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Manuscript Number: HAZMAT-D-14-03733R2

Title: The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

Article Type: Research Paper

Keywords: Soil stabilisation; novel binders; field trials; laboratory testing

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Abstract: This paper presents an investigation of the effects of novel binders and pH values on the effectiveness of the in situ stabilisation/solidification technique in treating heavy metals and organic contaminated soils after 1.5-year treatment. To evaluate the performance of different binders, made ground soils of SMiRT site, up to 5m depth, were stabilised/solidified with the triple auger system and cores were taken for laboratory testing after treatment. Twenty four different binders were used including PC, GGBS, PFA, MgO and zeolite. Unconfined compressive strength (UCS), leachate pH and the leachability of heavy metals and total organics were applied to study the behaviours of binders in treating site soils. Under various contaminant level and binder level, the results show that UCS values were 22-3476 kPa, the leachability of the total organics was in the range of 22-241 mg/l and the heavy metals was in the range of 0.002-0.225 mg/l. In addition, the combination of GGBS and MgO at a ratio of 9:1 shows better immobilization efficiency in treating heavy metals and organic contaminated soils after 1.5-year treatment, and the binding mechanisms under different binders were also discussed in this paper.

Dear Editor

Gerasimos Lyberatos: 22 Oct. 2014

Journal of Hazardous Materials

Journals Department

Ref. No.: HAZMAT-D-14-03733

Title: The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

Enclosed is the revised version of the paper entitled "The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil". I am pleased to submit for publication the revised version of the paper and appreciated the constructive and thorough reviews provide by the journal and two reviewers. In the revised paper, a few words and sentences have been corrected; some sentences have been added and two figures have been corrected. These changes I have made in response to reviewers' comments had been marked in Red in the body of the revised manuscript. Detailed response to their comments resulting in a number of clarifications is shown in the "detailed responses to reviewers" document.

Yours sincerely,

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Authors' Responses to Review Comments

Journal: Journal of Hazardous Materials

Ref. No.: HAZMAT-D-14-03733

Title: The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

Authors: Fei Wang, Hailing Wang, Fei Jin and Abir Al-Tabbaa

The authors would like to thank the insightful comments of the Editor and the reviewers, which are helpful to guide the authors' rework and revisions on the manuscript. Below is our response to the comments resulting in a number of clarifications.

Editor's summary:

The review of your manuscript, The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil, submitted to Journal of Hazardous Materials has been completed. The reviewers recommend the need for moderate revisions.

RESPONSE: We are pleased to have been given the opportunity to revise our work for Journal of Hazardous Materials. All of these review comments are considered carefully and responded point by point below.

Reviewer #1:

It looks as the beginner author.

RESPONSE: Thanks a lot for taking your time and effort to review this paper. The author is a beginner author and is on her way to be a mature author with your help.

It looks there was a technical error in sending of my comment on this paper so very small response from the author on my comment...

RESPONSE: Thank you for your feedback. There must be a technical error, as I only received one sentence above from you beforehand. Detailed responses are as follows:

The original comments are below so please the authors should take the attention:

This manuscript presents a study on the performance comparation of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil. The results are well described. However I cannot recommend the publication of this manuscript in the current form, and would reconsider the revisions. Some specific comments as follows:

The Novelty Statement needs revision, it should be focused only on the sentence "Blends of PC, GGBS together with magnesia and zeolite are promising alternatives as S/S binders and are the main subject." Need more elaboration on this statement and put the highlight of findings in the last paragraph of introduction section.

RESPONSE: Thank you for your comment. "They are expected to provide added benefits due to their individual credentials in improving the overall stabilisation/solidification effectiveness." was added into the novelty statement.

"The results show that the combination of GGBS and MgO at a ratio of 9:1 presents higher strength and better immobilization efficiency in treating heavy metals and organic contaminated soils after 1.5-year treatment. Besides, it was found that high strengths of treated samples can only be achieved when the concentrations of organic pollutants are less than 30 mg/L." was added in page 5 line 5.

Page (P) 3 line (L) 44-54 should have references.

RESPONSE: Thank you for your comment. [9, 43] was added in page 3 line 49. [44] was added in page 3 line 54.

"[43] D. G. Snelsona, S. Wilda, M. O'Farrellb, Heat of hydration of Portland Cement–Metakaolin–Fly ash (PC–MK–PFA) blends, Cem. Concr. Res. 38 (6) (2008) 832–840." and "[44] R. B. Kogbara, Process Envelopes for and Biodegradation within Stabilised/Solidified Contaminated Soils, PHD Thesis, Cambridge University, UK, 2011." were added to the end of the reference in the manuscript in page 19 line 32.

P4-L46 the sentence should not started with the not popular abreviation like "(S/S)".

RESPONSE: Thank you for your comment. "S/S" has been changed into "Stabilisation/solidification" in page 4 line 46.

Please, the authors should comment and need more elaboration on the possibility conversion of MgO in the soil to other risky specieses such MgCl2 or magnesium compound with one oxidation state (Mg+1) specieses.

RESPONSE: Thank you for your comment. SMiRT project site investigation shows heavy metals and organics are the main contaminants. Even there is a small amount of Cl^- exist in the site soil (Cl^- was not reported in the site investigation report), MgO will react with $MgCl_2$ and H_2O as reported by Bilinski et al. (1984). They claimed that the products of this reaction are crystalline $Mg_3(OH)_5Cl{\cdot}4H_2O$, $Mg_2(OH)_3Cl{\cdot}4H_2O$,and $Mg(OH)_2$ [1]. Hence $MgCl_2$ will not be able to exist.

In the case of the (Mg^{+1}) species, stable magnesium(I) compounds with Mg-Mg bonds were reported by Green et al. (2007)[2], but they can react with air and water, and are not able to be exist in the stabilisation/solidification system.

REF: 1) H. Bilinski, B. Matkovic, C. Mazuravic and T. B. Zunic, The Formation of Magnesium Oxychloride Phases in the Systems MgO-$MgCl_2$-H_2O and NaOH-$MgCl_2$-H_2O, Journal of the American Ceramic Society, 67 (4)(1984) 266–269.

2) S. P. Green, C. Jones, A. Stasch, Stable Magnesium(I) Compounds with Mg-Mg Bonds, Science 318, 1754 (2007);DOI: 10.1126/science.1150856.

Reviewer #2:

This is the review of the paper HAZMAT-D-14-03733 "The performance of blended conventional and novel binders in the in-situ stabilization/solidification of a contaminated site soil" by Wang et al. The paper is well written and of interest to the readers of the Journal of Hazardous Materials. However, the paper needs minor revisions, before publication. The strong point of the paper is that it investigates samples taken from an in-situ treatment field study. My major objection is how the samples taken 40 d after treatment are representative of what happens in the field 1.5 y later. Why not take new samples from the field 1.5 years after treatment?

RESPONSE: Thanks a lot for the review comments. The reason why we did not use new samples from the field 1.5 years after treatment is due to cost issues at that time.

1. Novelty statement, line 1: The paper does not deal with "time-related performance". It only investigates the one time 1.5-year after treatment performance.

RESPONSE: Thank you for your comment. We agree with your comment, the words "time-related" have been deleted from the novelty statement in line 1.

2. Abstract, line 2: The paper does not investigate "longevity" of the stabilization technique. It only refers to one time point, that of the 1.5 years after treatment.

RESPONSE: Thank you for your comment. The word "longevity" was changed into "effectiveness" in line 2 page 2.

3. Abstract line 12: Spell out the first time before using abbreviations, such as PC, GGBS, PFA.

RESPONSE: Thank you for your comment. Full names of PC, GGBS, PFA (Portland cement (PC), ground granulated blastfurnace slag (GGBS), pulverised fuel ash (PFA)) have been added in line 12 page 2.

4. Page 3, line 31: Change "precipitations" to "precipitating".

RESPONSE: Thank you for your comment. The change of "precipitations" to "precipitating" has been done in line 31 page 3.

5. Page 3, line 41: Change "significant" to "significantly".

RESPONSE: Thank you for your comment. The change of "significant" to "significantly" has been done in line 39 page 3.

6. Page 4, line 44: Does the 500 m2 refer to the area occupied by the soil or it should be 500 m3?

RESPONSE: Thank you for your comment. The 500 m^2 refer to the surface area occupied by the soil.

7. Page 5, lines 49-51: Why was the diameter decreased from 100 mm to 55 mm and when was this done?

RESPONSE: Thank you for your comment. The sampling took place at the site 28 days after treatment, the reason why the diameter decreased from 100 mm to 55 mm is because of the coring technic used in 2011.A smaller-diameter core liner was used

when going into deeper soils in order to reduce the coring resistance. To make the statement clearer, "The diameter of the cores decreased from 100mmm to 55mm." was changed into "The diameter of the cores decreased with depth as follows: 0-1m: 90mm, 1-2m: 80mm, 2-3m: 70mm and 3-4m: 55mm." in line 48 page 5.

8. Page 9, line 56: Change "exits" to "exists".

RESPONSE: Thank you for your comment. The change of "exits" to "exists" has been done in line 56 page 9.

9. Page 11, lines 29-37: Rewrite, poor syntax.

RESPONSE: Thank you for your comment. These sentences have been changed into (line 42-56 page 11):

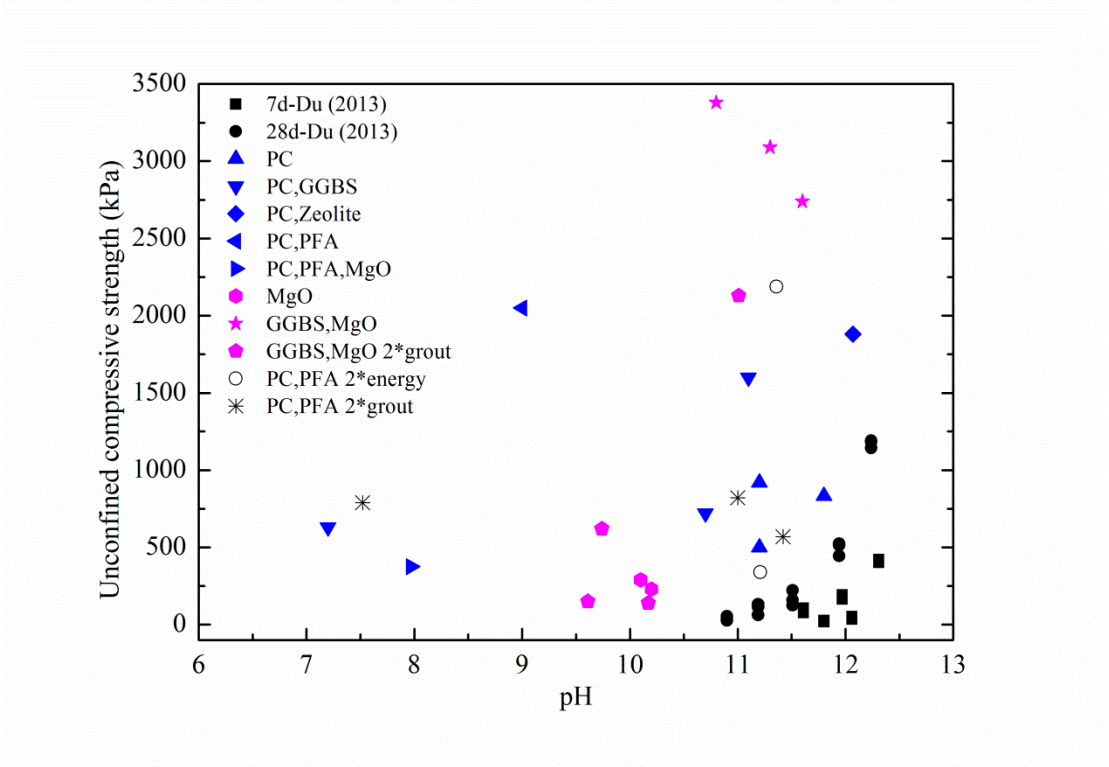
"Based on leachability studies and sequential extraction test, Wang et al. (2014) claimed that 40% of Ni was included in residual phase (fixed in a crystal structure or as a part of this structure) [32]. This explained the reason why the leachate concentration of Ni at pH 6-9 was lower than its solubility limit. When pH was in the range of 9.5-11.5, some Ni was reported to be immobilized in a Ni-Al layered double hydroxide (LDH) phase rather than Ni-hydroxides [42], hence Ni distributed slightly above its solubility line."

10. Figures 2a, 2b, 2c show error bars. What was the degree of replication in these measurements? It should be reported in the Methods section.

RESPONSE: Thank you for your comment. "All the tests were carried out at least in duplicate." was added in line 41 page 6.

11. Figure 3, y axis: What does "qu (kPa)(kPa)" mean?

RESPONSE: Thank you for your comment. qu (kPa) means Unconfined compressive strength, it is not necessary in figure 3. Revised Figure below will be uploaded.



12. Figure 4 and respective section 3.3 in the text: Why is the comparison made against single metal hydroxide solubilities? In addition to hydroxide solubility, additional phases and processes may be important (see Karamalidis and Voudrias, Journal of Hazardous Materials 141, 591 (2007); Environmental Engineering Science 26, 81 (2009) and Journal of Environmental Engineering, ASCE 134(6), 493 (2008)). How important are the metal carbonate phases, provided that carbonation is important after 1.5 years of treatment?

RESPONSE: Thank you for your comment. The solubility of the metal in an equilibrium solution in an all five metallic compounds (Ni, Cu, Zn, Pb, Cd) system was added for the comparison purpose. Relative added sentences below were marked in red and added in section 3.3:

Page 10 line 44: "while the solid lines with stars indicate the solubility of the metal in an equilibrium solution in an all five metallic compounds (Ni, Cu, Zn, Pb, Cd) system [31]."

Page 11 line 10: "both in a simple and a complicated environment."

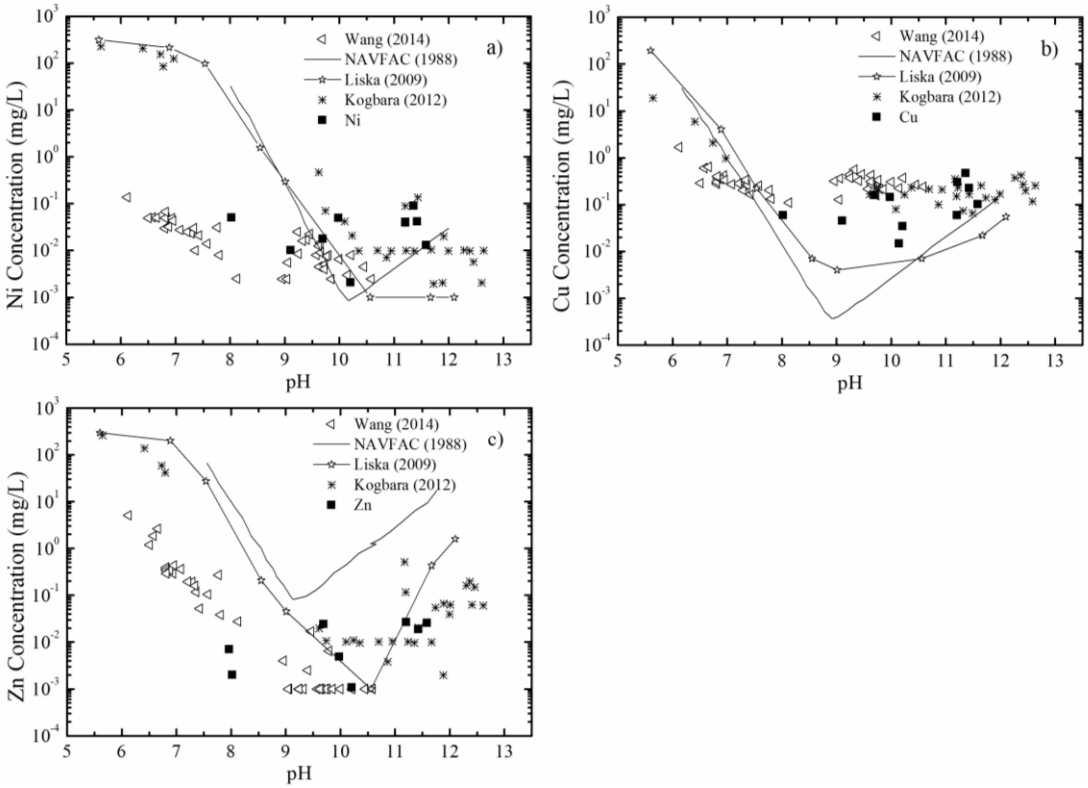
Page 11 line 26: "under a five metallic compounds system by Liska [31]"

Page 11 line 31: "and metallic compounds solubility limits at pH 6-9. However, under the pH of 9-12, the concentrations of Zn distributed along the solubility of Zn in a five metallic compounds system closely"

Page 12 line 17: "And at pH 9–12, $Zn(OH)_2$ was considered to be the controlling phase."

Revised Figure below will be uploaded.

Unfortunately, the degree of carbonation was not tested. We believed that the leachability of metals was mainly determined by metal hydroxides as we can see from Fig. 4 and hence was the main focus of this paper. Testing the degree of carbonation is a good suggestion for our further studies.



The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

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* Corresponding author

Highlights:

- MgO, GGBS (1:9) provided better physical and chemical performances at 1.5-year.
- MgO is effective in immobilising organics and inorganics, but produces low strength.
- Doubling mixing energy and grout content produce adverse impacts on treated samples.

The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

Fei Wang [a,*], *Hailing Wang* [b], *Fei Jin* [a] *and Abir Al-Tabbaa* [a]

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Novelty Statement: The objective of this paper is to investigate the performance of different binders in a heavy metals and organic contaminated site at 1.5-year after treatment. Blends of PC, GGBS together with magnesia and zeolite are promising alternatives as S/S binders and are the main subject. They are expected to provide added benefits due to their individual credentials in improving the overall stabilisation/solidification effectiveness. Currently these blends are not employed widely in large-scale field implementations. And most studies have focused on model contaminated soils rather than site soils and on heavy metals, rather than cocktail of organic and heavy metal contamination. Field applications of in-situ stabilisation/solidification using novel binders are essential in validating the efficiency of S/S.

*Abstract

The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

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ABSTRACT: This paper presents an investigation of the effects of novel binders and pH values on the effectiveness of the in situ stabilisation/solidification technique in treating heavy metals and organic contaminated soils after 1.5-year treatment. To evaluate the performance of different binders, made ground soils of SMiRT site, up to 5m depth, were stabilised/solidified with the triple auger system and cores were taken for laboratory testing after treatment. Twenty four different binders were used including PC, GGBS, PFA, MgO and zeolite. Unconfined compressive strength (UCS), leachate pH and the leachability of heavy metals and total organics were applied to study the behaviours of binders in treating site soils. Under various contaminant level and binder level, the results show that UCS values were 22-3476 kPa, the leachability of the total organics was in the range of 22-241 mg/l and the heavy metals was in the range of 0.002-0.225 mg/l. In addition, the combination of GGBS and MgO at a ratio of 9:1 shows better immobilization efficiency in treating heavy metals and organic contaminated soils after 1.5-year treatment, and the binding mechanisms under different binders were also discussed in this paper.

Keywords: Soil stabilisation; novel binders; field trials; laboratory testing

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The performance of blended conventional and novel binders in the in-situ stabilisation/solidification of a contaminated site soil

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A Paper Submitted for possible Publication as Research Papers to Journal of Hazardous Materials

ABSTRACT: This paper presents an investigation of the effects of novel binders and pH values on the effectiveness of the in situ stabilisation/solidification technique in treating heavy metals and organic contaminated soils after 1.5-year treatment. To evaluate the performance of different binders, made ground soils of SMiRT site, up to 5m depth, were stabilised/solidified with the triple auger system and cores were taken for laboratory testing after treatment. Twenty four different binders were used including Portland cement (PC), ground granulated blastfurnace slag (GGBS), pulverised fuel ash (PFA), MgO and zeolite. Unconfined compressive strength (UCS), leachate pH and the leachability of heavy metals and total organics were applied to study the behaviours of binders in treating site soils. Under various contaminant level and binder level, the results show that UCS values were 22-3476 kPa, the leachability of the total organics was in the range of 22-241 mg/l and the heavy metals was in the range of 0.002-0.225 mg/l. In addition, the combination of GGBS and MgO at a ratio of 9:1 shows better immobilization efficiency in treating heavy metals and organic contaminated soils after 1.5-year treatment, and the binding mechanisms under different binders were also discussed in this paper.

Keywords: Soil stabilisation; novel binders; field trials; laboratory testing

1. INTRODUCTION

Due to a range of different industrial activities, a large number of sites are polluted with a wide range of organics and/or inorganics and continue to be so every year. Heavy metals and certain types of organics are highly persistent in soils presenting a severe health risk to people and receptors in general. Over the past few decades, several techniques were applied to treat contaminated soils and more recently there have been strong sustainability drivers for the adoption of in-situ remediation strategies. One of the most efficient and low risk remediation methods is stabilisation/solidification (S/S) [1] whose in-situ applications has increased significantly in recent years [2]. In-situ S/S has three main advantages: 1) it is well established as efficient and cost-effective; 2) it produces no spoil and, hence no landfill waste and 3) there is no risk to site workers of exposure to contamination. Portland cement (PC) and lime are the most commonly used binders for stabilisation/solidification treatments [1, 3, 4]. The pore water of PC and lime with a high pH ($12.5 < pH < 13.5$) limits the mobility of many heavy metals by decreasing their solubility, precipitating as hydroxides, and increasing their sorption onto the cement/lime mineral phases. Alternative binders such as pulverised fuel ash (PFA) and ground granulated blastfurnace slag (GGBS) are receiving increasing attention because as industrial by-products, they reduce the overall costs and significantly increase the sustainability of the binder, reducing the environmental impact of PC. As a synthetic pozzolan, PFA-PC blends achieved lower heat of hydration, which reduces the possible emission of volatile organic compounds during remediation and the unburned carbon content in PFA acts as a sorbent for organics [9, 43]. When GGBS is blended with PC, the rate of hydration of GGBS is much slower than that of PC, which contributes to later hydration improving the strength at medium and later ages [44]. Novel binders and additives (e.g. magnesia, zeolites) blended with those conventional binders are expected to provide added benefits due to their individual credentials [5] in improving the overall S/S

effectiveness. For example, a recent study [6] showed that low-grade MgO provided long-term stabilisation of soils heavily contaminated by heavy metals due to its lower pH at 9-11, minimizing heavy metals solubility and redissolution. Allagoa (2011) reported that due to the highly porous nature of zeolite, which provides large reaction surfaces that zeolite blends with PC offers enhanced strength and durability over PC alone [7]. Hence blends of PC, GGBS and/or ash together with magnesia and zeolite are promising alternatives as S/S binders and are the subject of this paper. Currently these blends are not employed widely in large-scale field implementations and have been restricted to a few laboratory investigations [8]. Most studies have focussed on model contaminated soils rather than site soils and on heavy metals, rather than cocktail of organic and heavy metal contamination [8-10]. Field applications of in-situ stabilisation/solidification using novel binders are essential in validating the efficiency of such a remediation process [8, 11].

Project SMiRT (Soil Mix Remediation Technology) reported in this paper performed the largest R&D field trials on a contaminated site in the UK employing in-situ stabilisation/solidification with the use of augers, as well as other remediation techniques [12]. The project was funded by the UK Technology Strategy Board and involved collaboration with 16 industrial partners [12]. It involved the use of a range of different mixing tools and additives to construct permeable reactive in-ground barriers, low-permeability containment walls, and for "hot-spot" soil treatment by S/S to treat 500 m^2 of contaminated soils at a site in Castleford, Yorkshire in May 2011, shown in Fig. 1. Stabilisation/solidification was applied using a triple auger system, where the soils were mixed to a depth of 4 m with a range of different binder blends consisting of PC, PFA, GGBS, MgO and zeolite. This paper reports on the performance of treated soil samples from the site up to 1.5 years after treatment, including, unconfined compressive strength, leachate pH and leachability of heavy metals and total organics. The objectives of this paper are to enable a better understanding of the

time-related performance of the treatment binders, and the effectiveness of these conventional and novel binders in treating site soil which were contaminated with both heavy metals and organics. The results show that the combination of GGBS and MgO at a ratio of 9:1 presents higher strength and better immobilization efficiency in treating heavy metals and organic contaminated soils after 1.5-year treatment. Besides, it was found that high strengths of treated samples can only be achieved when the concentrations of organic pollutants are less than 30 mg/L.

2. SITE, MATERIAL AND METHODS

The soil strata consisted of top soil, made ground, natural drift deposits and coal bedrock at depth. The top soil has a depth of ~0.1-0.35 m, overlying made ground, down to 4.5 m, which consisted of black sand and/or silt containing fragments of plastic, concrete and wood. The groundwater level was reported to vary between 3.2 and 3.9 m below ground level [13]. The summary of contaminants and their corresponding total concentrations in the field soil are detailed in Table 1, indicating the relatively high and variable concentrations of organic compounds and heavy metals.

A total of 24 soil-grout compositions were applied at the site, amongst which 12 mixes are selected for a detailed study in this paper, as shown in Table 2, other mixes will be discussed later in another paper. The layout of the 24 installations can be found in Fig. 1. The binder components ratios used in this project were varied as they were based on a preliminary laboratory study. As shown in Table 2, mixes in group 1 are PC based, mixes in group 2 are MgO based, and different mixing energy/installation methodologies were employed in the mixes of PC+PFA in group 3. The slurry content of all mixes was 15%, and the water to cement ratio was 1:1.

The depths of sampling went down to 4 m and 1 m long cores were collected in sealed plastic tubes from the site 28 days after treatment. The diameter of the cores decreased with depth as follows: 0-1m: 90mm, 1-2m: 80mm, 2-3m: 70mm and 3-4m: 55mm. The samples tested in this study, were kept after the testing at 40 days to assess the longer-term performance and which were then cured in their original sealed plastic tubes in the laboratory.

The cores were first trimmed into cylinders using a diamond saw cutter, to a length equal to twice the diameter [15-17] and the ends made flat with a deviation less than ± 0.05mm. The samples were then subject to UCS test in triplicate based on ASTM D4219-08 using a Uniframe 70-T0108/E loading frame. The crushed samples were then subjected to batch leaching following BS 12457-2 [18]. A liquid to solid ratio (L/S) of 10:1 was used by adding 50 g of crushed core sample with particle sizes between 1 and 4 mm into 500 ml of carbonated deionised water (pH=5.4) . After 24±3 hours of agitation, the leachate solutions were filtered through a 0.45 µm filter and tested for pH by using a pH meter of EUTECH pH510 and the concentrations of the heavy metals were analysed using a Perkin Elmer 7000 inductive couple plasma optical emission spectroscopy (ICP-OES).

The remaining liquid from the batch leaching test was transferred into a 1000 mL plug-contained conical flask for organic extraction. 5 ml of 12 M hydrochloric acid was added to speed the extraction reaction and also acts as a pH buffer. The extraction was conducted by adding 30 ml dichloromethane (DCM) into the flask and shaking for 2 mins. After repeating this extraction work three times, the complete extracted sample was then poured into a container, whose weight was recorded before pouring, for DCM evaporation, in a fume cupboard. The extracted volume and the waste volume were recorded. After about 48 hours' evaporation, the mass of the residual was recorded [19]. All the tests were carried out at least in duplicate.

3. RESULTS AND DISCUSSION

3.1 Effects of binders

Fig. 2 (a-c) shows the detailed results of UCS, leachate pH, leachate concentrations of total organics and heavy metals respectively in 12 mixes at 1.5 years after treatment. In Fig. 2a, the UCS values of mixes are all above the design values of 350 kPa used in the UK [20]. The UCS values of SS5,6,10,11 are less than the 440 kPa standard for controlled utilisation set by the Environment Canada WTC [21], and only the UCS values of SS2,3,4,7,9 are well above the 1 MPa standard for landfill disposal under the UK Environment Agency 2006 [22]. In group 1, the UCS values of S4 (CEM I + PFA) is the highest at 2635 kPa. It also can be found that extra binders and additives added increase the strength development except SS5 with additional PFA and MgO. This is due to that the main hydration products of PC are calcium silicate hydrates (C-S-H) and $Ca(OH)_2$. When GGBS, PFA or zeolite was added, $Ca(OH)_2$ was able to react with SiO_2, Al_2O_3 contained in them and hence produced more C-S-H gel. SS5 produced a weak strength, which is due to that MgO will not react with PC and PFA and only a small amount of PC was added. In group 2, SS6, in which only MgO was used as the binder, produced much lower strength than those mixes containing PC. This is because the hydration product of MgO (i.e., magnesium hydroxide) is relatively weak compared to the C-S-H formed in PC [39]. However, the UCS value of SS7 (GGBS + MgO) is the highest at 3476 kPa. This is due to the activation of GGBS by MgO producing C-S-H, magnesium silicate hydrates (M-S-H) and hydrotalcite ($Mg_6Al_2CO_3(OH)_{16}\cdot 4H_2O$)-like phases (Ht) [23] which provide high strength development. Compared to C-S-H, hydrotalcite-like phases are more voluminous, and lead to higher strength developments [24]. In addition, it can be found that 10% MgO in SS7 is more effective in activating GGBS than 50% PC used in SS2, with higher strength developed. And since 90% by-product was used with better

performance, the cost can be reduced a lot by SS7 as well. Comparing SS4 with group 3, SS4 produced a higher UCS value, which indicates that doubling the mixing energy produced an adverse impact on strength development. The lower values observed could be a result of the higher content of organic contaminants in soil, which reduced the strength of SS10, 11. Comparing SS4 with SS12 and comparing SS7 with SS8, it was found that SS4 and SS7 produced higher UCS values than SS12 and SS8 respectively, which indicates that doubling the total slurry content also produces an adverse impact on strength. This is because higher water binder ratio leads to lower strength development [37].

The leachability of total organics is a key factor in assessing the effectiveness of S/S in immobilising organic compounds. Fig. 2b indicates that the leachability of total organics at 1.5 years after treatment was in the range of 10-240 mg/l, where SS2 produced the highest value followed by SS5, SS1, SS9 and SS12 at 70 mg/l, 57 mg/l, 55 mg/l and 43 mg/l, respectively. It can be seen that the overall performance of group 2 compared to group 1 (except SS2) in treating organic compounds are of significant privilege. Specifically, mixes of SS7, SS6 and SS8 in group 2, which contained MgO, leached only 22 mg/l, 24 mg/l, and 15 mg/l of organic pollutants respectively. This is because hydrotalcite-like phases having structural charge, which is similar to that of bentonite, can lead to intercalation of ionic species with opposite charge [38]. This indicates that MgO based mixes may immobilise organics with charges while PC-based ones may not. In addition, it was found that binders with PFA leached less total organics. In particular, SS4, SS10 and SS11 leached only ~13 mg/l. This is because of the high adsorption ability of PFA for organic compounds [25]. Based on the pH values and the leachate concentration of total organics in all mixes in Fig. 2b, no obvious relation was found between them. This indicates that the pH was not the controlling factor in the organic-binder mixing system. This agrees well with the findings of

Kogbara et al. [26], who concluded that the pH of the leachant did not dominate the leaching of total petroleum hydrocarbon (TPH).

Fig. 2c displays the leachate concentrations of Ni, Cu, Zn, Cd and Pb from mixes 1-12 and their pH values at 1.5 years. As can be seen from Fig. 2c, the leachate pH of the 12 mixes at 1.5 years varied from 7.97 to 11.59, which is due to the different natures of the used binders. For example, MgO dissolved and precipitated as $Mg(OH)_2$, the equilibrium pH of which is ~10.5, while the presence of $Ca(OH)_2$ and other alkalis from PC hydration had the potential to significantly increase the pH to 12.5 or higher [27]. The maximum deviation of the leachate pH results was ±0.86. As can be seen from Fig. 2c, it is evident that the leachates concentrations of Cu, Zn, Cd and Pb in all mixes were all well below their corresponding drinking water standards at 2 mg/L, 3 mg/L, 0.005 mg/L and 0.01 mg/L respectively [28]. On the other hand, the concentrations of Ni in the leachate of some of the samples in group 1 and 3 (SS1, 4, 9, 10, 12) were above their drinking water standards at 0.02 mg/L [28]. More specifically, SS9 leached the highest concentration of Ni at 0.09 mg/l. This was followed by SS12 at 0.05 mg/l, this may due to the uneven distribution of heavy metals in the site soil. It should be noted that the leachate concentration of Ni, Zn, Cd and Pb in SS5, SS6 and SS8 were below the limit of detection at 0.009 mg/l, 0.006 mg/l, 0.001 mg/l and 0.006 mg/l respectively, which indicates that MgO plays an important role in stabilising these heavy metals. This is due to the fact that $Mg(OH)_2$ is the main hydration product, the pH of which is ~10.5, minimizing the solubility of most of metal and metalloid (hydr)oxides such as Pb, Cd, Zn and Ni [6]. In addition, brucite has a layered structure in which each layer consists of two sheets of OH in hexagonal close packing, with a sheet of Mg atoms between them [39] and, hence heavy metals may be adsorbed on the surface of brucite or encapsulated within it [41].

3.2 The relationship between UCS and leachate pH

Fig. 3 shows the UCS values as a function of leachate pH for each individual test. According to Fig. 3, at 1.5 years, under a relatively low pH ~ 7, the UCS value is >500 kPa. The UCS value increased slowly until the pH is ~10, and when the UCS goes up to 3400 kPa the pH value is ~11. The results of Du et al. (2014) [40] using PC to stabilise zinc-contaminated kaolin also showed increasing trends with the rise of pH values at the range of 10.5-12.5. The reason why high strength exists when pH at 10-11 is because when pH<10.8, C-S-H is not stable and will dissolve, reducing the strength [30]. In addition, Fig. 3 displays the distributions of strengths under different binders. The pH values of the mixes were mainly distributed in the range of 10-12. In group 1, it can be found that due to the involved PC, the hydration product of which can increase the pH to >12.5 [27], and the carbonation in the past 1.5-year, the leachate pH values are in the range of 11-12. In mix 5, the ratio of PC: MgO: PFA is 1:4:5, this indicates that a small amount of PC was added. As MgO precipitated as ($Mg(OH)_2$), the pH of which is ~10.5, and as carbonation took place during the 1.5-year, the leachate pH of SS5 is low at ~8. In spite of that MgO is not reacting with PC and PFA, and due to hydration products of PFA and MgO produce a weak strength, a low strength <500 kPa of SS5 was achieved. In group 2, as mentioned above, MgO produces low strength, however, the GGBS+MgO blends produced the highest pH values at ~11. It is also necessary to point out that three points belonging to the GGBS+MgO blends using double grout (SS8) were found in the range of 9.5-10.5, the strength of which were only ~500 kPa, this is because the higher the water/cement (W/C) ratio, the lower the strength developed.

3.3 The relationship among leached metal concentration, leachate pH and solubilities of metals

The leachate concentrations of metals (Ni, Cu, and Zn) in all mixes and their related solubilities of metal hydroxides are shown in Fig. 4. The solid lines show the solubilities of

single metal hydroxide as a function of pH in NAVFAC [29], while the solid lines with stars indicate the solubility of the metal in an equilibrium solution in an all five metallic compounds (Ni, Cu, Zn, Pb, Cd) system [31]. The horizontal triangle symbols represent both the BS EN12457 and Toxicity Characteristic Leaching Procedure (TCLP) leachate concentrations of metals in the West drayton project at 17 years after treatment [31], and the asterisks represent the lab work study of the effect of water content and binder dosage on leachate pH and contaminant leachability at 28-day for Cu, Pb, Ni, Zn by Kogbara et al. (2012) [26]. Finally, the solid square symbols represent the BS EN12457 leachate concentrations of metals in this study at 1.5 years' service time. It should be noted that, although 6 types of metals exist in raw soils (Table 1), only leachate concentrations of Ni, Cu, and Zn were high enough to be tested.

It is apparent that leachate pH governs the solubility of metal hydroxides both in a simple and a complicated environment. In Fig. 4a, the leachate concentration of Ni at pH 6-9 was lower than its solubility limit, and this agrees well with the results of site samples at 17 years by Wang and Al-Tabbaa [31]. When the pH was in the range of 10-13, the leachate concentrations of Ni were mainly distributed above its solubility of metal hydroxide. As can be seen from Fig. 4b, the leachate concentration of Cu, along with the data by Kogbara et al. [26] as well as by Wang and Al-Tabbaa [31], follows the same trend of the solubility curve of Cu under a five metallic compounds system by Liska [31]. With regard to Zn, although raw soils contain different initial concentrations of Zn, the Zn leachability was slightly lower than the single and metallic compounds solubility limits at pH 6-9. However, under the pH of 9-12, the concentrations of Zn distributed along the solubility of Zn in a five metallic compounds system closely (Fig. 4c).

The results of the solubility of metal hydroxides in Fig. 4 provide important information on the immobilization of pollutants in the S/S system. Based on leachability studies and

sequential extraction test, Wang et al. (2014) claimed that 40% of Ni was included in residual phase (fixed in a crystal structure or as a part of this structure) [32]. This explained the reason why the leachate concentration of Ni at pH 6-9 was lower than its solubility limit. When pH was in the range of 9.5-11.5, some Ni was reported to be immobilized in a Ni-Al layered double hydroxide (LDH) phase rather than Ni-hydroxides [42], hence Ni distributed slightly above its solubility line. As the oxides in soil can adsorb Cu from precipitating and other forms of Cu like CuO may exsit, this part of Cu may leach into the solution easily [33], which is the reason why Cu leachate concentration curves of S/S treated samples are above the theoretic solubility curves in Fig. 4b. One reason why the leachate concentration curve of Cu in this study is closer to the solubility curve may be that the high concentrations of organic pollutants in the site soils can enhance the adsorption of Cu [32]. The lower concentration of Zn found in Fig. 4c, is attributed to the fact that some Zn was formed into calcium zinc complex hydrated compound, as confirmed by the Fourier transform infrared spectroscopy (FTIR) [34]. This agrees with the work of Wang and Al-Tabbaa [31] and Kogbara et al. [26]. And at pH 9–12, $Zn(OH)_2$ was considered to be the controlling phase.

3.4 The relationship between UCS and the leachate concentration of total organics

Fig. 5 shows the distribution of UCS values as a function of the leachate concentration of total organics for each individual test. As can be seen from Fig. 5, strength varies from 230 to 3380 kPa at the range of 0-30 mg/L of leached total organics. When the leachate concentrations of total organics are > 40 mg/L, the strength levelled off at around 500 kPa regardless of applied binders.

The results of Fig. 5 show that high strengths of S/S treated samples can only be achieved when the concentrations of organic pollutants are < 30 mg/l. Organic contaminants can hinder the strength development of S/S treated samples and produce significant micro- and macrostructural changes to their products [35, 36]. The interference mechanisms of organics

include wrapping the binder grains into organics from reaction with water, influencing water surface tension [35]. Since both PC-based binders and MgO-based binders are affected by organic compounds, more work is needed to find out which group is less affected by organics.

3.5 Evaluation Index

Table 3 shows the overall performance of UCS, leachability of heavy metals and total organics for all mixes. In terms of UCS, 3 stars were set when the UCS value reached 1500 kPa, 2 stars were applied when the value was between 500 kPa and 1500 kPa, and 1 star was set when the value was less than 500 kPa. When it comes to immobilization of heavy metals, 3 stars were used when the leachate concentrations of heavy metal was less than the limit of detection of ICP, 2 stars were set when the value was between the limit of detection and their drinking water standard, and 1 star was used when the value was higher than their drinking water standard. Finally, when the concentrations of leached total organics were lower than 30mg/L (10% of the maximum leached total organics), 3 stars were applied, 2 stars were used when the value was between 30 mg/L and 70 mg/L (30% of the maximum leached value), 1 star was applied when the concentrations of leached total organics were higher than 70 mg/L. As can be seen from Table 3, SS7 (GGBS, MgO) with 3 stars in all three assessment tests, is considered to be the best mix in this study. This was followed by SS4 (PC, PFA), SS8 (GGBS, MgO double grout) and SS3 (PC, Zeolite). These four mixes are less effective in one of the three tests with two stars labelled. SS12 with double grout content of PC and PFA is assessed as the worst mix in this case study.

4. CONCLUSIONS

In this paper, the physical and chemical performance of the in-situ S/S treated soils after 1.5 years of service using novel binders have been investigated. The objective of this project was

to test the whole range of performances (process envelope) rather than design to specific performance criteria, thus both acceptable and poor performances are expected. The main findings of this study are summarised as follows:

- SS7 (MgO, GGBS at a ratio of 1:9) and SS4 (PC, PFA at a ratio of 1:2) provided better performance in strength development and immobility of heavy metals and total organics at 1.5-year.
- From the leachability results of the mixes of PC and PFA, it is clear that PFA is effective in decreasing the leachability of organics but it also increases the leachability of metals.
- MgO is found to be very effective in immobilising both heavy metals and organics, but the mechanical performance is poor.
- The relationship between pH values and the hydroxide solubility is a fundamental determinant of the leachability of heavy metals. However, it is not the same in the relation to pH values and the organics.
- UCS values of mixes increased from ~400 to ~3400 kPa when their pH values increased from 7 to 11.5.
- High strengths of S/S treated samples can only be achieved when the concentrations of organic pollutants are less than 30 mg/L.
- Doubling mixing energy and doubling grout content produce adverse impacts on the performance of treated samples.

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List of Figure Captions

Figure 1. Plan of field trials treatment.

Figure 2. Physical and chemical performance of mixes 1-12 at 1.5 years (a) UCS results, (b) leachate concentrations of total organics and (c) leachate concentration of heavy metals.

Figure 3. The relationship between UCS and pH.

Figure 4. The relationship among metal concentration, leachate pH and solubilities of metal hydroxides (a) Ni, (b) Cu and (c) Zn.

Figure 5. The relationship between UCS and the leachate of total organics.

Table

Table 1. Soil contaminants in the site soils based on two site investigations in 2002 and 2007 [14].

Items in contaminated soils	Concentration range (mg/kg)
Pb	95-175
Zn	150-220
As	130-140
Cr	700-1150
Cu	1075-1600
Ni	1170-2200
Total Organics	7185-9230

Table 2. Description of the soil-grout mixes.

Group		Binder			Binder components ratio	Slurry content (wt%)	Water: Cement	Installation methodology/ variables
Group 1	SS1	PC				15	1:1	
	SS2	PC	GGBS		1:1	15	1:1	
	SS3	PC	Zeolite		9:1	15	1:1	
	SS4	PC	PFA		1:2	15	1:1	
	SS5	PC	PFA	MgO	1:4:5	15	1:1	
Group 2	SS6	MgO				15	1:1	
	SS7	MgO	GGBS		1:9	15	1:1	
	SS8	MgO	GGBS		1:9	15	1:1	2x grout content
Group 3	SS9	PC	PFA		1:2	15	1:1	2x cycles of mixing
	SS10	PC	PFA		1:2	15	1:1	2x speed of rotation
	SS11	PC	PFA		1:2	15	1:1	2x withdrawal rate
	SS12	PC	PFA		1:2	15	1:1	2x grout content

Table 3. Evaluation of the effectiveness of different binders.

	Binder			UCS	Immobilization of heavy metals	Immobilization of total organics
SS1	PC			★★	★★	★★
SS2	PC	GGBS		★★	★★★	★
SS3	PC	Zeolite		★★★	★★	★★★
SS4	PC	PFA		★★★	★★	★★★
SS5	PC	PFA	MgO	★	★★★	★
SS6	MgO			★	★★★	★★★
SS7	MgO	GGBS		★★★	★★★	★★★
SS8	MgO	GGBS		★★	★★★	★★★
SS9	PC	PFA		★★★	★	★★
SS10	PC	PFA		★	★★	★★★
SS11	PC	PFA		★	★★★	★★★
SS12	PC	PFA		★★	★	★★

Figure 1

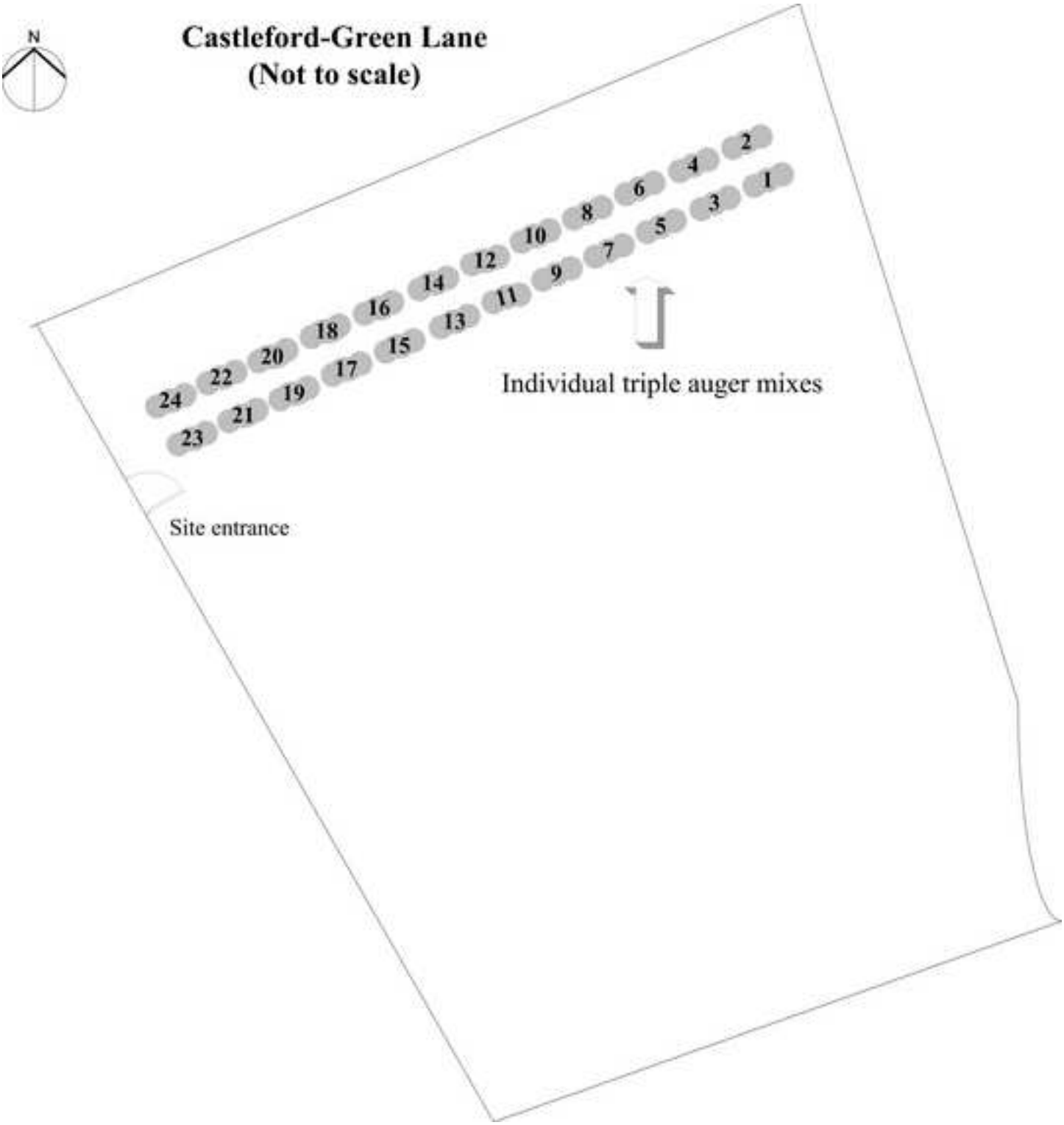

N
Castleford-Green Lane
(Not to scale)
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
Individual triple auger mixes
Site entrance

Figure 2a

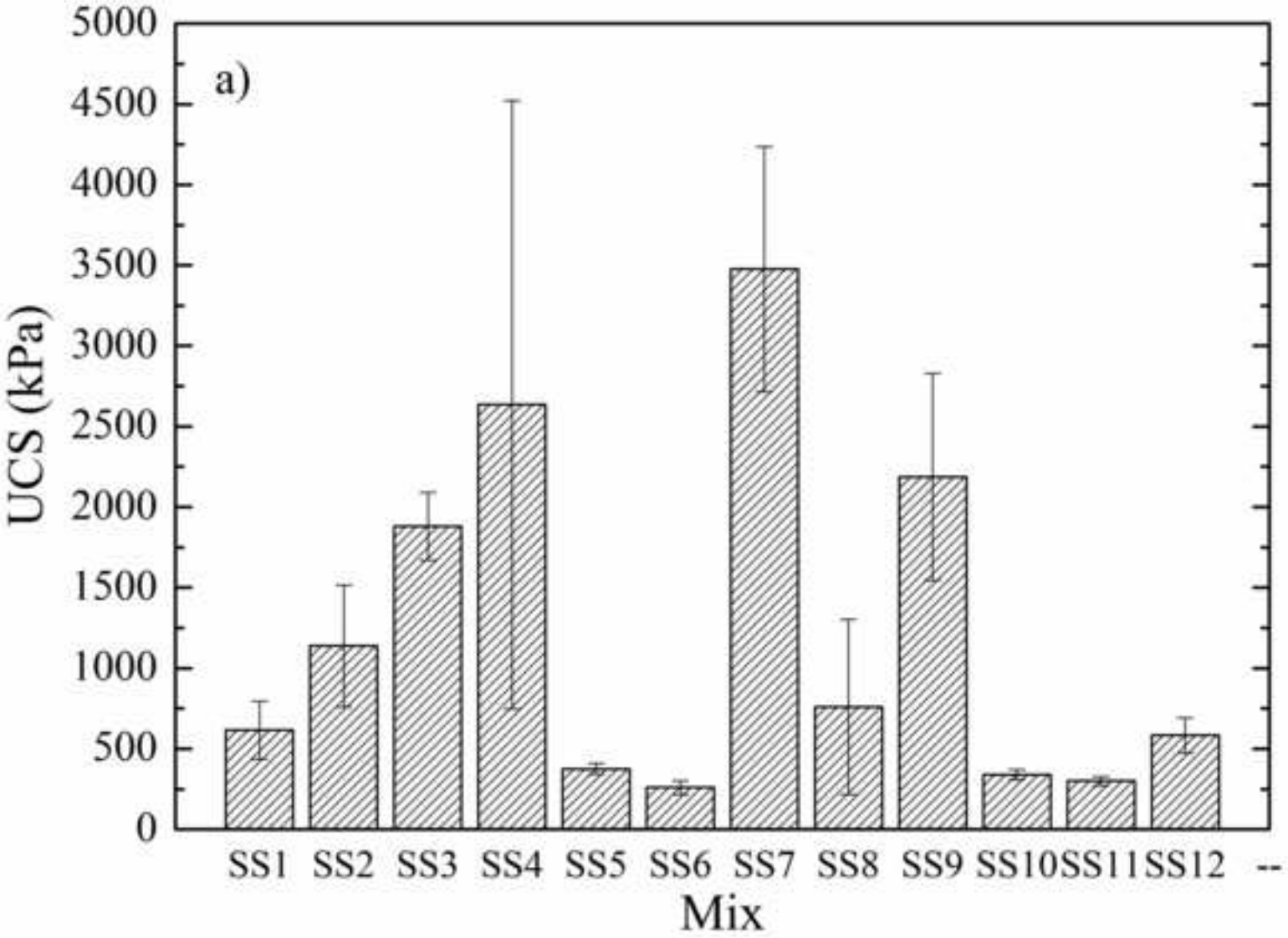

a)
UCS (kPa)
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
SS1
SS2
SS3
SS4
SS5
SS6
SS7
SS8
SS9
SS10
SS11
SS12
--
Mix

Figure 2b

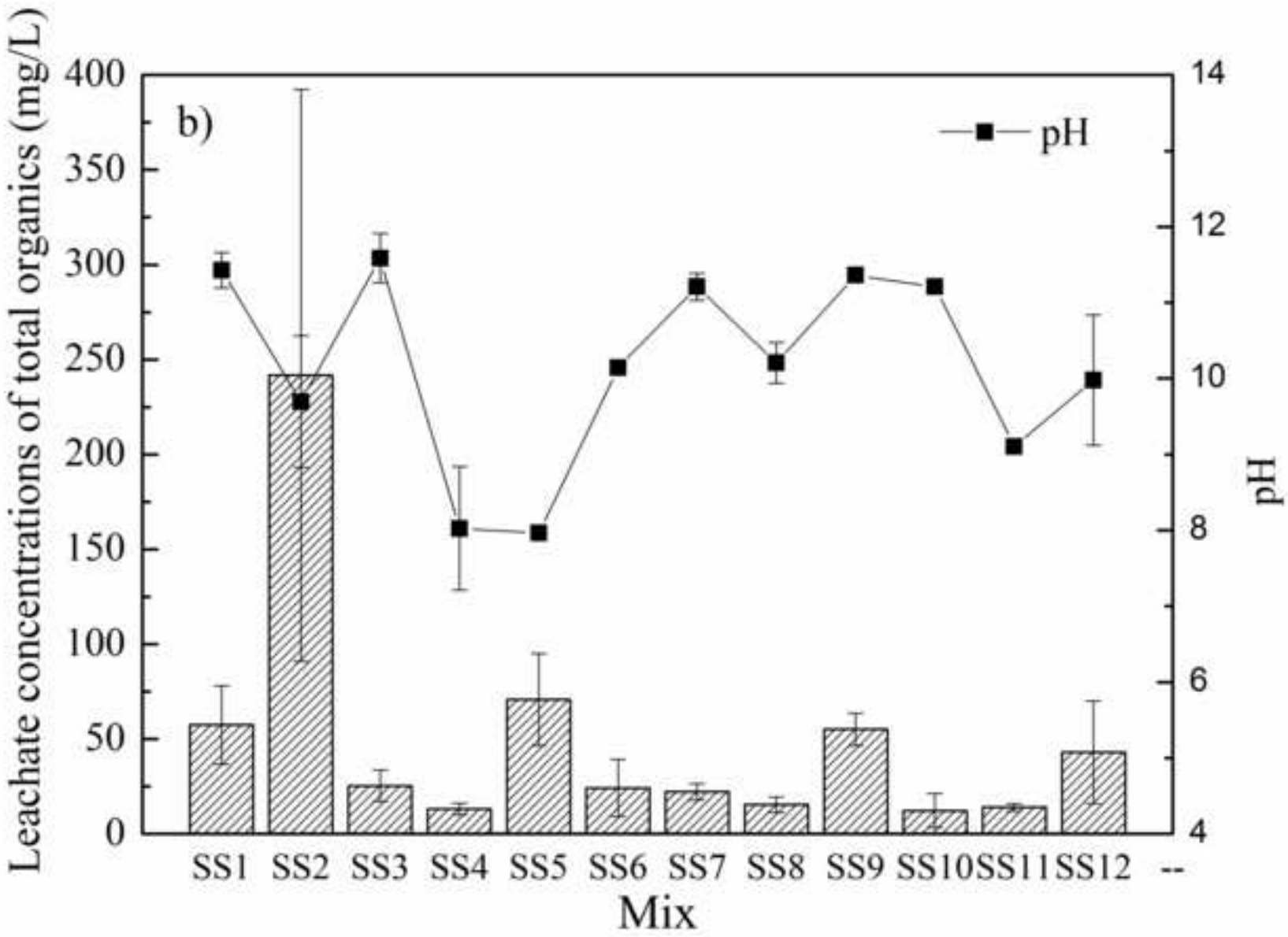

b)
pH
Leachate concentrations of total organics (mg/L)
pH
0
50
100
150
200
250
300
350
400
4
6
8
10
12
14
SS1
SS2
SS3
SS4
SS5
SS6
SS7
SS8
SS9
SS10
SS11
SS12
--
Mix

Figure 2c

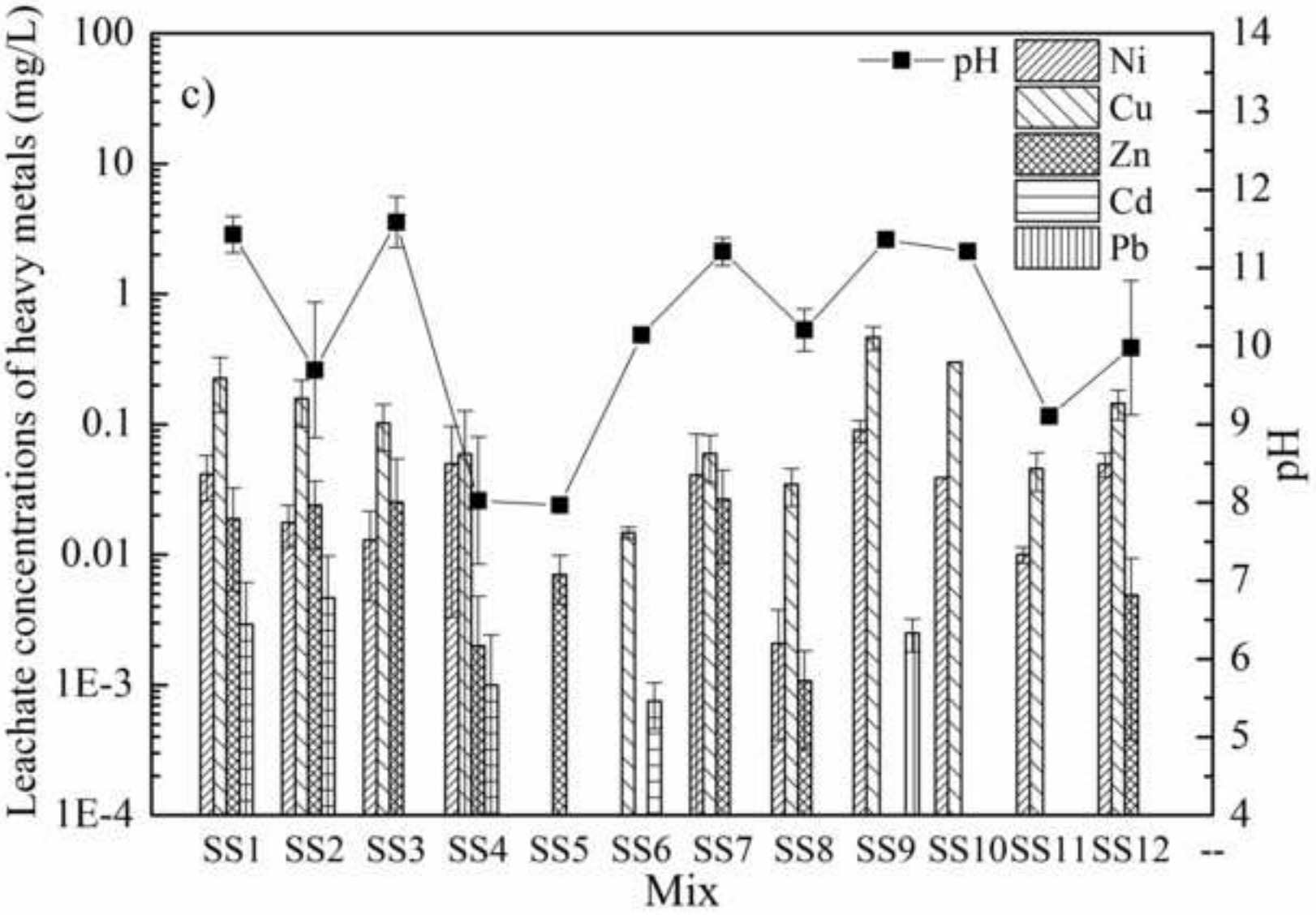

c)
pH
Ni
Cu
Zn
Cd
Pb
Leachate concentrations of heavy metals (mg/L)
100
10
1
0.1
0.01
1E-3
1E-4
14
13
12
11
10
9
8
7
6
5
4
pH
SS1
SS2
SS3
SS4
SS5
SS6
SS7
SS8
SS9
SS10
SS11
SS12
--
Mix

Figure 3

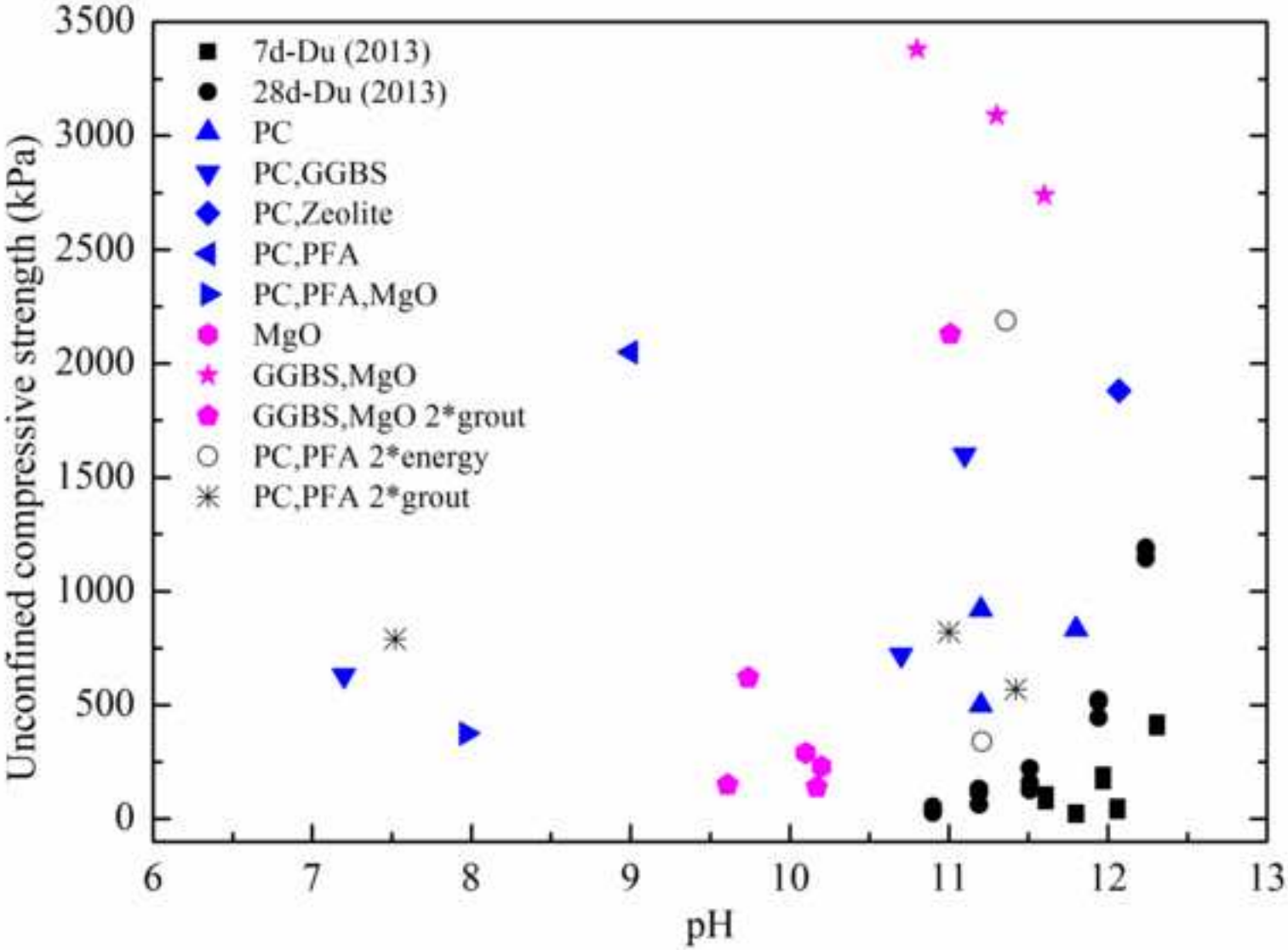

Unconfined compressive strength (kPa)
3500
3000
2500
2000
1500
1000
500
0
6
7
8
9
10
11
12
13
pH
7d-Du (2013)
28d-Du (2013)
PC
PC,GGBS
PC,Zeolite
PC,PFA
PC,PFA,MgO
MgO
GGBS,MgO
GGBS,MgO 2*grout
PC,PFA 2*energy
PC,PFA 2*grout

Figure 4

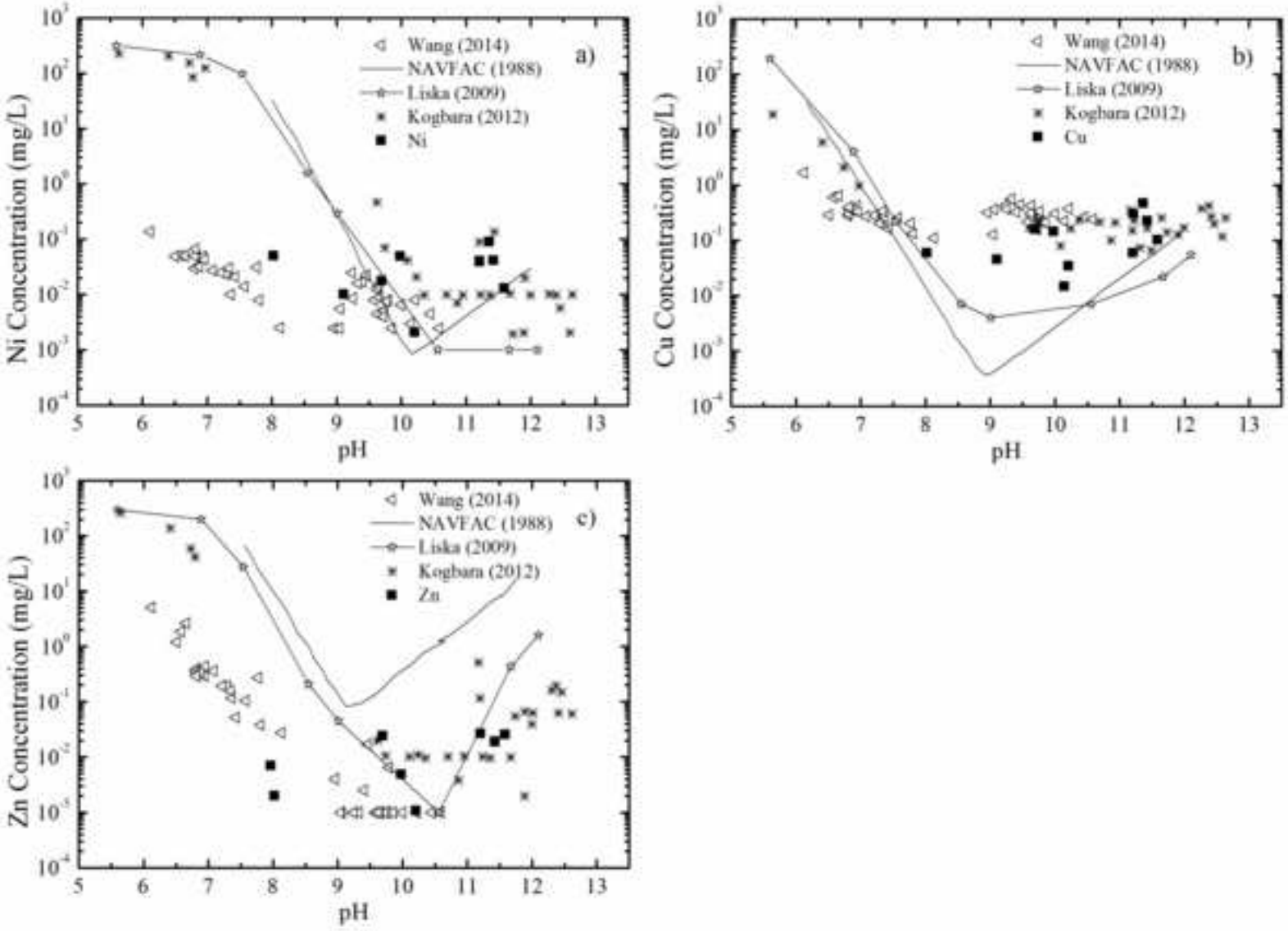
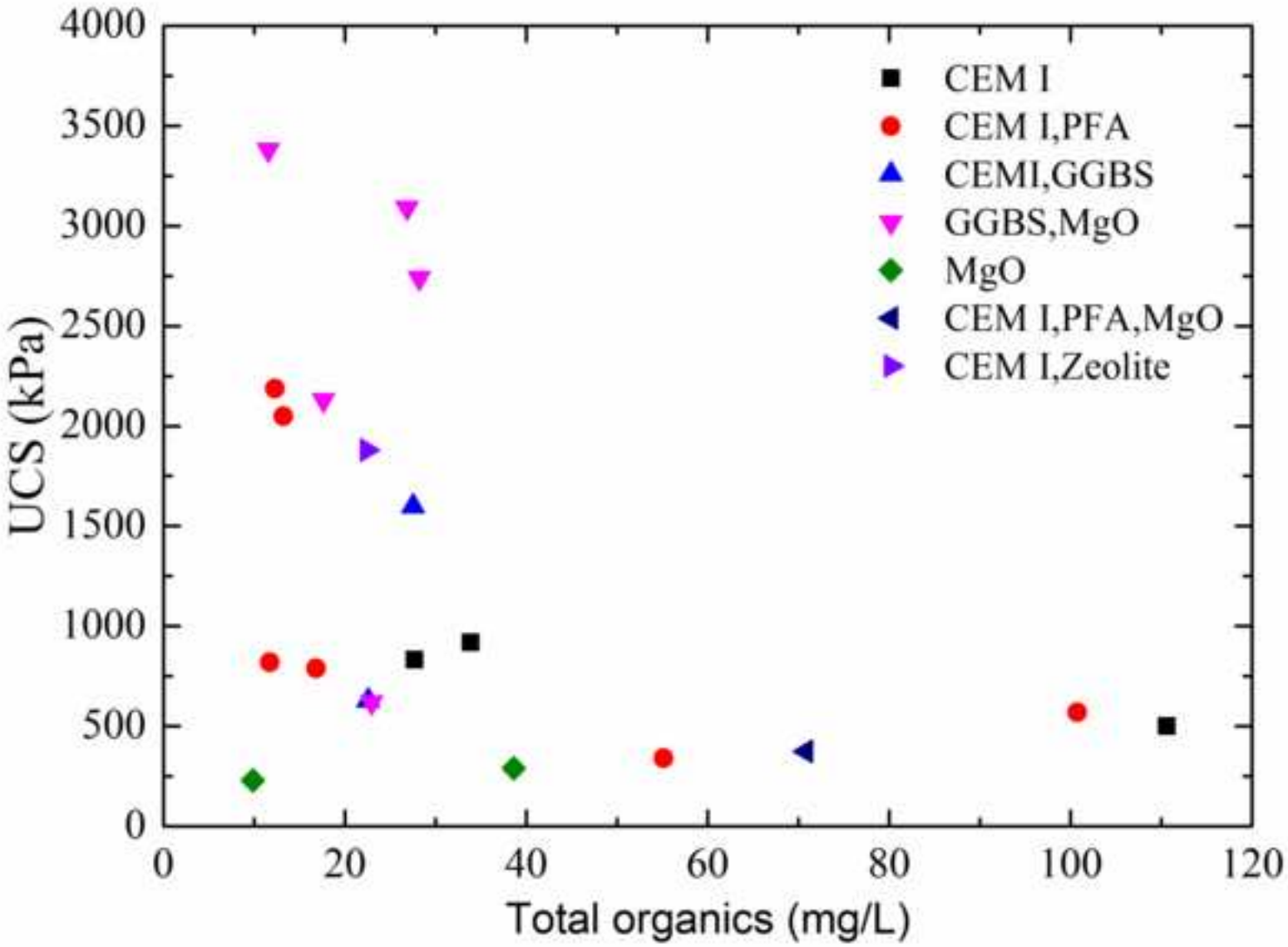

a)
Wang (2014)
NAVFAC (1988)
Liska (2009)
Kogbara (2012)
Ni
Ni Concentration (mg/L)
pH
b)
Wang (2014)
NAVFAC (1988)
Liska (2009)
Kogbara (2012)
Cu
Cu Concentration (mg/L)
pH
c)
Wang (2014)
NAVFAC (1988)
Liska (2009)
Kogbara (2012)
Zn
Zn Concentration (mg/L)
pH

Figure 5


UCS (kPa)
Total organics (mg/L)
0
500
1000
1500
2000
2500
3000
3500
4000
0
20
40
60
80
100
120
CEM I
CEM I,PFA
CEMI,GGBS
GGBS,MgO
MgO
CEM I,PFA,MgO
CEM I,Zeolite