Time-dependent performance of soil mix technology stabilised/ solidified contaminated site soils

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Highlights:

- The physical and chemical performances of most mixes were improved with time.

- Modified clays are efficient in immoblising Ni.

- A small amount of organo clay is able to improve the strength of samples.

- M (MgO) and MG (MgO+GGBS) are efficient in immoblising heavy metals.

- MgO based blends have showing outstanding effect on treating organic pollutants.

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Novelty Statement: The objective of this paper is to investigate the time-related performances of different binders (especially modified clays) in a heavy metals and organic contaminated site at 28 days and 1.5-year after treatment. Testing the time-related performances of these novel binders and addictives are required to expand the boundaries of the soil mixing technology and are necessary in validating the effectiveness of this remediation process.

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ABSTRACT: This paper presents the strength and leaching performance of stabilised/solidified organic and inorganic contaminated site soil as a function of time and the effectiveness of modified clays applied in this project. Field trials of deep soil mixing application of stabilisation/solidification (S/S) were performed at a site in Castleford in 2011. A number of binders and addictives were applied in this project including Portland cement (PC), ground granulated blastfurnace slag (GGBS), pulverised fuel ash (PFA), MgO and modified clays. Field trial samples were subjected to unconfined compressive strength (UCS), BS CN 12457 batch leaching test and the extraction of total organics at 28 days and 1.5 years after treatment. The results of UCS test show that the average strength values of mixes increased from 0-3250 kPa at 28 days to 250-4250 kPa at 1.5 years curing time. The BS EN 12457 leachate concentrations of all metals were well below their drinking water standard, except Ni in some mixes exceed its drinking water standard at 0.02 mg/l, suggesting that due to

varied nature of binders, not all of them have the same efficiency in treating contaminated soil. The average leachate concentrations of total organics were in the range of 20-160 mg/l at 28 days after treatment and reduced to 18-140 mg/l at 1.5 years. In addition, organo clay (OC)/inorgano-organo clay (IOC) slurries used in this field trial were found to have a negative effect on the strength development, but were very effective in immobilising heavy metals. The study also illustrates that the surfactants used to modify bentonite in this field trail were not suitable for the major organic pollutants exist in the site soil in this project.

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

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Keywords: Field trials; Modified clay; Novel binders; Soil stabilisation

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1. INTRODUCTION

Effectively treating organic and inorganic contaminated soil has been found to be a big challenge due to their nature and transport mechanisms in a soil environment [1]. Compared with existing treatment options, such as soil washing, biological methods, and disposal to landfill [2], soil mixing technology is a cost-effective, versatile, and low risk method for the implementation of a range of in-situ remediation treatments [3,4]. Stabilisation/solidification (S/S) as an application of soil mix technology has specific advantages relating to cost and environmentally friendliness [5]. The most popular materials in S/S are conventional binders such as Portland cement (PC) and lime [6], and some industrial by-products such as pulverised fuel ash (PFA) and ground granulated blastfurnace slag (GGBS) [7]. Recently, attention has been put on novel binders such as MgO, and modified clays such as organo clay (OC), Inorgano-organo clay (IOC) [7, 8, 9]. OC is reported as being able to adsorb more organic pollutants compared to raw clays [8, 10]. The modifications of bentonite to produce OC include oxide pillaring which modifies the layered crystalline inorganic compound to produce a material with micro and meso porosity, and organic surfactant modification through cation exchange with alkylammonium ions. The combination of these two methods forms IOC minerals, which can treat inorganics and organics simultaneously [11, 12]. However, studies of these binders, modified bentonites, have been mainly restricted to laboratory investigations [13]. Testing the performance of these novel binders and addictives is required to expand the boundaries of the soil mixing technology. In addition, whether a treatment technique is successful or not depends on its performance during its end-use expected lifetime [13]. Wang et al. [4, 25] studied the leaching performance of S/S treated samples at 0.2, 2.4, 5and 17 years, and found that lower concentration of heavy metals leached at 17 years after treatment than this at 5 years, and found that the hydration process of treated samples did not completed at 5 years but fully completed at 17 years. Roy and

Cartledge [37] studied the long term behaviour of PC treated sludge waste and found that the appearance of the principal Cu-bearing phase (CuO·3H$_2$O) was both time and concentration dependent. Subtle changes of Cu in the microchemistry occurred over time. Hence, an assessment of the time-dependent performance of novel binders and modified clays in treating organic and inorganically polluted site soil is necessary in validating the effectiveness of this remediation process.

The objectives of this trial were to: 1) compare the strength and leaching performance of S/S treated site soil samples at 28 days and 1.5 years, 2) enable a better understanding of binder-contaminant interaction mechanisms, and 3) assess the application of OC/IOC in treating organic and inorganic contaminated soil.

2. SITE, MATERIAL AND METHODS

Project SMiRT (Soil Mix Remediation Technology), was the largest contaminated land remediation project funded by the Technology Strategy Board. It involved collaboration with 16 industrial partners, over a four-year period which started in October 2007 and finished in September 2011 [3]. Soil treatment by S/S took place at a site in Castleford, Yorkshire in May 2011, as shown in Fig. 1. A triple auger system was applied which mixed contaminated soil with a range of binder blends consisting of PC, PFA, GGBS, MgO, Zeolite, OC and IOCs.

The geology at the site consists of top soil (0.1-0.35 m), made ground (0.35-4.5 m) (consisting of black sand and/or silt containing fragments of plastic, concrete and wood), silts and clays (4-6m) and sand and gravel (6-8m). Natural drift deposits were found in the silts and clays zone as well as the sand and gravel zone. The groundwater level varies between 3.2 and 3.9 m below ground level [14]. The water content of the soil is ~25%, the liquid limit is ~30% and the plastic limit is ~24%. Due to historic disposal of waste materials, significant

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contamination was anticipated. Soil and groundwater samples were forwarded to Alcontrol Geochem in Chester (a URS approved laboratory) for chemical analysis at regular intervals during the fieldwork period [14]. Contaminants and their concentrations in the soil are listed in Table 1.

Although a total of 24 soil-grout compositions were applied at the site, only 14 of these mixes were selected for a detailed study in this paper. The layout of the 24 installations can be found in Fig. 1. PC (P), PFA (F), GGBS (G), MgO (M), OC and IOCs were materials used in the mixes, where each mixes were named after these materials' abbreviations. The 14 mixes were divided into 4 groups based on binder compositions for purpose of comparison, as shown in Table 2. The materials (excluding IOCs) used in this project were bought from material supply companies. The PC used in this project is CEM I. Granular OC used in the study was obtained from Amcol Minerals Europe Ltd (with di(hydrogenated tallow) dimethyl ammonium chloride and di(hydrogenated tallow) methyl ammonium chloride surfactants on sodium bentonite clay) and IOCs were prepared in the laboratory with their compositions detailed in Table 3. The binder ratios used in this project were based on a preliminary laboratory study. In Table 2, PG, PF, PFM and P in group 1 are PC based, the slurry content of which is 15%; P-OC, P-2IOC1, P-IOC1, P-IOC3 in group 2 are 15 % PC with different types of modified clays; PG-IOC3, PF-IOC3, PFM-IOC3, MG-IOC3 in group 3 are IOC3 based mixes, with 15% binders + 7.5% extra IOC3 slurry; M, MG in group 4 are MgO based binders.

Sampling took place at 28 days after treatment, 1 m length cores were collected in sealed plastic tubes to 4 m depth. The diameter of the cores decreased with depth from 0-1m: 90mm, 1-2m: 80mm, 2-3m: 70mm and 3-4m: 55mm. After testing at 28 days, samples were cured in their original sealed plastic tubes in a temperature controlled laboratory at ~20˚C. The relative humidity of the lab is ~50%.

A saw cutter was used to trim cores into sections each with a length equal to twice the diameter of the core [15-17]. The ends of the sections were made flat to within ± 0.05 mm (see Fig. 2). The samples were tested at 28 days and again at 1.5 years. A core length and trimmed section are illustrated in Fig. 2. The samples were subject to UCS testing in triplicate based on ASTM D4219-08 test method using a Uniframe 70-T0108/E loading frame. The crushed samples were then screened before subjected to batch leaching following BS 12457-2 [18]. 50 g of crushed core sample with particle sizes between 1 and 4 mm was added to 500 ml of carbonated deionised water (pH=5.4) to achieve a liquid to solid ratio (L/S) of 10:1. After 24 hours of agitation, the leachate solutions were filtered through a 0.45 μm filter and tested for pH by a pH meter (EUTECH pH510) and the concentration of the heavy metals was analysed using a Perkin Elmer 7000 inductively coupled 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 to act 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 process three times, the DCM with the extracted organics was poured into a container, and evaporated in a fume cupboard. After ~48 hours, the mass of the residual organic mass was recorded [11]. All experiments were carried out at least in duplicate, in order to reduce the random error.

3. RESULTS AND DISCUSSION

3.1 UCS

Fig. 3 shows the average UCS values of triplicate samples for each mix at 28 days and 1.5 years. The deviation of UCS values for different binder mixes was in the range of 10-149 kPa.

As can be seen from Fig. 3, the strength of PF-IOC3, MG-IOC3, M and PFM-IOC3 were too weak to be tested at 28 days after treatment, but the strength of these mixes increased significantly at 1.5 years. The strength of all group 1 mixes increased with time, apart from the strength of PFM which was found to be weaker at 1.5 years than at 28 days. This is due to the cement content in PFM being very low (1.5%), MgO does not react with PC and PFA. The weaker strength of PFM at 1.5 years suggested that the mixing conditions affect the strength development much more severely than the hydration between a small amount of PC and PFA during the past 1.5 years. It was found that PFM produced the weakest strength at both time points because: 1) of the PC content was very low, 2) the chemical composition of PFA is somewhat different from PC and needs both lime and water to hydrate, and 3) the hydration product of MgO (magnesium hydroxide) is relatively weak compared to the calcium silicate hydrates (C-S-H) formed in PC [19]. PG produced the highest UCS value at ~ 1300 kPa in group 1 at 28 days after treatment and high value (~1600 kPa) at 1.5 years. While the UCS values of P at 28 days and 1.5 years are ~700 kPa and ~ 800 kPa, respectively. This agrees well with the study by Kogbara and Al-Tabbaa [20] that a well-proportioned mix of GGBS+PC has higher early and later strengths than PC (CEM I). This is because $Ca(OH)_2$ in PC is able to react with SiO_2, Al_2O_3 contained in GGBS and produce more C-S-H gels. PF produced the highest UCS values at 2635 kPa in group 1 at 1.5 years after treatment, however, the value of which at 28 days is low at ~500 kPa. This is because the strength development speed of PFA grout is lower than PC grout, but since PFA also contains some SiO_2, Al_2O_3, PF is able to produce a much higher later strength than P [5].

In group 2, at both time points, PC mixed with a small amount of OC (3.2%) produced 2 or 3 times the strength of PC alone or PC with different IOCs. This may be due to 1) OC has good adsorption of heavy metals and better adsorption of organic compounds than the IOCs used in this study, (Table 4 and Fig. 4) therefore less organic pollutants were able to be inhibit the

hydration process; 2) Bayat et al. [21] and Teerawattanasuk and Voottipruex [22] reported that the shear and yield strength increased when the content of bentonite increased, which can act as plastic fines; 3) the IOCs slurries increased the water content, which decreased the strength development [23]. When comparing P-2IOC1 with P-IOC1, it was found that doubling the IOC1 content reduced the strength to half at 28 days. It also was found that P-2IOC1's strength increasement with time is significant.

At 28 days, most of mixes in group 3 were too weak to be tested. At 1.5 years the strength of all mixes in group 3 increased due to the continued hydration process. It was found that extra IOC3 added in this group resulted in decreased strength development, as can be seen from PG-IOC3, PF-IOC3, PFM-IOC3 in group 3 and PG, PF, PFM in group 1, and from MG-IOC3 and MG at 28 days and at 1.5 years in Fig. 3. This is due to the adverse effect of higher water to binder ratio [23] as discussed above.

In group 4, the strength value of MG at 28 days at ~3250 kPa when other mixes in this group were too weak to be tested. The strength of MG continued to increase to ~ 4200 kPa at 1.5 years. MG was able to produce the highest UCS values among all mixes at both time points in this study. This is because MgO can activate GGBS and produce hydration products such as C-S-H, magnesium silicate hydrates (M-S-H) and hydrotalcite ($Mg_6Al_2CO_3(OH)_{16} \cdot 4H_2O$)-like phases (Ht), which contribute to strength development [24]. In addition, the 90% GGBS used in MG can reduce the carbon footprint significantly. On the contrary, M in this group produced low strength as the hydration product of MgO ($Mg(OH)_2$) is very weak.

3.2 Leachate pH and leachability of the heavy metals

Table 4 details the average leachate pH values and average leachate concentrations of Cu, Ni, Pb and Zn in 4 groups at 28 days and 1.5 years, with a maximum variation of ±0.097 mg/l. A decrease of pH values with time can be found among mixes in all groups. The pH at 28 days

was in the range of 11.1-13.1, the values of which reduced to 8-12.2 at 1.5 years, due to carbonation or reactions with natural leachants [4, 25, 26]. This agrees well with previous S/S pH values as a function of time reported by Wang et al. [4], who found that pH values of S/S treated site samples decreased from ~10.5 at 0.2 years to ~7.6 at 2.4 years and ~7 at 5 years and levelled off in the next 12 years.

From Table 4, it can be seen that the leachate concentrations of Cu, Pb and Zn in all mixes were below the drinking water standard while the leachate concentrations of Ni in some mixes exceeded its drinking water standard (0.02 mg/l). Since cores were sampled after 28 days' in-site treatment and cured in the lab afterwards, the tested concentrations of heavy metals were supposed to be higher than the actual field samples. This is because these samples did not suffer from the rain wash. For most mixes, it was found that the leachate concentrations of heavy metals declined as a function of time. Specifically, group 1 showed limited efficiency in treating Ni, with the leachate concentration of Ni in most mixes greater than its drinking water standard. This can be related to the higher leachate pH (at ~11-12) given by the hydration products of PC. The leachate concentrations of Ni reported by Wang et al. [4], were mainly above its solubility when pH > 11. Group 2 performed slightly better than groups 1 in terms of Ni immobilisation, with the leachate concentration of Ni in some of these mixes slightly higher than its drinking water standard, indicating the advantage of adding OC/IOCs.

In the presence of IOC3 and MgO, groups 3 and 4 performed better than the other 2 groups in immobilising Ni. This is because the non-ionic surfactant PPG in IOC3 increases the interlayer basal spacing of bentonite, which is sufficient for the removal of metal ions [27]. The findings also agree with studies by De Leon et al. [28] and Guerra et al. [29] that pillared bentonites have improved efficiency for metal removal. Comparing group 3 with group 2, it

is obvious that IOC3 performed better than other modified clays for the immobilisation of heavy metals.

In group 4, the equilibrium pH of $Mg(OH)_2$ is ~10.5, the value producing the lowest solubility of $Ni(OH)_2$, hence Ni can be effectively immobilised [4]. Additionally, $Mg(OH)_2$ has a layered structure which can adsorb heavy metals onto the surface of $Mg(OH)_2$ or encapsulate heavy metals in its structure [19]. Furthermore, MG (with 10% MgO) proved to have much better buffering capacity than the PC based group. The leachate pH values, given by the high content of hydrotalcite-like phases in the hydration products of MG, were near the minimal solubility of most heavy metals [19].

3.3 The leachability of total organics

As can be seen from Fig. 4, the leachate concentrations of total organics in most mixes decreased in various degrees as a function of time. The deviation of concentrations of leached total organics was 1.9-72 mg/l. Group 2 leached the highest concentrations of total organics, followed by group 3 and group 1. Group 4, which leached the lowest concentrations of total organics, was observed as the best group in stabilising organic compounds.

In group 1, except PFM, the leachate concentrations of total organics in all mixes declined with time. This was due to further hydration taking place during the time intervals. PFM leached more total organics at 1.5 years, due to mixing conditions playing a major role in not only the strength development as mentioned above but also the leachability of total organics, while the hydration between a small amount of PC and PFA was not as severe as the mixing condition. Among the four PC based mixes, PF leached less organics: <60 mg/l at 28 days and <20 mg/l at 1.5 years, related to the high adsorption ability of PFA which has been previously studied a lot [30].

In group 2, high amounts of total organics were leached from samples at both time points suggesting that the hydration process of PC was inhibited by the extra water in IOCs slurry. When comparing the leached concentrations of total organics at 28 days and 1.5 years, a slight increase was found in the average concentrations of leached total organics of P-OC and P-2IOC1, while a slight decrease took place in P-IOC1 and P-IOC3. When considering the deviation of triplicate samples, no obvious trend can be found in group 1. The same situation was found in group 3, as can be expected by a high level of heterogeneity in the site soil.

After comparison among P and all the mixes in group 2, it is clear that mixing OC/IOCs with PC together is not very effective in immobilising organics. Although the intercalation of OC/IOCs was extended to some level, the layer structure factor was pointed out by Jiang and Zeng [10] as not significant to influence the organic pollutants adsorption but the surfactant modifiers. This deduced that the surfactants used to modify bentonite in this study are not suitable for the major organic pollutants in the site soil. This was proved by Ouellet-Plamondon et al. [27], who claimed that although PPG used in IOC3 can increase swelling of montmorillonite at lower concentration, the intercalation remained in the crystalline region, hence only small hydrocarbons can be removed by PPG modified bentonite.

When comparing PG, PF, PFM, MG with mixes in group 3 in sequence, IOC3 mixed with different binders showed adverse impact on immobilising organic compounds, suggesting that IOC3 is not effective in treating big molecular organics. Further studies of suitable surfactants for treating voluminous organic contaminants are needed.

In group 4, the average leachate concentrations of total organics in all mixes at 28 days (at ~80 mg/l) were ~3 times higher than these at 1.5 years (at ~25 mg/l), with the lowest deviation value (< 10 mg/l), which indicates that the remediation effectiveness of group 4 in treating organic pollutants is more evident. The three most intense interplanar spacings (D) of brucite are 0.2365 nm, 0.477 nm and 0.1794 nm [31], and it was reported by Jordan and

Rammensee [32] that the OH-Mg-OH layer parallel to (001) with a height of ~ 0.43 nm. At the same time, the d-spacing of PPG modified bentonite was reported Ouellet-Plamondon et al. [27] that PPG increased the interlayer spacing of bentonite to 1.83 nm at 2 cation exchange capacity (CEC). This indicates that using the layer structure of brucite to adsorb organics into its layers is not the main stabilisation mechanism of brucite. Since seldom studies about the binding mechanism between brucite and organics can be found, a hypothesis was deduced that it involves simple physical adsorption alone with some chemical bonding eg. cation exchange, complexing of the hydroxide with the organics [33]. In addition, the hydration products of MG are C-S-H, M-S-H and Ht. Compared to C-S-H, hydrotalcite-like phases were found more voluminous, providing a bigger surface to trap organics. Hydrotalcite-like phases was also reported as having structural charge, which may immobilise more organics with charges [34].

4. CONCLUSIONS

This paper investigates the strength and leaching performance of stabilised/solidified organic and inorganic contaminated site soil as a function of time, and the application of modified clays in the field. The main findings of this study are summarised as follows:

- The average UCS values of mixes in 4 groups increase from 28 days curing time to 1.5 years curing time, with most of them exceed the design values of 350 kPa used in the UK.

- The leachate pH, the leachate concentrations of heavy metals and total organics of all mixes in this study decrease with time.

- The leachate concentrations of Cu, Pb and Zn meet their drinking water standard, but the concentrations of Ni in some mixes are above the drinking water standard at both time points.

- A small amount of Organo clay is able to improve the strength of samples.

- Modified clays, especially Inorgano-organo clay 3, are efficient in immoblising Ni, but not very effective in stabilising big molecular organics and strength development. Further studies of modified clays which are suitable for treating large organic contaminants are needed.

- M (MgO) and MG (MgO+GGBS) are efficient in immoblising Ni and have showing outstanding effect on treating organic pollutants.

- MG (MgO+GGBS, used 90% by-product) had better physical and chemical performances compared to other mixes at 28 days and 1.5 years.

- PFA is effective in decreasing the leachability of organics but it increases the leachability of heavy metals.

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List of Figure Captions

Figure 1. Plan of field trials treatment.

Figure 2. Description of SMiRT site cores and a trimmed core at 1.5 years.

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Table

Table 1. Soil contaminant concentrations [35].

Metals or Organic contaminants	Concentration range (mg/kg)
Pb	95-175
Zn	150-220
As	130-140
Cr	700-1150
Cu	1075-1600
Ni	1170-2200
VOCs: BTEX	≤ 7
SVOCs: anilines, chloroanilines, nitrobenzenes, dinitrotoluene	≤ 1400
TPHs	≤ 8000

Table 2. Description of the soil-grout mixes.

Group		Binder			Organo clay	Binder components ratio	Slurry content (wt%)	Water Cement ratio
Group 1	PG	PC	GGBS			1:1	15	1:1
	PF	PC	PFA			1:2	15	1:1
	PFM	PC	PFA	MgO		1:4:5	15	1:1
	P	PC					15	1:1
Group 2	P-OC	PC			OC		18.2	1:1
	P-2IOC1	PC			2*IOC1		30	1:1
	P-IOC1	PC			IOC1		22.5	1:1
	P-IOC3	PC			IOC3		22.5	1:1
Group 3	PG-IOC3	PC	GGBS		IOC3	1:1	22.5	1:1
	PF-IOC3	PC	PFA		IOC3	1:2	22.5	1:1
	PFM-IOC3	PC	PFA	MgO	IOC3	1:4:5	22.5	1:1
	MG-IOC3	MgO	GGBS		IOC3	1:9	22.5	1:1
Group 4	M	MgO					15	1:1
	MG	MgO	GGBS			1:9	15	1:1

Table 3. Composition of inorgano-organoclay (IOC) slurries [36].

Slurry IOC per 1 m³:			
		Amount (kg)	**Dilution**
IOC 1	Bentonite KM	75	
	Water	900	
	Chlorhydrol	34	1:9
	MCB50	37.5	1:2
IOC 3	Bentonite KM	75	
	Water	900	
	Chlorhydrol	34	1:9
	PPG	37.5	1:2

Table 4. Leachate pH and leachability of heavy metals in the made ground.

Group	Mix	28 days (mg/l)					1.5 years (mg/l)				
		pH	Ni	Cu	Zn	Pb	pH	Ni	Cu	Zn	Pb
Group 1	PG	11.7	0.017	0.173	0.001	0.001	9.7	0.018	0.157	0.024	0.000
	PF	11.9	0.111	0.389	0.000	0.003	8.0	0.050	0.059	0.002	0.000
	PFM	12.0	0.098	0.071	0.003	0.004	8.0	0.000	0.000	0.007	0.000
	P	12.5	0.051	0.140	0.001	0.004	11.4	0.042	0.225	0.019	0.000
Group 2	P-OC	13.1	0.011	0.045	0.002	0.002	12.2	0.037	0.241	0.026	0.000
	P-2IOC1	12.5	0.121	0.411	0.001	0.008	11.8	0.084	0.153	0.025	0.001
	P-IOC1	12.3	0.041	0.141	0.001	0.004	11.6	0.040	0.260	0.002	0.001
	P-IOC3	12.5	0.030	0.101	0.001	0.005	11.8	0.026	0.116	0.033	0.001
Group 3	PG-IOC3	12.1	0.023	0.104	0.001	0.002	10.3	0.006	0.084	0.000	0.000
	PF-IOC3	11.7	0.037	0.195	0.001	0.001	10.5	0.012	0.142	0.000	0.000
	PFM-IOC3	12.1	0.012	0.043	0.001	0.001	10.7	0.001	0.038	0.003	0.000
	MG-IOC3	12.4	0.029	0.040	0.006	0.002	9.1	1.237	3.098	0.237	0.005
Group 4	M	11.1	0.004	0.020	0.001	0.022	10.1	0.000	0.015	0.000	0.000
	MG	12.0	0.010	0.097	0.002	0.002	11.3	0.027	0.040	0.018	0.000
DWS			0.020	2	5	0.025		0.020	2	5	0.025
LOD			0.009	0.004	0.006	0.006		0.009	0.004	0.006	0.006

LOD: limit of detection; DWS: drinking water standard (HMSO, 2009); standard deviation: 0.001-0.097.

Figure 1

Castleford-Green Lane
(Not to scale)
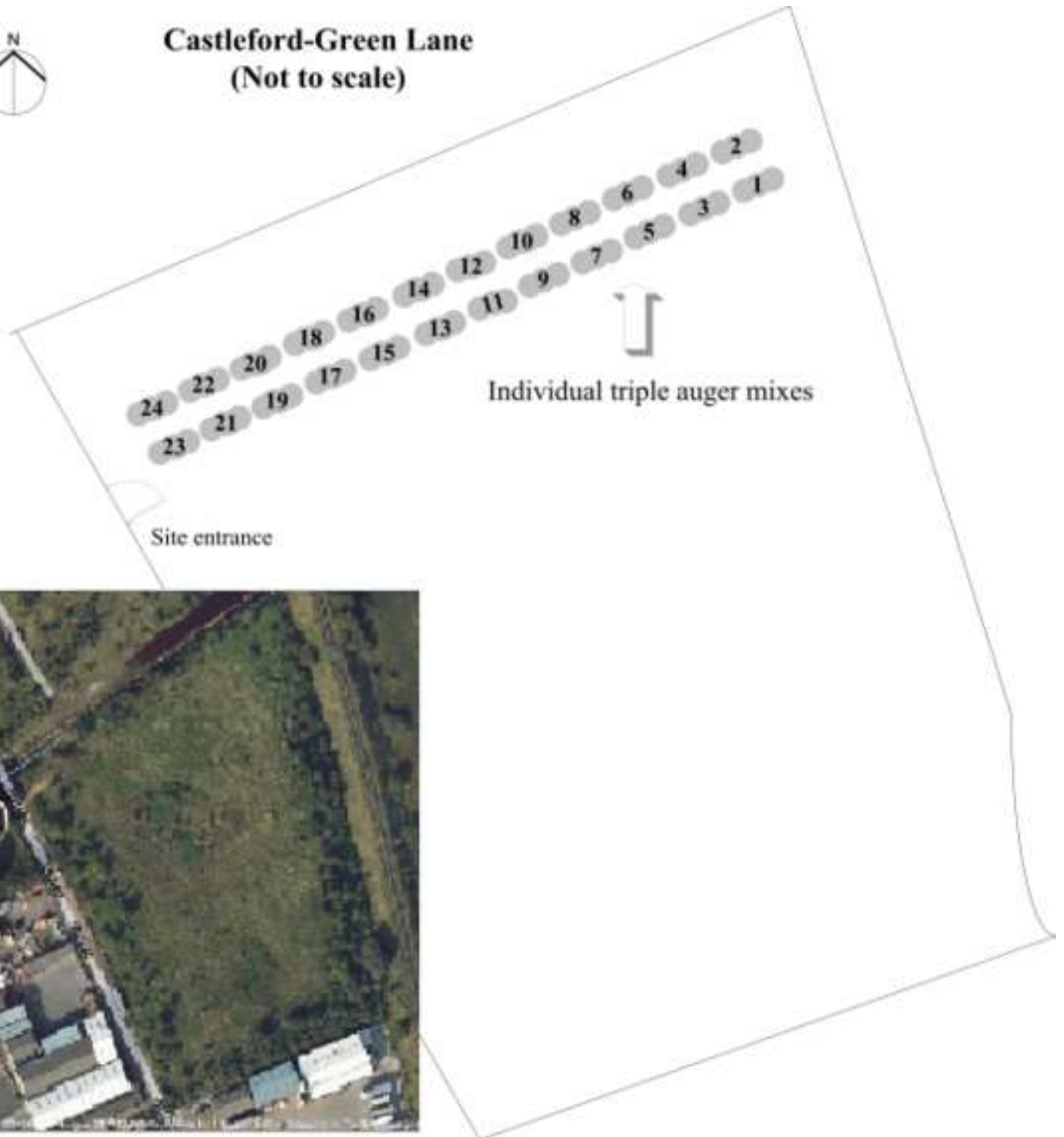
Individual triple auger mixes

Site entrance

Figure 2
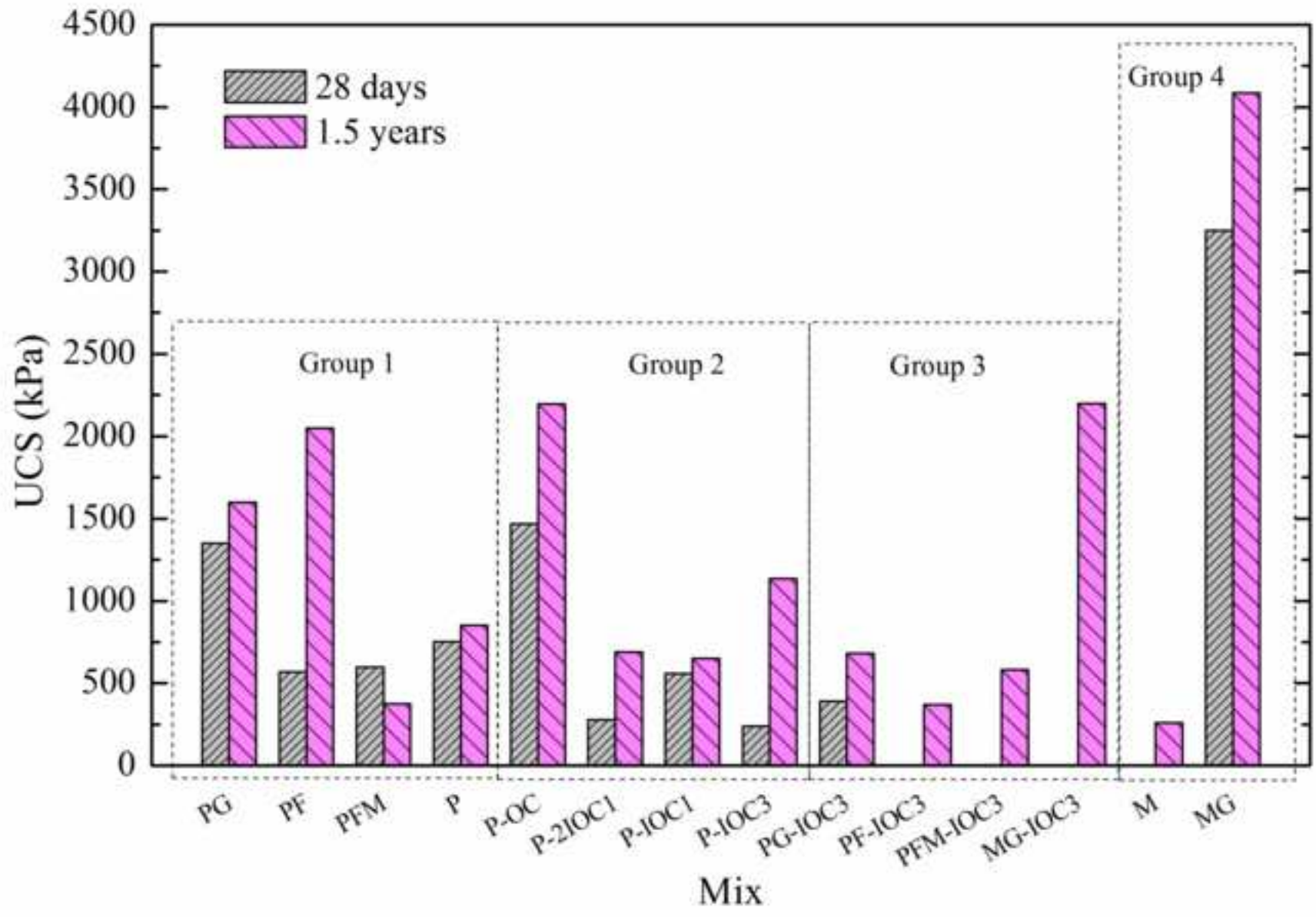
Figure 3

Figure 4