



Three-year performance of in-situ mass stabilised contaminated site soils using MgO-bearing binders



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HIGHLIGHTS

- No apparent degradation of the mass stabilised materials after 3 years' exposure to the field conditions was found.
- The overall range of strength and leachability results at different depth was varied throughout.
- MgO-GGBS blends provided better performances compared to PC and MgO-only mixes in mass stabilised soils.

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ABSTRACT

This paper provides physical and chemical performances of mass stabilised organic and inorganic contaminated site soils using a new group of MgO-bearing binders over 3 years and evaluated the time-dependent performance during the 3 years. This study took place at a contaminated site in Castleford, UK in 2011, where MgO, ground granulated blastfurnace slag (GGBS) and Portland cement (PC) were mixed with the contaminated soils in a dry form using the ALLU mass mixing equipment. Soil cores were retrieved 40-day, 1-year and 3-year after the treatment. The core quality, strength, and the leaching properties were determined via physical observation, unconfined compressive strength (UCS) and batch leaching tests. After 3-year treatment, the UCS values of ALLU mixes were in the range of 50–250 kPa; the leachate concentrations of Cd, Pb, Cu and Zn (except Ni) in all mixes were lower than their drinking water standards; and the leachability of total organics was in the range of 10–105 mg/L. No apparent degradation of the mass stabilised materials after 3 years' exposure to the field conditions was found. MgO-GGBS blends were found able to provide higher strength and less leachability of contaminants compared to PC and MgO-only mixes in mass stabilised soils.

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1. Introduction

Several techniques were applied to treat contaminated site soils over the past few decades, as contaminants can bring great risk to humans and animals [1,2]. Among these techniques cement-based soil mix technologies have been widely employed and investigated by a large number of studies [3–7]. Mass stabilisation as one of these soil mix technologies is effective and environmentally friendly for combining contaminated soil remediation and ground improvement treatments on the same site. It has been implemented for

a few brownfields in the Nordic countries during the past few decades [8].

Portland cement (PC) is the most popular material in soil mix technologies. However, the production of PC is known to be highly energy consuming and releases ~5–10% CO₂ during its production process [9]. Under the drive of sustainability, by-products and novel binders such as ground granulated blastfurnace slag (GGBS) and MgO are drawing people's attention recently [9,10]. It was argued that MgO-based binders have advantages over CaO-based binders and provides additional unique benefits due to their adjustable properties and improved pH buffering capacity [10–12]. The immobilisation mechanism of MgO is mainly through precipitating the hydroxides and encapsulating contaminants into its hydration products (e.g. brucite). The main advantages of using MgO include: 1) the hydration of magnesia gives an equilibrium pH of ~10.5, and hence has a better precipitating action compared with PC/lime as the solubility of many heavy metals is lowest at

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that pH range [13,14]; and 2) brucite is able to accommodate a wide variety of heavy metals and organics by hydrogen bonding or through ion exchange [15,16]. In addition, the use of reactive MgO with GGBS for soil stabilisation has been reported as being able to offer a range of mechanical and chemical advantages over PC or lime-slag blends [1,9,10,17–19]. This is because the hydration products of MgO-GGBS mixes are calcium silicate hydrate (CSH)/magnesium silicate hydrates (MSH) and hydrotalcite-like (Ht) phases. The higher MgO/CaO ratio in these mixes increases the homogeneity of CSH gel structure, leads to a decreased Ca/Si ratio of CSH gel and the increased amount of voluminous Ht phases, which can fill the pores in the matrix more effectively [19,20]. However, limited studies about the effectiveness of mass stabilisation in treating organic and inorganic contaminated soils can be found, with even less field trials studies using MgO-bearing binders, hence assessment of site soils in real projects is essential for the validation of the efficiency of this type of binders in mass stabilisation system.

Apart from the type of binders, time is a significant factor that determines the remediation efficiency of contaminated lands. In the studies of Kogbara et al. [21] and Perera [22], it was found that with the aid of carbonation, the hydration process of PC-bearing binders were accelerated, hence the unconfined compressive strength (UCS) values of all mixes were improved with time at various extents. In most short-term cases (<10years) of stabilisation/solidification (S/S), due to the in-progress hydration, the release of contaminants is more likely to decrease with time [23]. However, Wang et al., [24] studied the leaching performance of S/S treated site soils at 0.2, 2.4, 5 and 17 years. It was reported that slightly higher concentrations of heavy metals were leached at 5 years after treatment compared to these at 0.2 and 17 years using a toxicity characteristic leaching procedure (TCLP) leaching test, and the hydration process of treated samples did not complete at 5 years but fully completed at 17 years. The leachate pH and the further carbonation are the cause of this fact. To date, very limited attention was given to real-life S/S materials, with even less to mass stabilised soils [22,25,26]. Although ground improvement (GI) treatment has been widely applied in the world, the application of MgO-bearing binders in mass stabilisation is a recent development, with very limited studies in the brownfield. For these reasons, it is necessary to understand the time-related performance of mass stabilised contaminated soils using MgO-bearing binders.

The objectives of this trial were to: (1) investigate the 3-year performance of mass stabilised contaminated soil samples at different depths, (2) compare the strength and leaching performance of the soil samples 40-day, 1-year and 3-year after the treatment, and (3) compare MgO-bearing binders and PC in mass stabilised contaminated soils.

2. Site, material and methods

The contaminated site characterisation can be referred to [1,27,28]. Individual GI ALLU mixes were installed in 2011 to form 31 square pits, with a plan area of 1.96 m², and 3–4 m in depth. Soils were pre-wetted, and then a total of 31 different combinations of binders were added in a dry form to the made ground soils. The soil strata consisted of made ground (~5% clay and silt, and ~95% sand) and the groundwater level was reported to vary between 3.2 and 3.9 m below ground level. The water content of the made ground soils was ~25%, while the liquid limit was ~30% and the plastic limit was ~24%. The main contaminants in the made ground tested in 2010 are Pb (38 mg/kg), Zn (99 mg/kg), As (128 mg/kg), Cr (495 mg/kg), Cu (823 mg/kg), Ni (806 mg/kg) and total organics (3605 mg/kg) [9]. The detailed contents of heavy metals and organic contaminants in the original soils at different depths can be seen in

Table 1
Soil-binder constituents in percentage weight (wt%).

Mix denotation	Soil	Binder Contents		
		MgO	GGBS	PC
C5	95	–	–	5
M10	90	10	–	–
M5	95	5	–	–
M2.5	97.5	2.5	–	–
MG10(1:4)	90	2	8	–
MG5(1:4)	95	1	4	–
MG2.5(1:4)	97.5	0.5	2	–
MG10(1:9)	90	1	9	–
MG5(1:9)	95	0.5	4.5	–
MG2.5(1:9)	97.5	0.25	2.25	–

Table 2
Compositions of the binders used in the SMiRT project work (wt%) [33].

Main elements	CEMI	GGBS	MgO
SiO ₂	19.6	36.5	0.9
CaO	64.2	39.5	1.9
Al ₂ O ₃	5.3	12.5	0.1
Fe ₂ O ₃	3.5	0.5	0.8
MgO	1.0	8.5	93.5
SO ₃	–	–	–
K ₂ O	–	0.4	–
Na ₂ O	0.2	0.2	–
TiO ₂	–	0.5	–
Cl	0.04	–	–
LOI	3.1	–	2.78
Free CaO	2.1	–	–

Fig. S1 (See in the online version at DOI: [10.1016/j.jhazmat.2016.07.018](https://doi.org/10.1016/j.jhazmat.2016.07.018)).

Although 31 GI mixes in ALLU mass treatments were applied in the SMiRT project, only these treated by PC (C), MgO (M) and MgO-GGBS (MG) were selected for a detailed study in this paper. The compositions, by weight percentage, of those soil-binder mixes are presented in Table 1. The compositions of the binders used are shown in Table 2. The cores of decreasing diameters of 85, 75, 65 and 55 mm, were obtained and tested at around 1 month after treatment, they were then stored in their plastic cover until reopened 1 year after the treatment as part of this study for further testing. Those containing MgO, together with CEM I for comparison purpose, were sampled again after 3-year treatment from the site for detailed study. The core samples were sealed in $\varnothing 0.1 \times 1.5$ m plastic tubes and sent to the laboratory for testing.

Detailed testing procedure can be found in [1,27]. After 3-year treatment, the undrained shear strength on the soil cores was determined by a handheld shear vane tester and converted to the unconfined compressive strength by a factor of 2 (ASTM D2166). The crushed samples were then subjected to batch leaching according to BS EN 12457-2 (2002). The liquid to solid ratio (L/S) used was 10:1, adding 500 mL of carbonated deionised water in a bottle (pH=5.4) containing 50 g of core sample. After 24 ± 3 h of agitation on a bottle roller, ~10 mL of the leachate solutions was filtered through a 0.45 μ m filter and the pH of which was measured. Finally, a Perkin Elmer inductive couple plasma optical emission spectrometer (ICP-OES) was used to measure the leachate concentrations of heavy metals. The remaining extraction fluid from the batch leaching test was abstracted into a 1000 mL plug-contained conical flask for organic extraction, where 5 mL of 12 mol/L hydrochloric acid was added to speed up the extraction reaction and also acted as a pH buffer. Then, 90 mL of dichloromethane (DCM), serving as the solvent, was divided into 3 equal parts and each was added separately to the flask. After the extraction process, the entire extracted sample was then poured into a container, whose weight was pre-recorded, for DCM evaporation in a fume cupboard. The

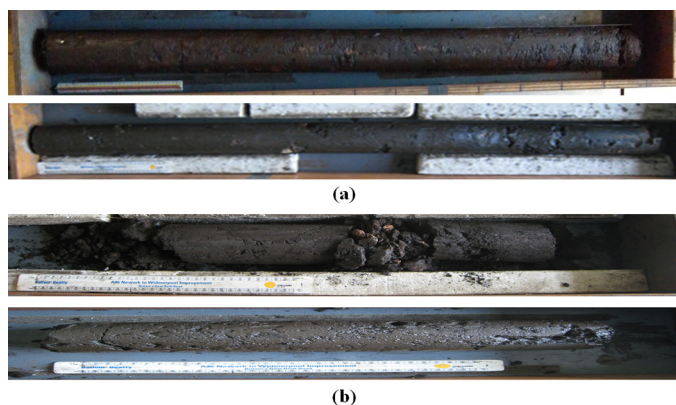


Fig. 1. Images of some of the good and poor quality cores from the ALLU mass stabilisation system: (a) good quality cores and (b) poor quality cores.

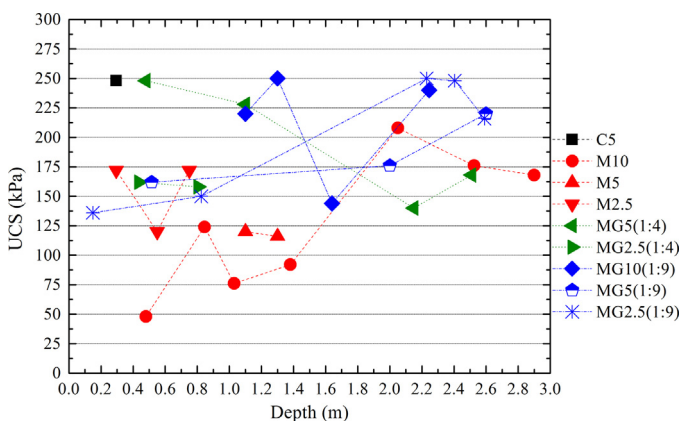


Fig. 2. The UCS values of ALLU mixes at different depths of selected cores after 3-year treatment.

extracted volume and the waste volume were recorded. After about 48 h of evaporation time, the mass of the residual was recorded in order to determine the total organic content leached.

3. Results and discussion

3.1. Core quality

The physical conditions and integrity of the GI ALLU cores were observed 3 years after the treatment, with both good quality intact cores (Fig. 1a) and cores with significant fragmentation and fracturing and with missing sections in Fig. 1b. Different from the results found in [9] that qualities of the S/S treated cores vary significantly depending on the binder compositions, there was no significant difference observed between ALLU mixes taken from different depths and different binder compositions. This may be due to different mixing methods applied in-situ.

3.2. Strength

Fig. 2 shows that the UCS values of these GI ALLU mixes were in the range of 50–250 kPa 3 years after the treatment. Note that no value of MG10 (1:4) is shown due to the poor quality of the cores. The UCS values of mixes showed slightly continued hydration, as shown by the in-situ Dynamic Probe ‘Super Heavy’ (DPSH) testing results obtained from the ALLU mass stabilisation mixes within a couple of months after the installations. The results showed very low strength development of the treated heavily contaminated soils, with corresponding c_u in the range of ~20–50 kPa [29]. Mixes

blended with different contents of MgO-only (M2.5, M5 and M10) produced lower strength compared to other mixes, which is due to the weak binding nature of brucite (the hydration product of MgO). Many research studies observed that blending GGBS with MgO has many advantages such as improved long-term strength and durability [10,18,19,30–32]. This is verified through our field study of S/S, with weak strength at the very beginning and an obviously improved strength after 3-year treatment for all the MgO-GGBS blends [9,33]. A laboratory study of MgO-GGBS in stabilising uncontaminated soils was reported that covering the 7-day, 28-day and 90-day UCS values of the 5% optimum GGBS-MgO mixes were 2–3.9 times higher than that of corresponding PC mixes [18]. In this study, MgO-GGBS blends at ratios of 1:4 and 1:9 were able to produce similar strength compared to PC-only. The discrepancies among these two studies are due to the contaminants in the site soils, for example, Zn has a significant negative effect on the hydration of the cement-based binders, which has been extensively addressed by Du et al. [6,7], and the more complicated site conditions [34]. In addition, values over the full depth cored were varied without any obvious trends, which could be due to the poor mixing and/or varying soil properties.

3.3. Leachate analysis after 3-year treatment

The leaching characteristics of treated soils are presented in Fig. 3 in terms of the leachate pH, the concentrations of leached contaminants and depth 3 years after the treatment. The average values of pH and heavy metals were calculated from duplicate samples of mixes taken from different depths. The average leachate pH values of all these mixes were in general in the range of 6.5–10 (Fig. 3b). M2.5 produced the highest pH value after 3-year treatment, which may be due to the heterogeneity of the contaminated site soils.

The metals considered in this research are Ni, Cu, Zn, Cd and Pb. However, the leachate concentrations of Cd and Pb are lower than their limit of detections (0.5 and 20 $\mu\text{g/L}$ for Cd and Pb, respectively). Therefore, they are not shown in Fig. 3. In Fig. 3a, it is clear that the leachate pH of M2.5 was ~10. Since leachate pH was reported as governing the solubility of Ni hydroxides, the value of ~10 produces the lowest solubility of $\text{Ni}(\text{OH})_2$. Hence, M2.5 leached the lowest Ni and is one of the only two mixes which did not exceed Ni’s drinking water standard (0.02 mg/L) [35]. At a pH of ~7.5, M5 leached the lowest Ni as well, the uneven distribution of Ni in the made ground may be the cause of the fact. This is followed by C5, the average leachate concentration of Ni of which is ~0.1 mg/L. The result agrees well with the findings in [4] that the leachate concentration of Ni at pH 6–9 was lower than its solubility limit. MgO-GGBS blends did not show any advantages over other blends in immobilising Ni. This is due to the pH values of these mixes (at ~6.5) were lower than the pH values of C5, M5 and M2.5, which producing a higher solubility of $\text{Ni}(\text{OH})_2$ [27]. Fig. 3b&c show that the leachate concentrations of Cu and Zn in all mixes are lower than their drinking water standard (2 mg/L and 3 mg/L respectively). This agrees well with the relationship of pH, Cu and Zn, and solubility of their hydroxides [27].

The variation of metals’ concentrations leached from different depths is varied and up to more than 1 order of magnitude. In addition, it was found that all mixes (except few mixes in Fig. 3b) leached higher concentrations of these three metals from deeper soils, indicating that the mixing of the deeper soils is not as good as that of the surface soils on the site. In addition, it is noted that extensive testing is necessary and it is acknowledged that a large body of data is needed to arrive at more firm conclusions.

Fig. 4 shows that the leachability of the total organics was in the range of 10–105 mg/L, suggesting big variability in the contaminant level and binder composition. As can be found from the

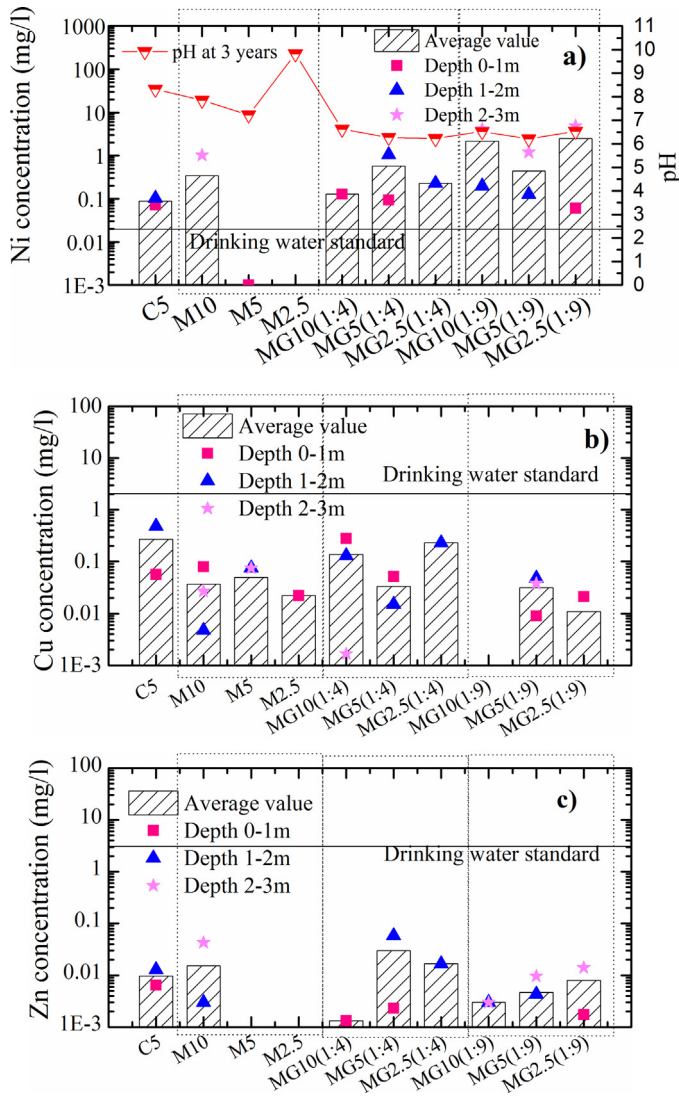


Fig. 3. The leachate concentrations of metals at different depths of selected cores after 3-year treatment.

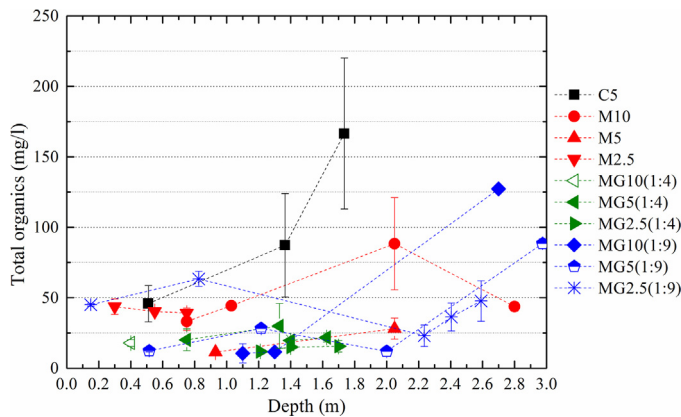


Fig. 4. The leachate concentrations of total organics at different depths of selected cores after 3-year treatment.

distribution of the leachability of total organics along the depth, in general, the adsorption ability of binders for organics is in the order of MgO-GGBS (1:4) > MgO > PC. The leachability of the total organics stabilised by MgO-GGBS (1:9) were generally <50 mg/L, with two

Table 3
Average UCS results of GI ALLU mixes at 40 days, 1 year and 3 years.

Mix	UCS-40 days (kPa)	UCS-1 year (kPa)	UCS-3 year (kPa)
C5	263 ± 21	310 ± 17	248
M10	N/T ^a	289.7 ± 83	127.4 ± 24
M5	N/T ^a	N/A ^a	118.7 ± 2
M2.5	N/T ^a	108.5 ± 30	154.7 ± 21
MG10 (1:4)	936 ± 347.80	N/A ^a	N/A ^a
MG5 (1:4)	220 ± 57.29	N/A ^a	193.6 ± 29
MG2.5(1:4)	N/T ^a	N/A ^a	160 ± 3
MG10 (1:9)	192 ± 59.40	N/A ^a	213.5 ± 28
MG5(1:9)	91 ± 12.50	N/A ^a	184.6 ± 21
MG2.5(1:9)	N/T ^a	N/A ^a	202 ± 36

^a N/T: Too weak to test. N/A: Not available.

points at a depth of 2.6–3 m leached higher concentrations of total organics. This may due to the uneven distribution of organic compounds in-situ. The higher immobilisation degree of organics by the application of MgO and MgO-GGBS blends is due to the structure of brucite produced by MgO and voluminous hydrotalcite-like phases produced by MgO-GGBS blends. They are able to accommodate a wide variety of extraneous anions, cations as well as organic compounds by hydrogen bonding etc, thus providing an excellent host for contaminants [14].

A few conclusions can be drawn from these figures: (a) In general, under different depths, the average strength (UCS) of binders obtained from Fig. 2 is in the order of MgO-GGBS ≈ PC > MgO; (b) the average leachability of binders for organic contaminants is in the order of MgO-GGBS (1:4) > MgO > PC; (c) the average leachability of binders for inorganics is in the order of MgO-GGBS (1:4 and 1:9) > MgO > PC; (d) no significant trend can be draw along the depth among all mixes.

3.4. Time-dependent performance of different binders in mass stabilisation

In this study, GI ALLU samples tested 40-day, 1-year and 3-year after the treatment were used to study the time-dependent performance of mass stabilised contaminated soils. It is meaningful to mention that the samples tested after 1-year treatment were different from these after 40-day and 3-year treatment (window sampled from the site directly), as they were stored outdoor covered with hessian cloth in the Engineering Department, University of Cambridge (window sampling is the process uses either drop weight or hydraulic hammer to drive 1 m or 2 m long sample tubes into the ground. The sample tubes have open slots or “windows” along their length to allow for logging and sampling).

Table 3 shows average UCS results of GI ALLU mixes up to 3 years. The deviation of UCS for different binder mixes was in the range of 8–21 kPa after 40-day treatment, 12–93 kPa after 1-year treatment and 2–36 kPa after 3-year treatment. In Table 3, The UCS value of C5 is ~263 kPa 40-day after the treatment, the value of which increased to 310 kPa after 1-year treatment and was ~248 kPa after 3-year treatment. The higher strength of C5 after 1-year treatment is due to its lower water content (stored ex-situ). M10, M5, M2.5 and MG2.5 (1:4&1:9) were too weak to be tested at 40 days. For MgO-only mixes, this was due to the slow hydration of MgO and the weak binding nature of brucite. The low strength of mixes MG2.5 (1:4&1:9) at 40 days’ treatment is due to the very low binder dosage of 2.5% adopted in these two mixes, and the relatively long duration that needed for the strength development of MG2.5 (1:4&1:9) caused by the nature of MgO as a weak alkali activator of GGBS. After 1-year treatment, the strength of 10% MgO-only mixes was ~300 kPa, which is ~3 times higher than the 2.5% MgO-only mixes. Furthermore, in spite of the common hydration products such as CSH, Ht produced during the curing process assist

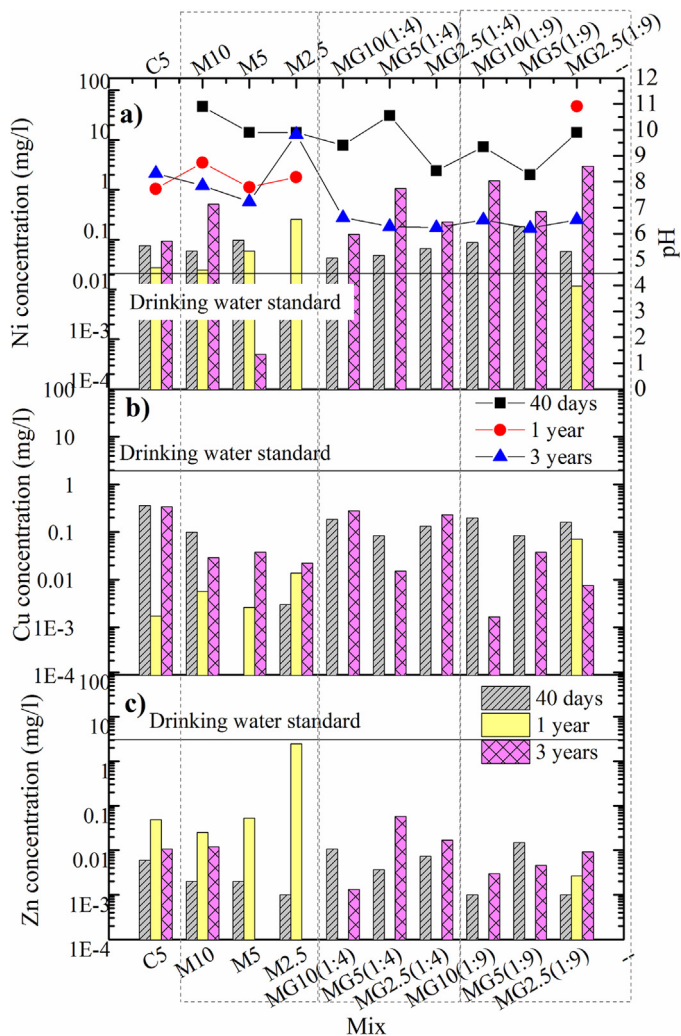


Fig. 5. Average results of leachate pH and leachability of heavy metals in GI ALLU mixes 40-day, 1-year and 3-year after the treatment.

to produce a finer microstructure [20]. Hence, the strength development of MgO-GGBS blends after 40 days and 3 years of curing were found higher than MgO-only mixes. In general, the strength of all mixes did not change significantly with time. This may be due to the high moisture content of raw soils (before mixing soils were pre-wetted) that the hydration degrees of most binders were very high after 40 days. High water to cement ratio also leads to a reduction of strength development of these mixes [36].

The average leachate pH values and concentrations of Cu, Ni and Zn in the leachates as a function of time are displayed in Fig. 5. In addition, the leachability of heavy metals in MG-10(1:9), MG-5(1:9) and all MG(1:4) mixes after 1-year treatment did not display in Fig. 5 due to there were no data of these mixes available. As can be seen, the pH values for all mixes after 40-day treatment were in the range of ~9.8–11.2, and these after 1-year treatment displayed lower average values but with a broader range at ~7.7–10.9, while the average of these after 3-year treatment were even lower at ~6.5–10. Comparing the pH values of 40-day with these 3-year, decreased pH values were found in all mixes after 3-year treatment. This is due to the in-situ atmospheric carbonation, which has been extensively explored by previous study reported in Du et al. [6].

Comparing the leachate concentrations of heavy metals with their drinking water standard individually, it can be found that the majority mixes exceeded Ni's standard limit during the whole testing period. Although the Ni concentration of most mixes declined

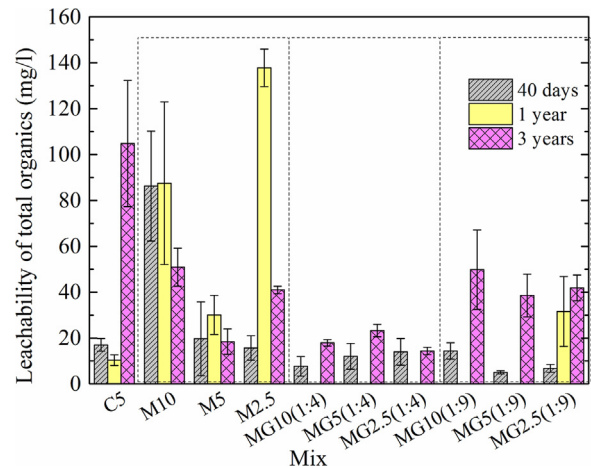


Fig. 6. Leachability of total organics in GI ALLU mixes 40-day, 1-year and 3-year after the treatment.

slightly from 40 days to 1 year, they were found even higher in some mixes 3 years after the treatment. The decreased concentrations of leached Ni from 40 days to 1 year are due to the different curing environment. When comparing the samples cured in-situ, the higher leachability after 3-year treatment compared to 40 days agrees well with the relation of Ni, pH and solubility curve of Ni hydroxide studied in [1]. The leachate concentrations of Cu and Zn in all mixes were found well below their drinking water standards at different time points. Although atmospheric carbonation and complex site environment can degrade treated soils and increase the release of heavy metals to the environment with time, the leachate concentrations of metals in most mixes were in the same magnitude at 3 time points.

The leachability of total organics of GI ALLU samples 40-day, 1-year and 3-year after the treatment is shown in Fig. 6. It shows that the leachability of total organics of majority mixes increase with time. Binding mechanisms between binders and organic compounds are physical entrapment in the produced matrix and sorption onto the surface of hydration products rather than chemical reactions [37,38]. Thus the leachability of organics was not improved with the hydration process during the past 3 years. In addition, the curing condition such as rain flush (destroy the structure of produced matrix), atmospheric CO₂ (lower soils' pH) may be the main cause of this fact.

MgO-GGBS blends at a ratio of 1:4 were found to be the most effective mixes in immobilising organics 40-day and 3-year after the treatment. It was followed by MgO-GGBS blends at a ratio of 1:9. This is because the hydration products of MgO-GGBS mixes are CSH/MSH and Ht, which are more voluminous and can fill the pores in the matrix more effectively [20]. Hence, it is considered that more organic compounds are supposed to be trapped in the reaction products of MgO-GGBS mixes. In addition, the results of MgO-bearing mixes show that higher binder content did not result in a lower leachability of total organics. Since the leachability of total organics was determined by a large number of factors such as the contaminants level, soil type etc., this result indicates that an increase of binder content among these mixes (from 2.5% to 10%) may not be as significant as factors mentioned above in improving the effectiveness of immobilising organics. According to the relationship studied between UCS and leachability of total organics in a previous study [1], when the leachate concentrations of organic pollutants are >30 mg/L, high strengths of S/S treated samples cannot be achieved. High organic contaminants are able to inhibit the hydration of binders [37] and produce significant micro- and macrostructural changes to their products may be the cause of this

result. The results of UCS tests and the leachability of heavy metals and organics at 3 time points indicate that there is no degradation of the GI ALLU mixes up to 3 years. This agrees well with the finding of Jin et al. [9] that no degradation of S/S materials was found after 3 years' exposure to the field conditions.

4. Conclusions

The time-related performance of mass stabilised soils was evaluated in terms of their physical and leaching properties. It also investigates the application of the innovative MgO-bearing binders in mass stabilisation for the first time. The results showed that MgO-bearing binders are very promising binders which can be used as a replacement of PC in this field. The main findings of this study are summarized as follows: 1) No degradation of mass stabilised materials was found after 3 years' exposure to the field conditions, while the full range of high to poor quality cores were observed at both sampling time points (40 days and 3 years); 2) The overall range of strength and leachability results at different depths was varied throughout, suggesting a complicated distribution of contaminants in soils; 3) After 3-year treatment, regardless of the depth, the strength of binders is in the order of MgO-GGBS \approx PC > MgO, while the leachability of binders for contaminants is in the order of MgO-GGBS > MgO > PC; 4) MgO is found to be very effective in immobilising both heavy metals and organics, however, it showed weak mechanical performance. MgO-GGBS blends at a ratio of 1:4 were found to be the most effective GI ALLU mixes in immobilising organics; 5) High moisture content in-situ has adverse effect on the efficiency of mass stabilisation system; 6) The leachate concentrations of Cu, Pb, Cd and Zn meet their drinking water standard, but the concentrations of Ni in some mixes are above its drinking water standard regardless of time. And it was found that the leachability of organics was not improved with the hydration process during the past 3 years.

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