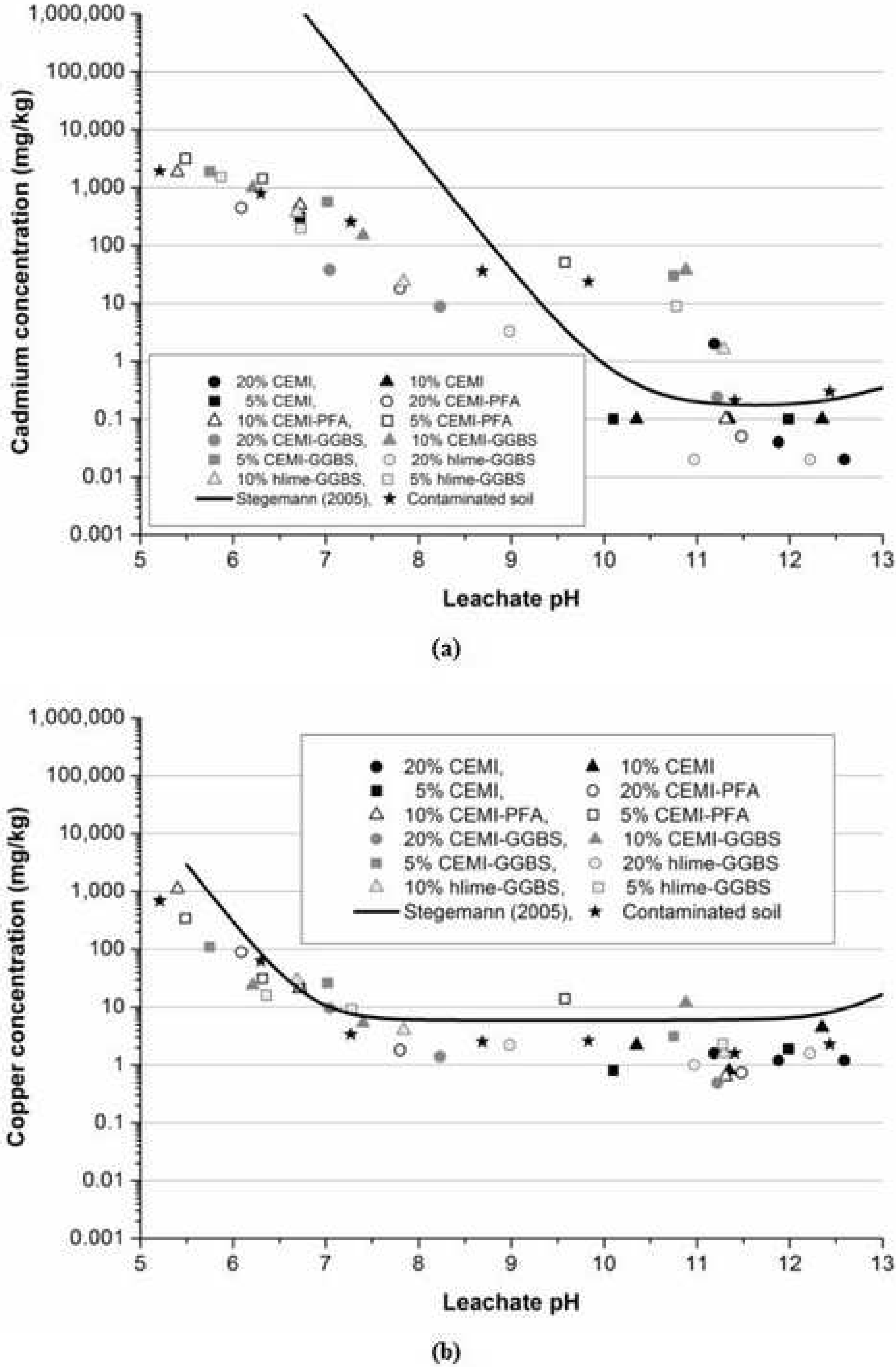
(a)



Hydraulic conductivity (m/s)
1.E-06
1.E-07
1.E-08
1.E-09
28 days
84 days
5% CEMI
10% CEMI
10% CEMI-PFA
10% CEMI-GGBS
5% hlime-GGBS
10% hlime-GGBS

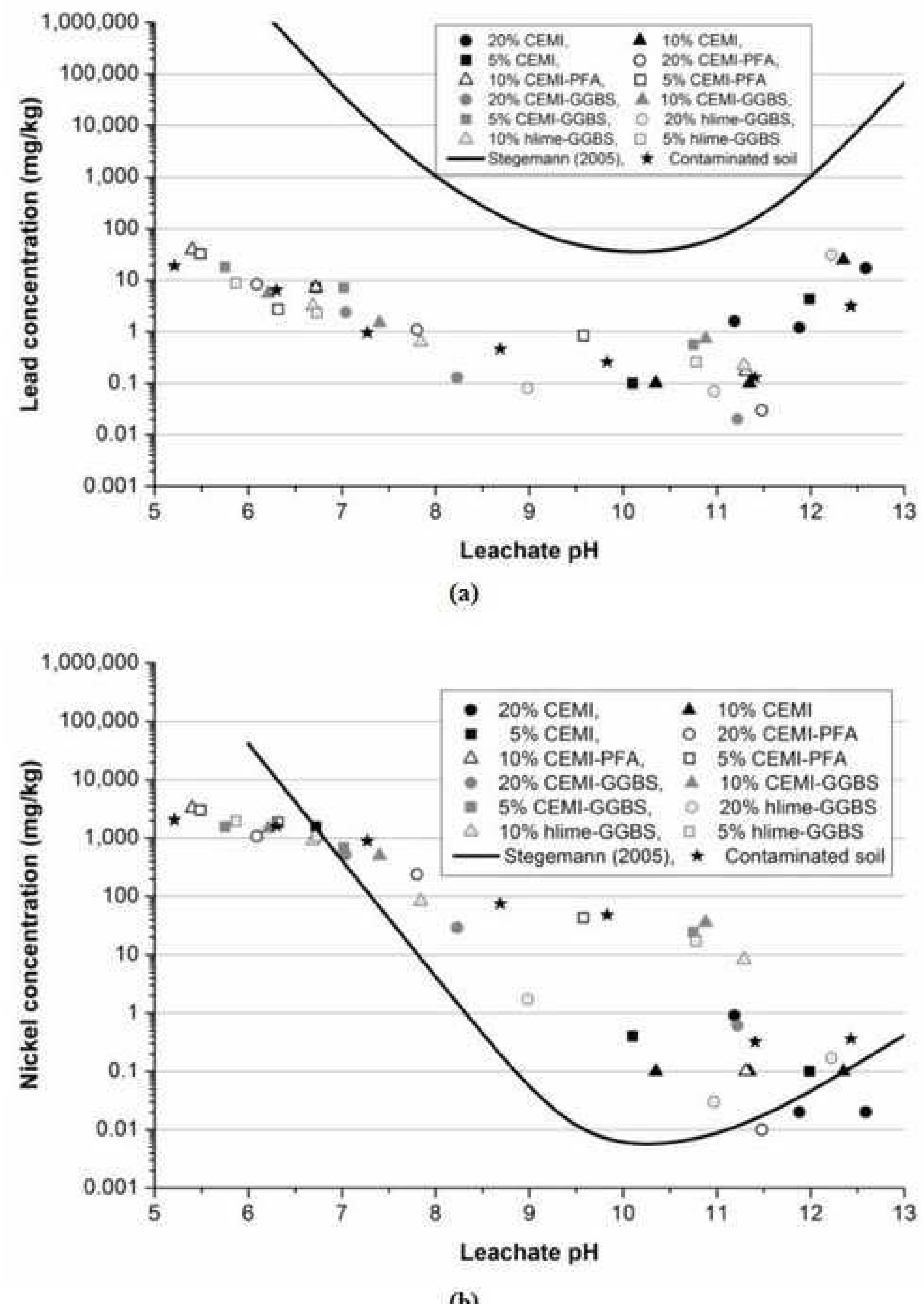
(b)

Figure 5



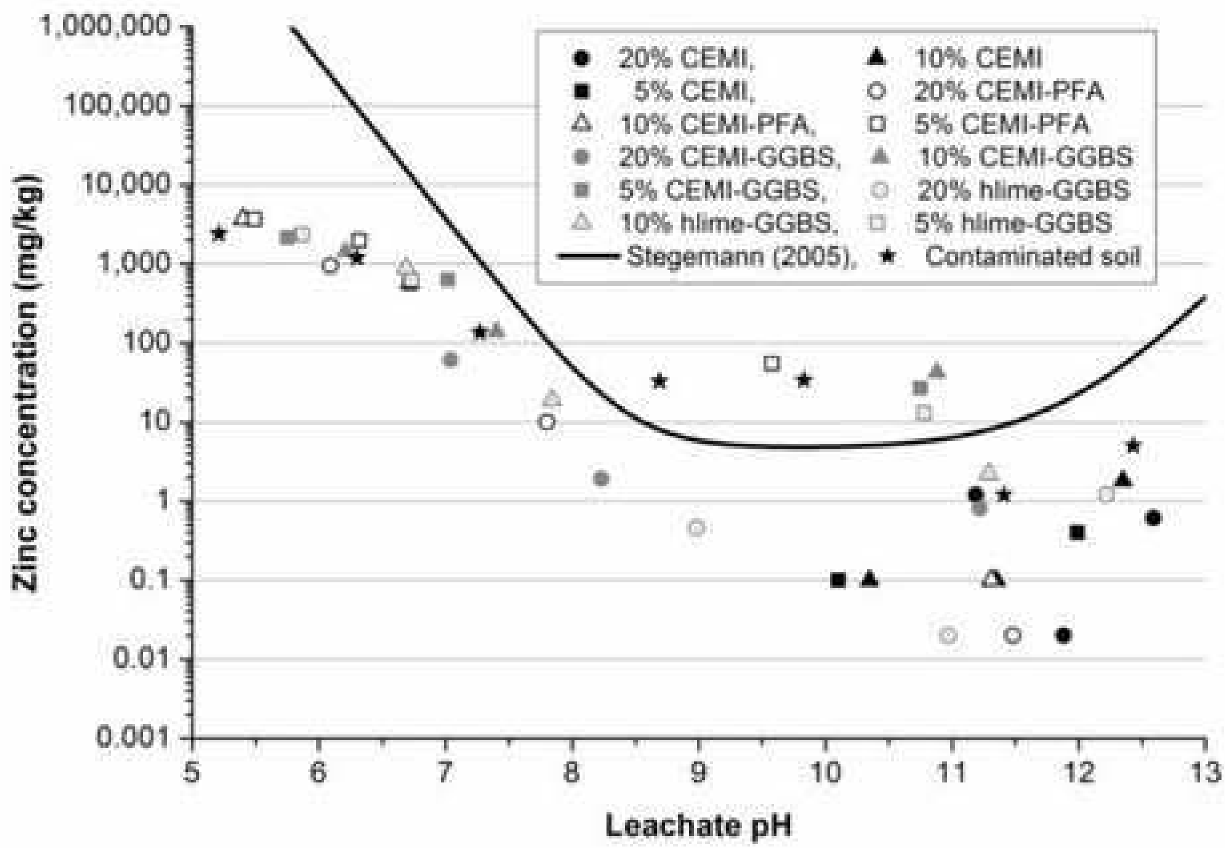
Cadmium concentration (mg/kg)
1,000,000
100,000
10,000
1,000
100
10
1
0.1
0.01
0.001
5
6
7
8
9
10
11
12
13
Leachate pH
20% CEMI,
5% CEMI,
10% CEMI-PFA,
20% CEMI-GGBS,
5% CEMI-GGBS,
10% hlime-GGBS,
Stegemann (2005),
10% CEMI
20% CEMI-PFA
5% CEMI-PFA
10% CEMI-GGBS
20% hlime-GGBS
5% hlime-GGBS
Contaminated soil
(a)
Copper concentration (mg/kg)
1,000,000
100,000
10,000
1,000
100
10
1
0.1
0.01
0.001
5
6
7
8
9
10
11
12
13
Leachate pH
20% CEMI,
5% CEMI,
10% CEMI-PFA,
20% CEMI-GGBS,
5% CEMI-GGBS,
10% hlime-GGBS,
Stegemann (2005),
10% CEMI
20% CEMI-PFA
5% CEMI-PFA
10% CEMI-GGBS
20% hlime-GGBS
5% hlime-GGBS
Contaminated soil
(b)

Figure 6



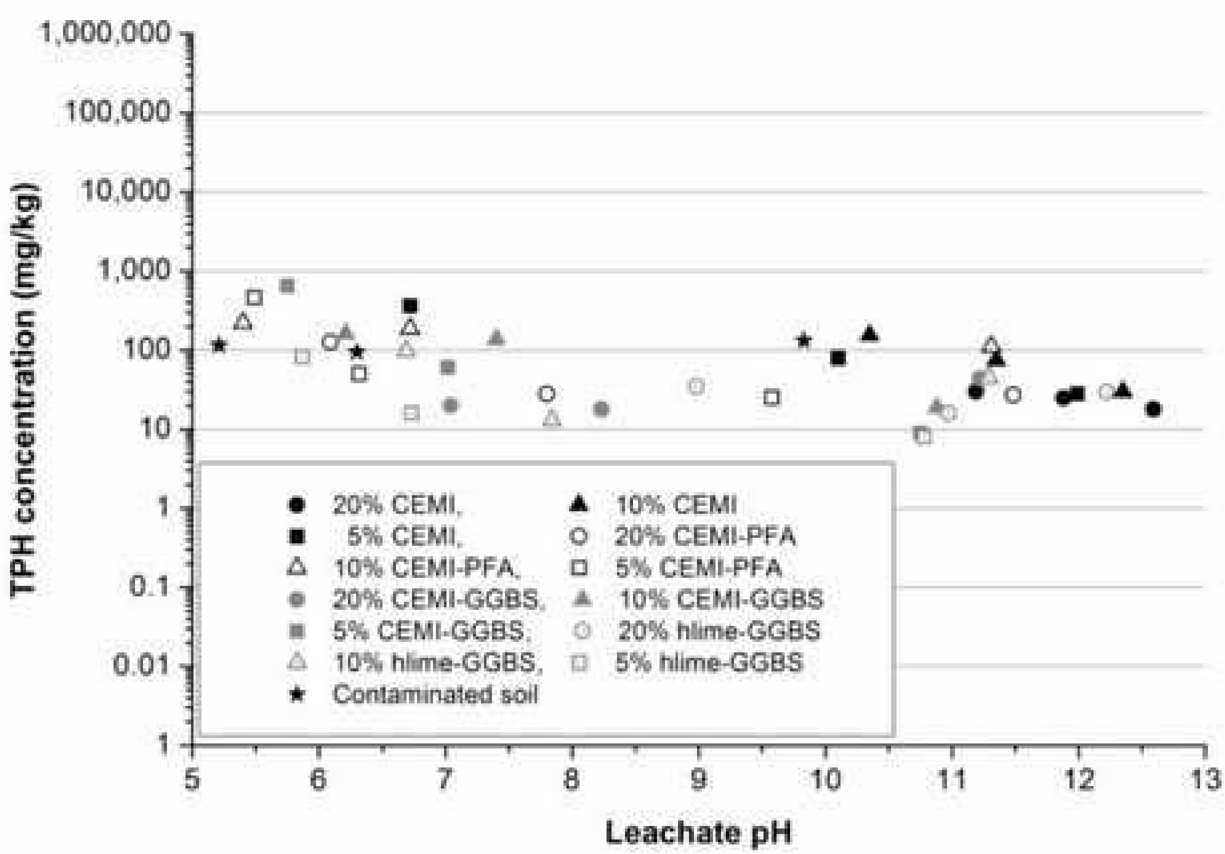
Lead concentration (mg/kg)
Leachate pH
20% CEMI, 10% CEMI, 5% CEMI, 20% CEMI-PFA, 10% CEMI-PFA, 5% CEMI-PFA, 20% CEMI-GGBS, 10% CEMI-GGBS, 5% CEMI-GGBS, 20% hlime-GGBS, 10% hlime-GGBS, 5% hlime-GGBS, Stegemann (2005), Contaminated soil
(a)
Nickel concentration (mg/kg)
Leachate pH
20% CEMI, 10% CEMI, 5% CEMI, 20% CEMI-PFA, 10% CEMI-PFA, 5% CEMI-PFA, 20% CEMI-GGBS, 10% CEMI-GGBS, 5% CEMI-GGBS, 20% hlime-GGBS, 10% hlime-GGBS, 5% hlime-GGBS, Stegemann (2005), Contaminated soil
(b)

Figure 7



Zinc concentration (mg/kg)
Leachate pH
20% CEMI,
10% CEMI
5% CEMI,
20% CEMI-PFA
10% CEMI-PFA,
5% CEMI-PFA
20% CEMI-GGBS,
10% CEMI-GGBS
5% CEMI-GGBS,
20% hlime-GGBS
10% hlime-GGBS,
5% hlime-GGBS
Stegemann (2005),
Contaminated soil

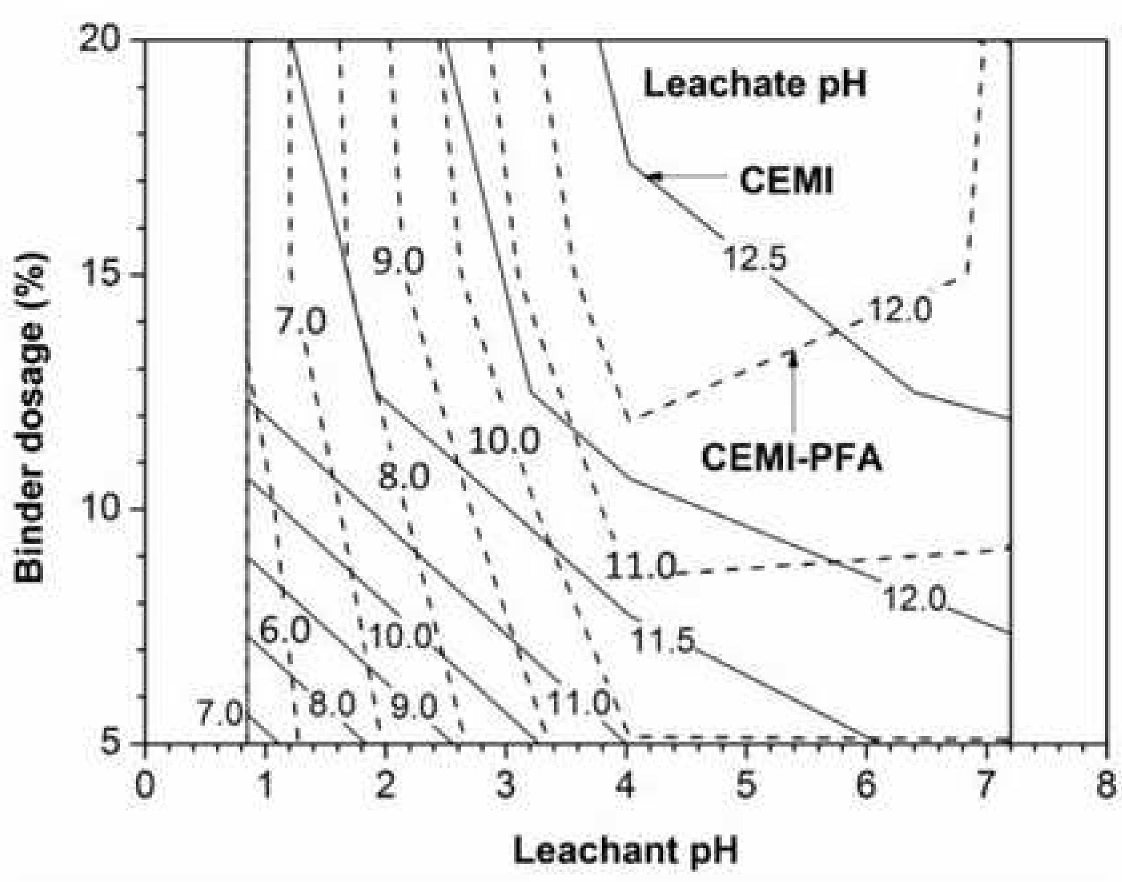
(a)



TPH concentration (mg/kg)
Leachate pH
20% CEMI,
10% CEMI
5% CEMI,
20% CEMI-PFA
10% CEMI-PFA,
5% CEMI-PFA
20% CEMI-GGBS,
10% CEMI-GGBS
5% CEMI-GGBS,
20% hlime-GGBS
10% hlime-GGBS,
5% hlime-GGBS
Contaminated soil

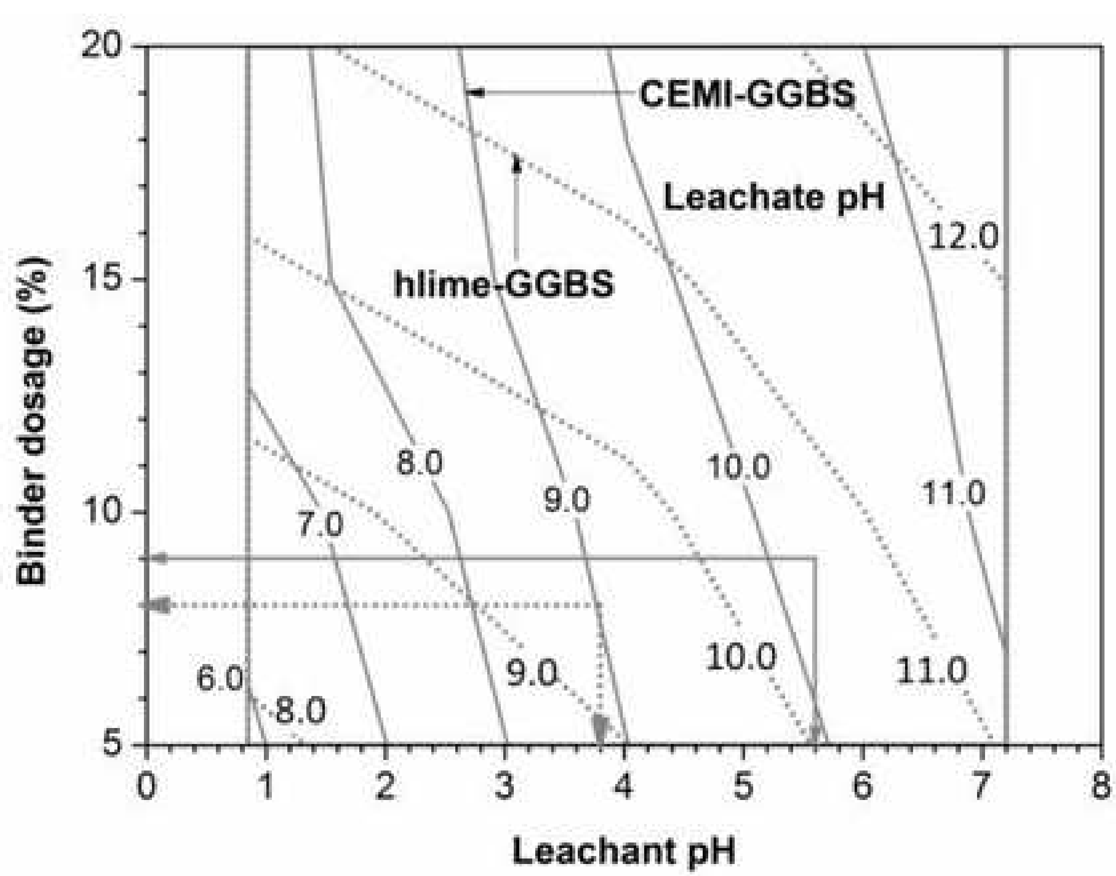
(b)

Figure 8



Leachate pH
CEMI
CEMI-PFA
12.5
12.0
9.0
7.0
10.0
8.0
11.0
12.0
11.5
6.0
10.0
7.0
8.0
9.0
11.0
Binder dosage (%)
Leachant pH

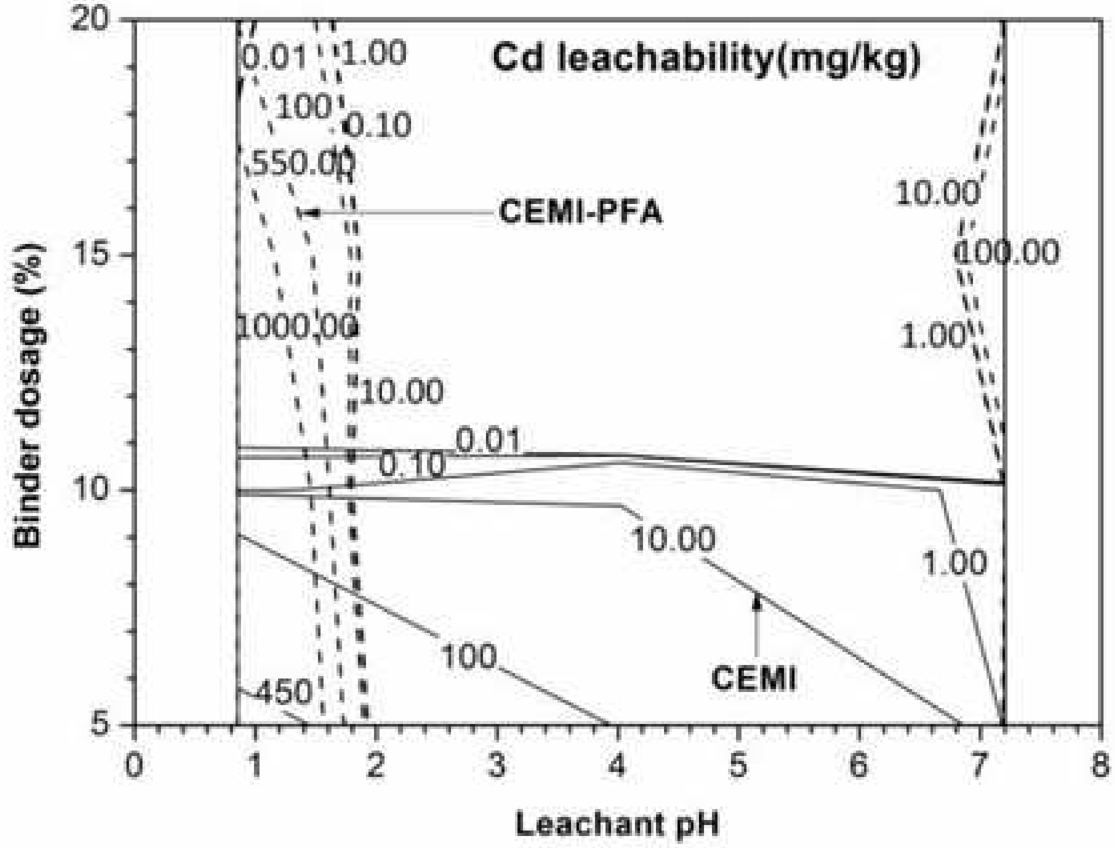
(a)



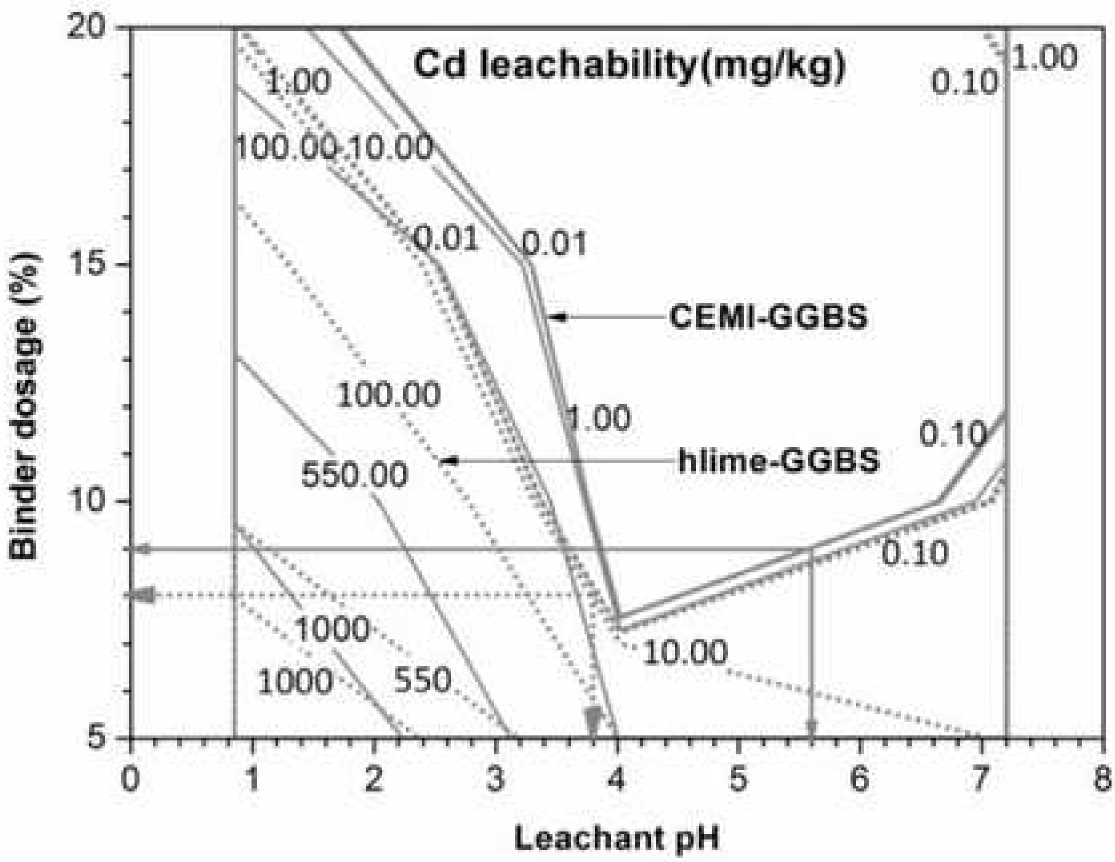
CEMI-GGBS
Leachate pH
hlime-GGBS
12.0
8.0
9.0
10.0
11.0
7.0
6.0
8.0
9.0
10.0
11.0
Binder dosage (%)
Leachant pH

(b)

Figure 9

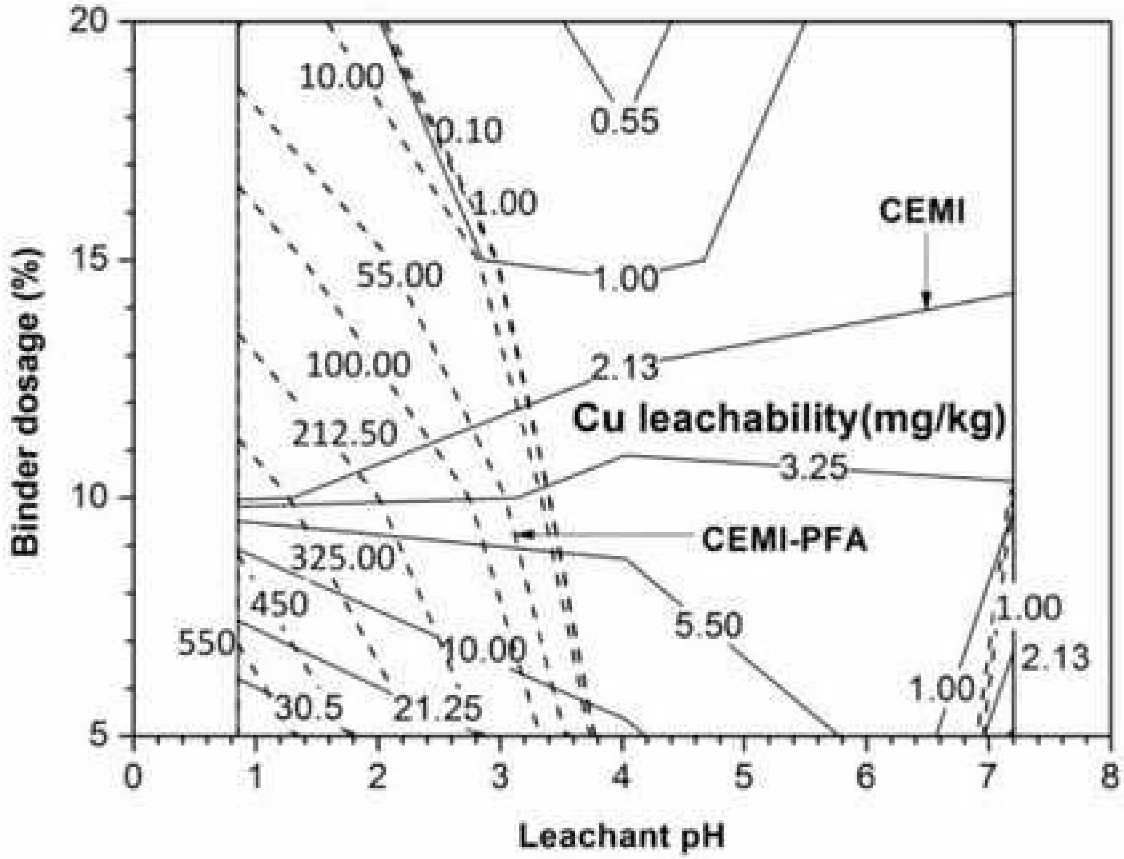

Cd leachability(mg/kg)
CEMI-PFA
CEMI
Binder dosage (%)
Leachant pH
0.01
1.00
100
0.10
550.00
10.00
100.00
1.00
1000.00
10.00
0.01
0.10
10.00
1.00
100
450

(a)

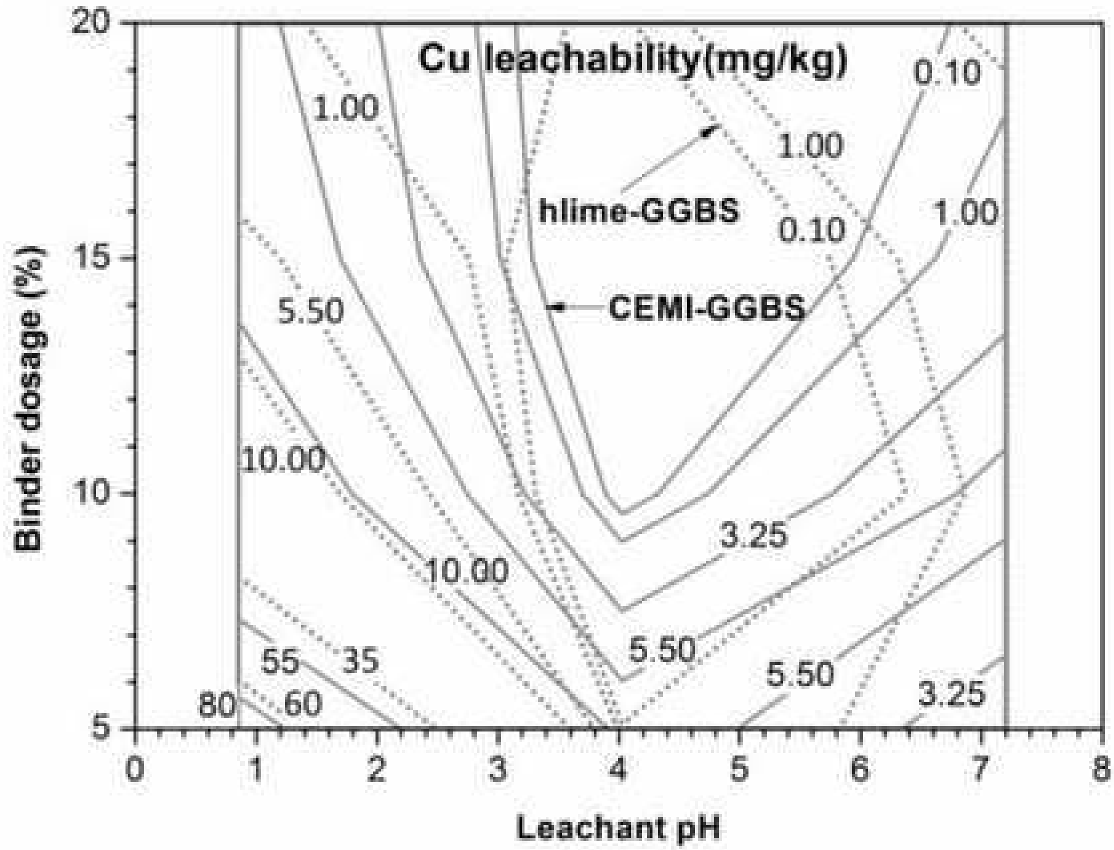

Cd leachability(mg/kg)
CEMI-GGBS
hlime-GGBS
Binder dosage (%)
Leachant pH
1.00
0.10
1.00
100.00
10.00
0.01
0.01
100.00
1.00
550.00
0.10
0.10
1000
10.00
1000
550

(b)

Figure 10

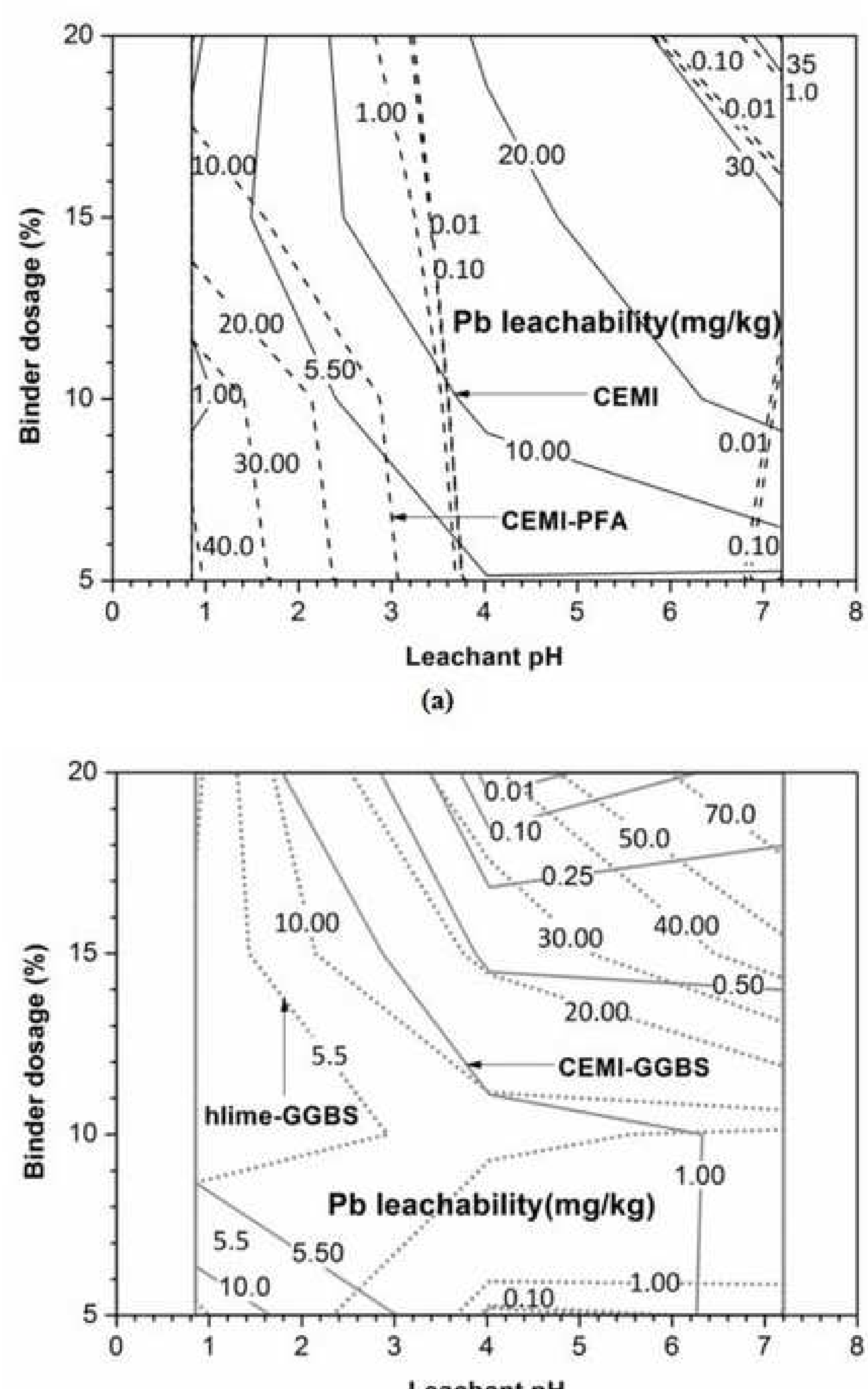

Cu leachability(mg/kg)
CEMI
CEMI-PFA
10.00
0.10
0.55
1.00
1.00
55.00
100.00
2.13
212.50
3.25
325.00
450
550
10.00
5.50
30.5
21.25
1.00
2.13
1.00
Binder dosage (%)
Leachant pH
20
15
10
5
0
1
2
3
4
5
6
7
8

(a)


Cu leachability(mg/kg)
hlime-GGBS
CEMI-GGBS
1.00
0.10
1.00
0.10
1.00
5.50
10.00
10.00
3.25
5.50
5.50
3.25
55
35
80
60
Binder dosage (%)
Leachant pH
20
15
10
5
0
1
2
3
4
5
6
7
8

(b)

Figure 11

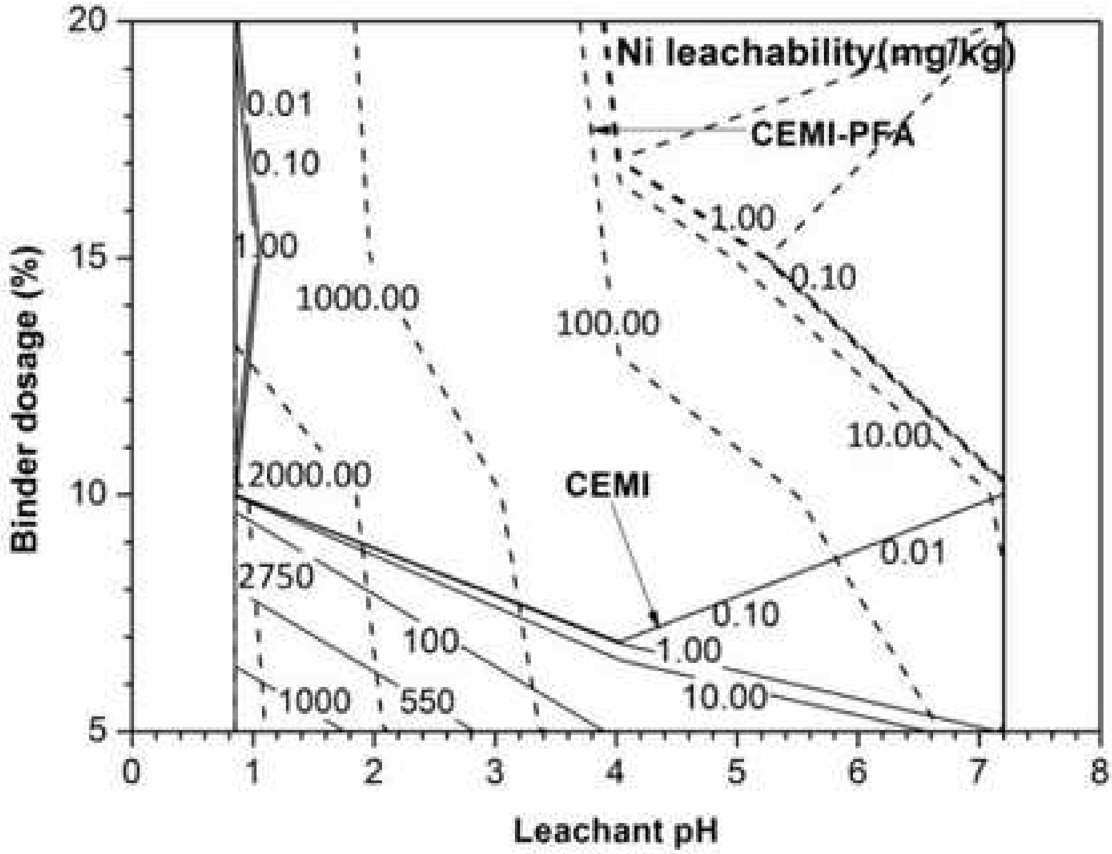


Pb leachability(mg/kg)
CEMI
CEMI-PFA
Binder dosage (%)
Leachant pH
Pb leachability(mg/kg)
CEMI-GGBS
hlime-GGBS
Binder dosage (%)
Leachant pH

(a)

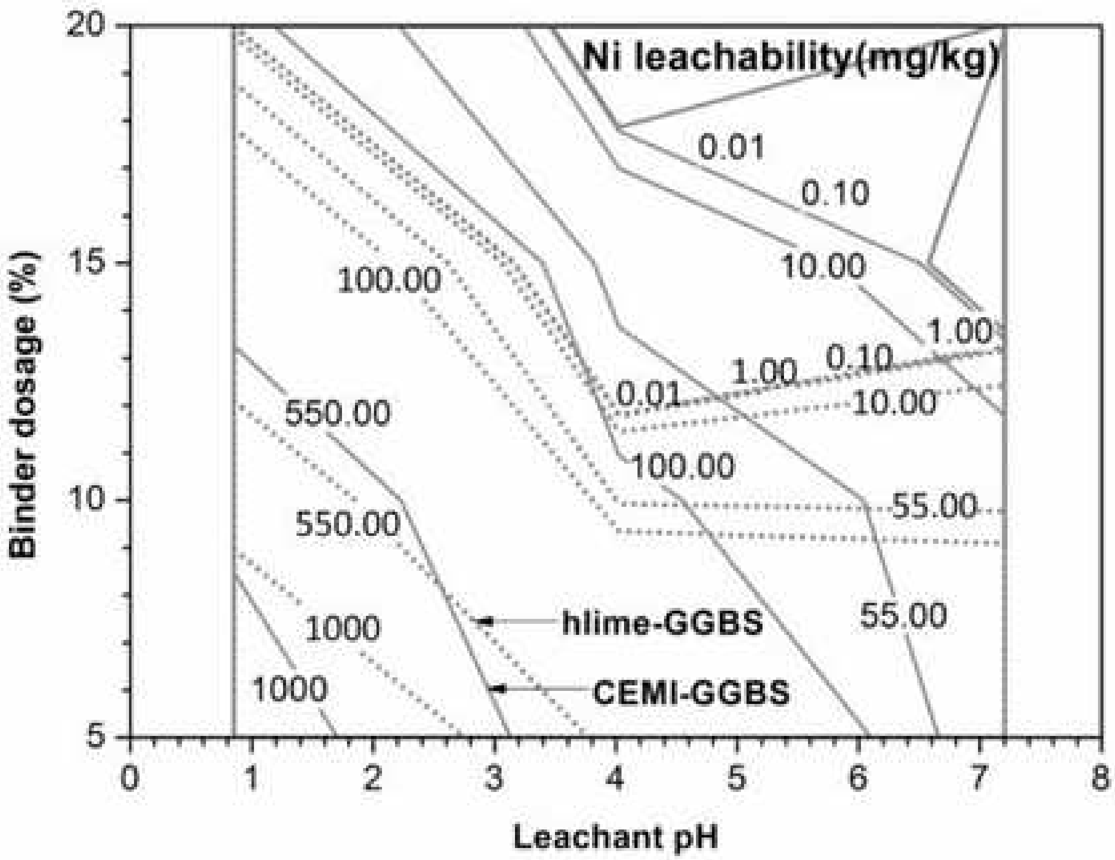
(b)

Figure 12



Ni leachability(mg/kg)
CEMI-PFA
CEMI
0.01
0.10
1.00
1000.00
100.00
2000.00
2750
100
1000
550
0.10
0.01
1.00
10.00
Binder dosage (%)
Leachant pH

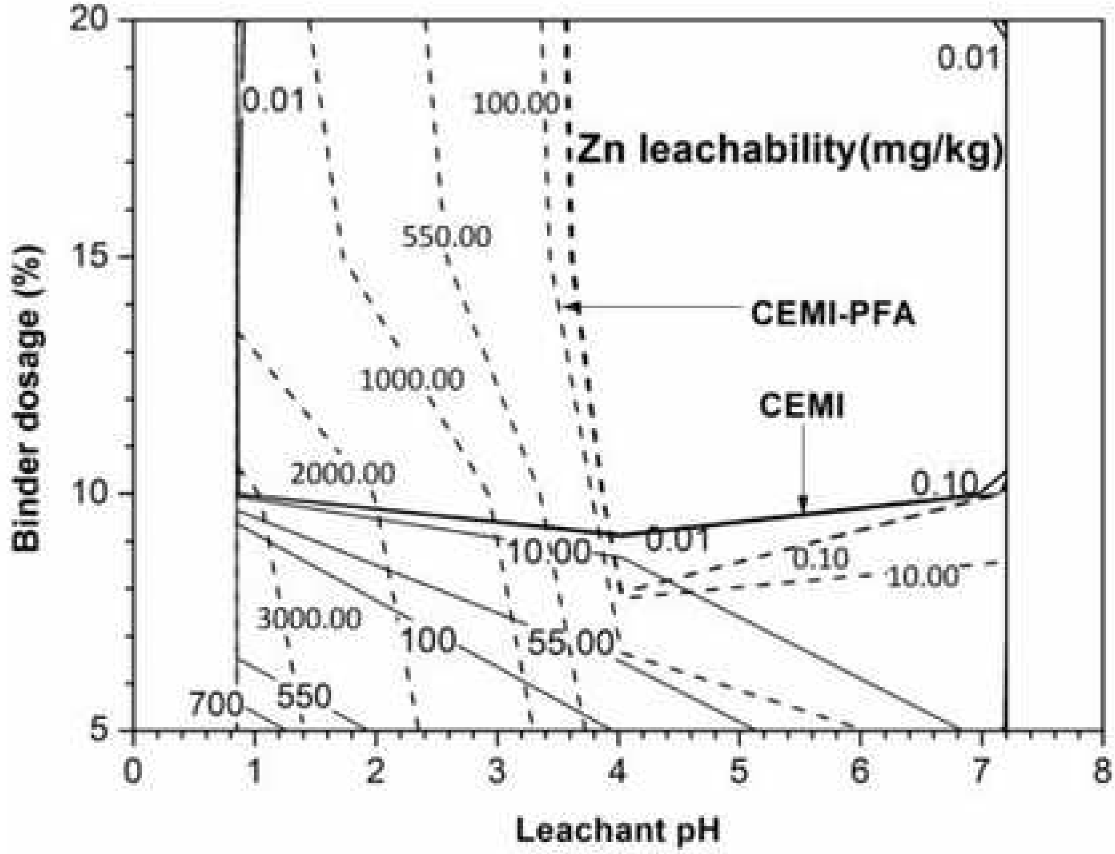
(a)



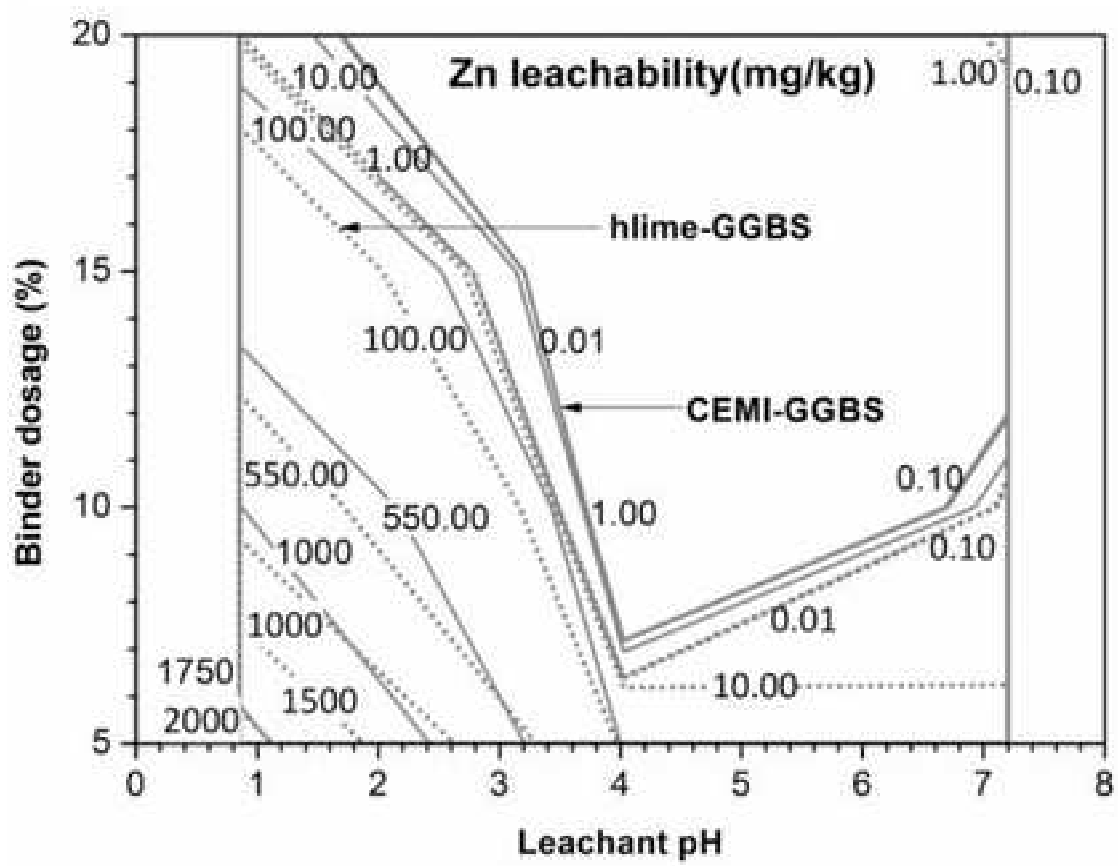
Ni leachability(mg/kg)
0.01
0.10
10.00
100.00
1.00
0.10
0.01
10.00
550.00
100.00
550.00
55.00
hlime-GGBS
1000
55.00
1000
CEMI-GGBS
Binder dosage (%)
Leachant pH

(b)

Figure 13

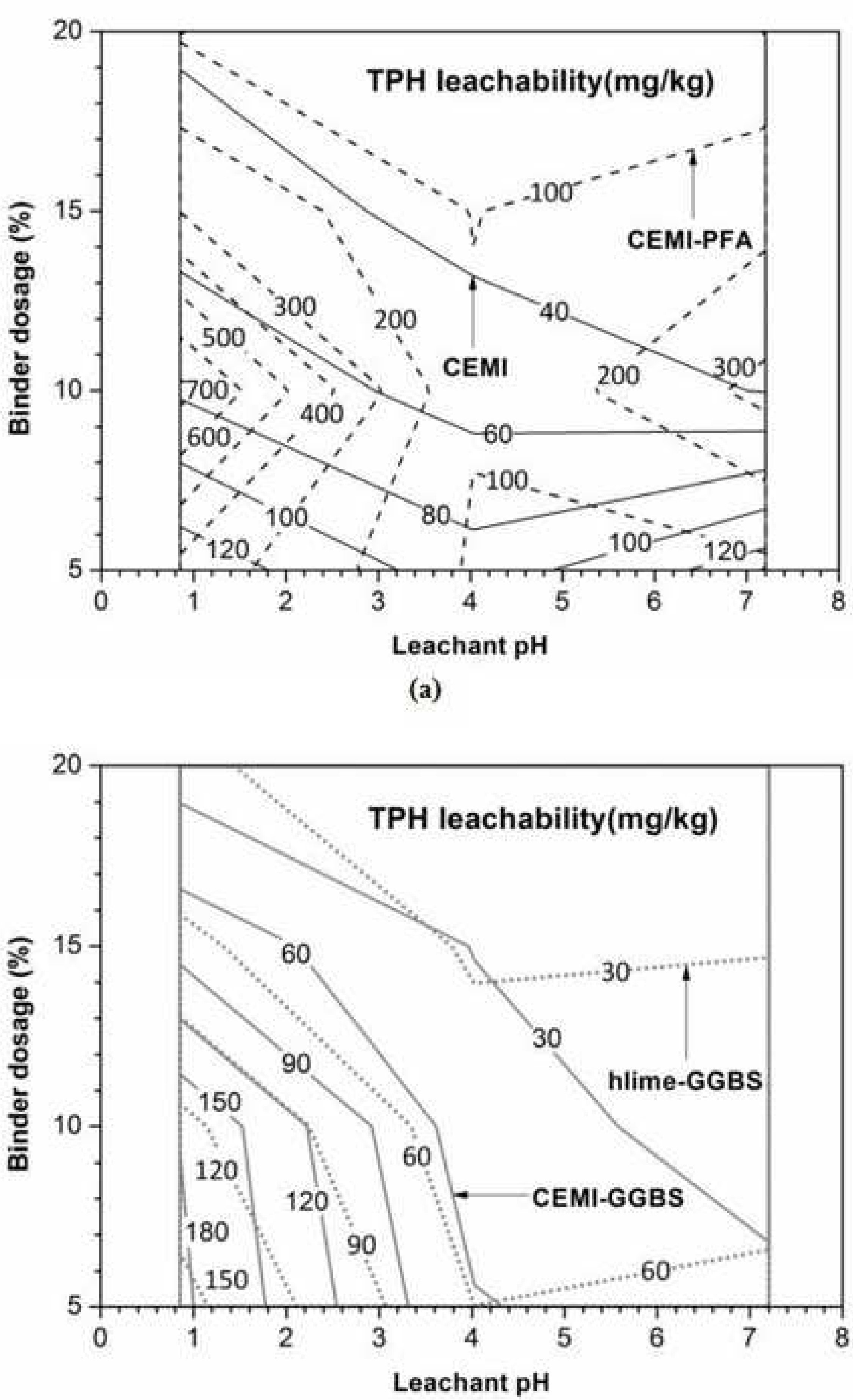

Zn leachability(mg/kg)
CEMI-PFA
CEMI
Binder dosage (%)
Leachant pH
0.01
100.00
550.00
1000.00
2000.00
3000.00
10.00
0.10
55.00
100
550
700

(a)


Zn leachability(mg/kg)
hlime-GGBS
CEMI-GGBS
Binder dosage (%)
Leachant pH
10.00
100.00
1.00
0.01
0.10
550.00
1000
1500
1750
2000

(b)

Figure 14


TPH leachability(mg/kg)
CEMI-PFA
CEMI
100
300
200
40
500
300
200
700
400
60
600
100
80
100
120
100
120
Binder dosage (%)
Leachant pH
TPH leachability(mg/kg)
hlime-GGBS
CEMI-GGBS
60
30
30
90
150
60
120
120
180
90
150
60
Binder dosage (%)
Leachant pH

(a)

(b)