



Long-term performance of aged waste forms treated by stabilization/solidification

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ABSTRACT

Current regulatory testing of stabilized/solidified (S/S) soils is based on short-term performance tests and is insufficient to determine their long-term stability or expected service life. In view of this, and the significant lack of data on long-term field performance in the literature, S/S material has been extracted from full-scale remedial operations and examined using a variety of analytical techniques to evaluate field performance. The results, including those from X-ray analytical techniques, optical and electron microscopy and leaching tests are presented and discussed. The microstructure of retrieved samples was found to be analogous to other cement-based materials, but varied according to the soil type, the contaminants present, the treatment applied and the field exposure conditions. Summary of the key microstructural features in the USA and UK is presented in this work. The work has shown that during 16 years of service the S/S wastes investigated performed satisfactorily.

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1. Introduction

Solidification/stabilization (S/S) is used to reduce the mobilization of contaminants into the environment. This is achieved by the physical incorporation of contaminants within a hardened mass with lower permeability (solidification), and the chemical conversion of contaminants into less soluble forms (stabilization) [1]. The binders typically used include Portland cement, quicklime, pulverised fly ash (PFA), blastfurnace slag (BFS), natural or modified clays and sometimes waste/by-products with cementitious properties and proprietary additives [2].

The long-term performance of S/S soils is closely linked to both the physical and chemical characteristics developed after binder addition and the exposure conditions in the field. To date, most studies relating to S/S soils longevity focused on understanding their chemical performance by applying a number of accelerated short-term leaching tests to synthetic S/S soils [1], and very limited attention was given to real-life S/S materials [2,3]. The general con-

sensus is that although these tests offer an insight into the behavior of the S/S soils, they do not fully reproduce the conditions in real-exposure environments [4–6]. Therefore it is critical to obtain more field data pertaining to the long-term stability of the S/S soils. For this reason a series of cement-treated soils from full-scale remedial operations in the USA and the UK were obtained and analyzed in order to gain understanding of the long-term behavior of S/S soils.

2. Experimental

Cores of between 30 mm and 100 mm diameter, were extracted from 7 full-scale and 1 pilot scale sites. These sites comprised three Superfund and two private sites in the US, and three private sites in the UK. The key data on each remedial operation, including the binder formulations used, are given in Table 1.

The approach to sampling the sites differed based on availability of equipment, the specific site characteristics and the nature of the stabilized soils, and included wet and dry coring. Approximately half of the recovered cores were well-indurated and monolithic, whilst the other half, were poorly to non-indurated in nature. Consequently, the cores were often cracked, primarily due to sample extraction procedure. The presence of sampling artifacts was noted for each cored sample and this factor was also taken into consideration during the interpretation of microscopic observations.

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Table 1
Characteristics, contaminants and remediation formulation of the cement-stabilized sites studied.

Site and location (abbreviation)	Contaminants of concern	Contaminant concentrations (mg/kg)	Treatment date	Type of remediation and cover system	Depth of remediation	Remediation formulation ^a	Site reuse
Pepper Steel, USA (PS)	Pb, PCBs	2,000; 116	1989	Ex situ S/S (compacted) Capped with limestone	0.6 – 2.4 m	12% PC, 8% PFA	Industrial storage River front park
Columbus MGP, USA (MGP)	PAHs, TPHs	2,400; 5,500	1992	Ex situ and in situ S/S HDPE cap and topsoil	10 m	10% PC (site)	Not known
Quarry Dump, USA (QD)	TPH	1,200	1994	In situ S/S Concrete cap	1.2 – 1.5 m	25% PC	Barge terminal
South Eight Street, USA (S8)	Sulphuric acid, Pb	pH < 1.0; 500	2000	In situ S/S GCL cap and topsoil	5.1 m	25% crushed limestone, 20% PC, 10% PFA	Storage area for construction materials
American Creosote, USA (AC)	As, PAHs, PCFs, Dioxins	225; 335; 3,000; 0.00225	2000	Ex situ S/S (compacted) GCL cap and topsoil	0.6 – 1.5 m	5% PC, 4.5% PFA and 1.3% activated carbon	Football pitch-not in use
Halton, UK (HA)	Pb, As, Zn, Cu	1,550; 825; 980; 639	2000	In situ S/S Permeable membrane, topsoil	0.3 m	6% PC, 0.5% proprietary additive	Disused
Astra Pyrotechnics, UK (AP)	Pb, Cu, Zn	203; 64,089; 17,498	2000	Ex situ S/S (uncompacted) No cover	0.3 – 0.6 m	20% PC cell2; 20% Envirocem cell3	Residential development
Caerphilly, UK (CA)	PAHs, TPHs	32,960; 27,845	2006	In situ S/S Topsoil	1 – 1.5 m	12% proprietary mix	

^a Percentage by weight of soil: HDPE: high density polyethylene; GCL: geosynthetic clay liner; PAH: polycyclic aromatic hydrocarbons; PCF: polychlorinated biphenyls; PCB: polychlorinated biphenyls; TPH: total petroleum hydrocarbons; PFA: pulverized fuel ash; PC: Portland Cement.

2.1. X-ray diffraction (XRD)

The XRD analyses were made with a Siemens D500 diffractometer with a CuK α radiation source at 40 kV and 30 mA. The soils samples were ground into powder and scanned between 5° and 65° 2 θ , with a step size of 0.02° and a step time of 1.2 s. Peak identification and interpretation of the X-ray diffractograms was assisted by the DIFFRAC^{plus} EVA software (Bruker AXS).

2.2. X-ray fluorescence (XRF)

Bulk chemical analyses of the cement-stabilized soils were determined by X-ray powder fluorescence. The major elements were measured on glass beads prepared by fusion with lithium tetraborate, using a wide range oxide program. The trace elements were measured on pressed pellets using UniQuant[®] Thermo Scientific software.

2.3. Transmitted light microscopy

Replicate thin sections from the interior regions of the extracted cores were prepared by a specialized laboratory in Denmark, according to the procedure described in [7]. The fluorescent epoxy resin-impregnated thin sections were examined under polarized transmitted light (Optiphot-Pol, Nikon Instruments Inc.), equipped with a digital camera. The soil-derived minerals, pore structure, primary hydration products and secondary products (including carbonate, ettringite and gypsum) were examined.

2.4. Scanning electron microscopy (SEM)

A Jeol JSM 5310-LV Scanning Electron Microscope, equipped with a LINK-ISIS energy dispersive spectrometer (EDS) was used to study the S/S soils. Highly polished thin sections and resin blocks coated with carbon, were prepared from the S/S soils for microanalysis (accelerating voltage 20 kV). The (Ca/Si) elemental ratios of the C-S-H gel were determined using quantitative EDS point analysis on polished blocks.

3. Results and discussion

3.1. Physical characterization of S/S soils

3.1.1. Unconfined compressive strength (UCS)

UCS is a measure of resistance of monolithic or cohesive materials to stress, and can be applied to S/S materials to determine their performance [5]. UCS testing was performed on core samples from each S/S soil and the results are indicated in Fig. 1a together with the remediation targets. These targets were calculated depending on the location and extent of contamination, the potential site reuse and other site specific conditions. However, target UCS values are not always defined, as seen for the UK sites studied. Thus, the values presented in Fig. 1a for the AP and CA sites are not compared with any target values.

The results showed that in all cases but one, the UCS met or exceeded the remediation targets by up to one order of magnitude. This suggested that the S/S soils were still performing to the designed strength, up to 16 years after remediation.

Stabilized/solidified soils are often compared with concrete [3,8], but as seen from the results above, this is not appropriate. The UCS of the S/S soils ranged between 0.1 MPa and 4.2 MPa, which was comparable to stiff soil or very weak to weak rock and not concretes (Fig. 2). Although all S/S soils were treated with Portland cement-based formulations, no correlation was found between the quantity of binder and the strength of the soils. Additions of high percentages (up to 25%) of cement binder did not necessarily equate to high

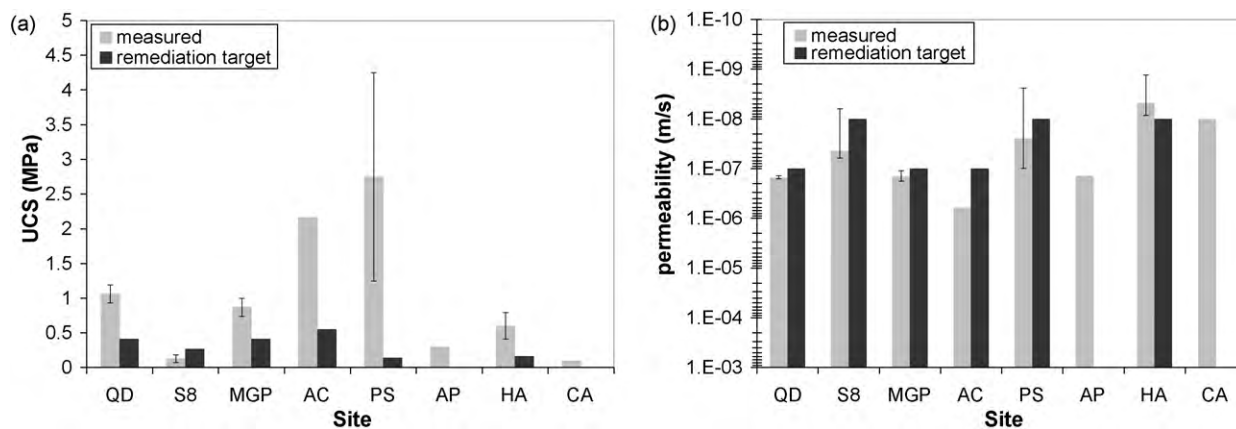


Fig. 1. Physical properties of the S/S soils (a) unconfined compressive strength and (b) permeability of the S/S soils compared with their site specific remediation targets (error bars interquartile range).

strength and this could be attributed to the method of delivery of the S/S or to degradative processes occurring over time.

3.1.2. Permeability

Permeability is a key transport property and influences the durability of treated wastes by preventing external agents from entering the S/S soils. The permeability of the S/S soils was in the order of 10^{-7} to 10^{-9} m/s (Fig. 1b), interestingly even for the low strength samples retrieved. This corresponds to the 'very low' permeability range, as described in [9]. Although the S/S soils were within the same order of magnitude with the remediation targets, the permeability materials remains slightly higher. However, according to reports by the USEPA, they remain suitable for the purpose for which they were designed [13].

It is useful to refer to the permeability of natural materials to place S/S soils into context, which shows that they are dissimilar to concrete, but similar to other cement-bound materials such as stabilized structural soil. The permeability of naturally occurring materials was described in Fig. 3. The values cited by [9], are also included, showing some soils have a permeability one order of magnitude lower than those given for S/S soils.

3.2. Chemical and mineralogical characterization

3.2.1. XRF

The major and trace element composition of the S/S soils was determined by XRF and the results presented in Fig. 4. The S/S soils were silica rich, contained up to 68% SiO_2 , 29% CaO , 14% Al_2O_3 , and 9% Fe_2O_3 .

Other elements identified included magnesium, sodium, potassium, chromium, titanium, zinc, lead and manganese, with

concentrations accounting for less than 1% of the S/S soil mass. The loss on ignition had a substantial contribution, up to 31% from the total weight of sample (Fig. 4).

3.2.2. XRD

The main crystalline phases identified were ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{16}\cdot 26\text{H}_2\text{O}$), gypsum ($\text{CaSO}_4\cdot 2\text{H}_2\text{O}$), calcite and aragonite (CaCO_3), mullite ($3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) and quartz (SiO_2), pyrite (FeS_2), dolomite ($\text{CaMg}(\text{CO}_3)_2$), clay minerals, mica, feldspars, hence a mixture of cement derived phases and soil minerals, respectively. Since the mineralogy was dependent upon specific site conditions e.g. soil and binder type and the environmental exposure, a list of site specific minerals was compiled (Table 2).

All sites contained quartz, calcite and ettringite, whilst gypsum was identified only in the PS, S8, QD and HA derived samples. Although the origin of quartz was entirely from the soil, calcite was present in both the untreated soil and resulted from the atmospheric carbonation of the cement phases. Ettringite and gypsum formed as a result of secondary reactions, which varied from site to site. Mullite, an aluminium silicate formed by burning coal at high temperature, was observed in the soils remediated with PFA mixtures.

Portlandite ($\text{Ca}(\text{OH})_2$) resulting from cement hydration was absent in most S/S soils, except for AP. The absence of portlandite could be explained by the use of pozzolanic materials, such as PFA, but also by atmospheric carbonation. With the exception of AP, the pH of the treated soils varied between 7.5 and 10.5. In this pH range most metals of concern have minimum solubility [8]. Portlandite presence in the AP soil is most likely due to slow hydration of the cement binder, which is still present in unhydrous form as shown from the SEM examination.

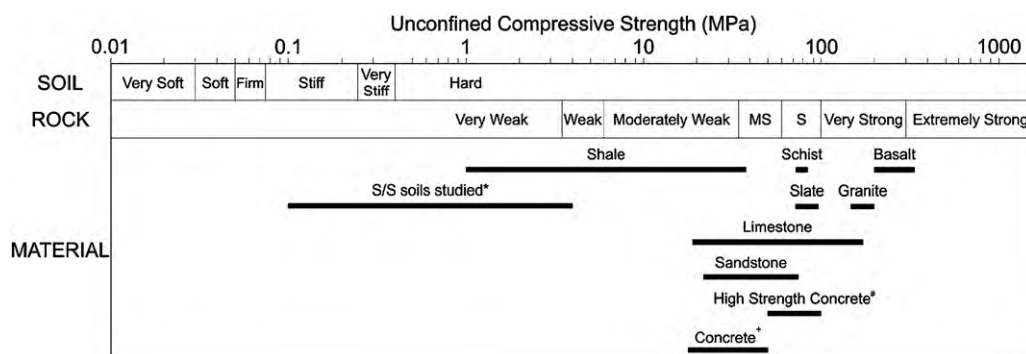


Fig. 2. Comparison between the unconfined compressive strength of S/S soils and various natural and synthetic materials.

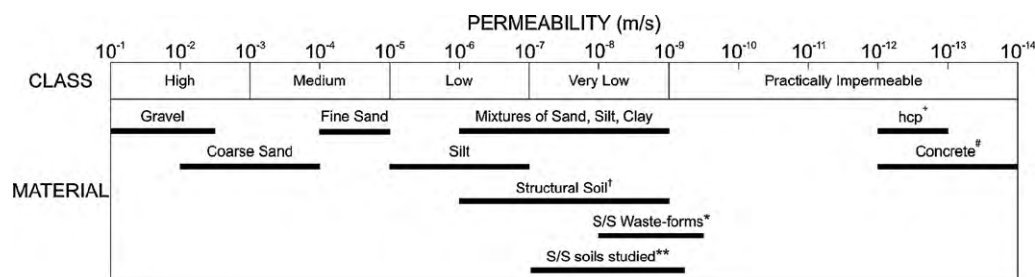


Fig. 3. Permeability of S/S soils in comparison with various materials (modified from [9]) * [10], + hcp: hydrated cement paste [10], # [11], † [12].

3.3. Microstructure

The S/S soils were subject to physical and chemical alteration and the characteristic features observed are shown schematically in Fig. 5. Different polymorphs of calcium carbonate, sulfate minerals (secondary ettringite and gypsum), unhydrated cement grains, remnant secondary binders (PFA) and weathered minerals were amongst the phases observed.

The treated soils, even after years of service, contained isolated residual anhydrous cement grains from the original S/S treatment. These grains are derived from the larger size fractions of the cement, with diameters of 60–100 μm . Previous research has shown that, in concrete, residual unhydrated cement (notably the larger sized grains) is not unusual even after many years of service [14]. In the absence of cement hydration, the main hydration phase, C-S-H, will not form. This hydrate phase plays a key role in metal immobilization and the resistance of cementitious materials to acid attack, as shown by numerous authors [15,16]. The C-S-H gel composition is variable and the incorporation of anions or cations has been linked to the Ca/Si molar ratio. The average Ca/Si ratio for each site is shown in Fig. 6 and varies from 0.5 to 1.0. Glasser [17] showed that at low Ca/Si ratios, the surface charge of C-S-H gel is negative, and thus has an improved cation absorption capacity. A Ca/Si ratio of lower than 1, is ideal for retention of heavy metals [16]. This hypothesis was supported by the data from pH dependent leaching tests performed for the S/S and the MINTEQA2 geochemical modeling of results [18]. In general, the measured concentrations for metal leaching was much lower than the theoretical solubility derived for oxide, hydroxide and silicate mineral forms included in the MINTEQA2 thermodynamic database. This finding suggests that the heavy metals are efficiently retained in the solidified matrix as a result of immobilization mechanisms within cement hydration phases.

Cracking was observed in all cores recovered in this investigation. This could result from drying shrinkage, plastic settlement, freeze–thaw, as well as deleterious chemical reactions such as alkali aggregate reaction, sulfate attack or carbonation [22]. Cracks varying from 1 micron to a few tens of microns were observed, but in many cases the cracks were empty, suggesting they may have occurred during core extraction or may be an artifact of the sampling procedure. However, some cracks were partially or com-

pletely filled with secondary products, such as ettringite, or calcium carbonate, indicating that they must have formed in situ, after treatment. It should be noted that some samples contained significant cracks that were not caused by the formation of secondary products such as ettringite [3].

As can be seen from Table 2, all S/S soils were subjected to carbonation. The XRD and SEM results showed that carbonation occurred at all sites, irrespective of their age or placement in the environment. It must be noted that impermeable geomembranes were used to protect the S/S soils from water ingress; however this did not display the same efficacy for carbon dioxide permeation. Calcium carbonate was observed predominantly within voids, but also within microcracks and in the matrix, primarily leading to a densification of the S/S soil. Calcite was common in the S/S soils, and resulted from the carbonation of portlandite. Fig. 7a–c are transmitted light micrographs showing calcium carbonate growth in large voids. Well-formed blade like-crystals between 20 and 30 μm in size were frequently observed indicating that sufficient space was available for their growth [14].

Ettringite was observed in all the S/S soils retrieved. However, using optical techniques it was seen to form predominantly in large voids or within air voids, at aggregate–paste interface, in spaces within porous carbon-rich/coal particles, between mica lamellae, and in the matrix in a limited number of sites (Fig. 8a–d). The mechanisms of expansion and ettringite formation in cementitious systems have been discussed in detail in [23,24]. Klich [25] argue that large ettringite crystals often appear in the available space offered by pre-existing cracks and voids, which is a benign process [14,23,26].

Gypsum was scarcely observed in the S/S soils by SEM/EDS and this may be caused by the similarity between its backscattered coefficient and that of calcium carbonate or the localized distribution of this mineral. Anhydrous crystals of gypsum, in the order of 200 μm diameter, were observed to infill an entrapped air void, with no apparent signs of distress of the matrix. In this study the gypsum identified was not necessarily a degradation product following years of environmental exposure, but the result of acidic soil conditioning prior to the S/S treatment (see S8 site).

Altered mica was identified in the S/S soils in the most southerly located two sites, QD and MGP. The alteration of mica may be ascribed to natural weathering of the soil prior to the remediation,

Table 2
Mineral phases identified in the S/S soils.

Site	Quartz	Calcite	Aragonite	Gypsum	Ettringite	Portlandite	C ₂ S, C ₃ S	Mullite	Clays	Feldspars	Dolomite	Pyrite	Micas
AC	●	●			●					●	●		
PS	●	●	●	●	●			●					
S8	●	●		●	●					●			
MGP	●	●			●				●	●			●
AP	●	●			●	●	●			●		●	
QD	●	●		●	●				●	●			●
HA	●	●	●	●	●				●	●		●	
CA	●	●			●			●	●		●		

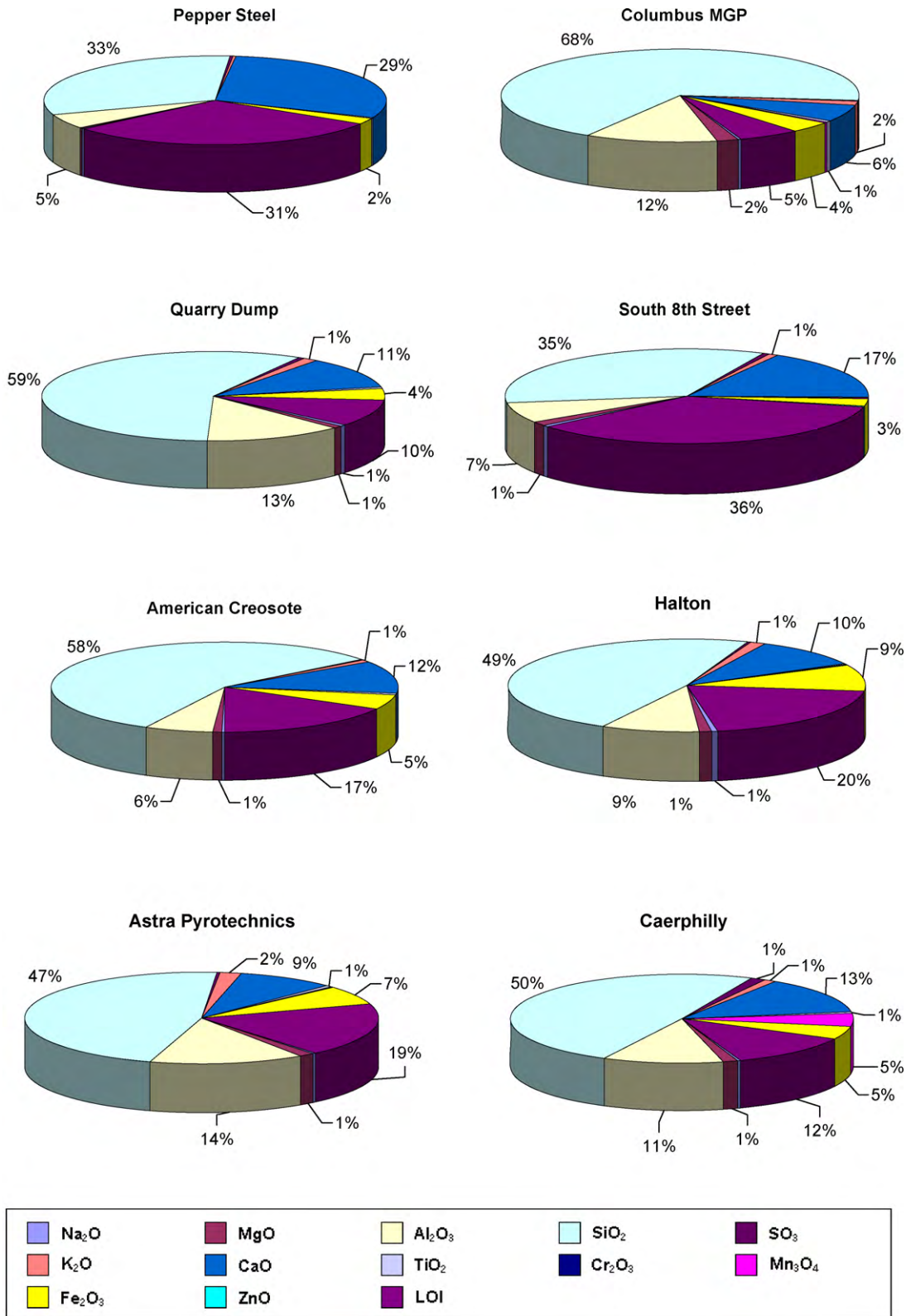


Fig. 4. Elemental oxide compositions of S/S soils and corresponding loss on ignitions (LOI).

rather than degradation in the high pH conditions of a cement-bound system [27]. The soil consists of saprolite, a chemically weathered rock occurring in wet and warm climate [28]. Highly altered alkali-bearing mica and feldspar are known to be potentially deleterious due to the increase risk of alkali release into the

pore solution, promoting alkali aggregate reaction [27,29]. However, no evidence of alkali aggregate reactivity was observed in the soils. Instead, a rather interesting reaction within the weathered mica was observed, i.e. ettringite forming between the mica lamellae leading to expansion in the direction normal to the layers (Fig. 9).

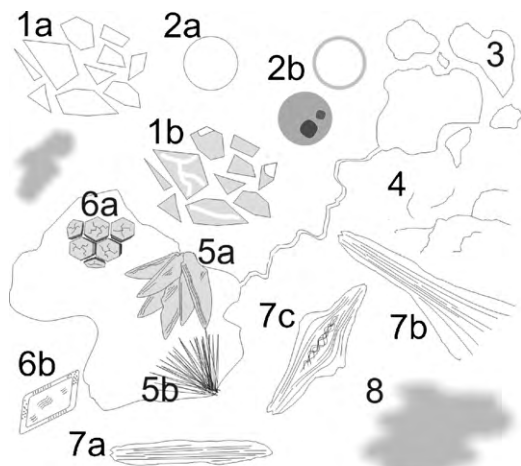


Fig. 5. Schematic representation of the main alteration features in aged *S/S* soil microstructures based on microscopy studies: 1a, unhydrated cement grains; 1b, hydrated cement grain "relics"; 2a, unreacted fly ash; 2b, partially reacted fly ash cenospheres; 3, voids; 4, microcracks; 5a, calcite; 5b, aragonite; 6a, ettringite; 6b, gypsum; 7a, mica aggregate; 7b, kaolinised mica; 7c, altered mica aggregate and ettringite intergrowth; 8, organic inclusions within soil/cement matrix.

This phenomenon is a type of sulfate attack, which results due to the reaction between sulfate ions and reactive alumina supplied by an aggregate, for example. This occurrence was also reported by Diamond [26] in concrete and is acknowledged to pose a risk for the dimensional stability. A detailed analysis of this phenomenon and the mechanisms involved will be discussed elsewhere.

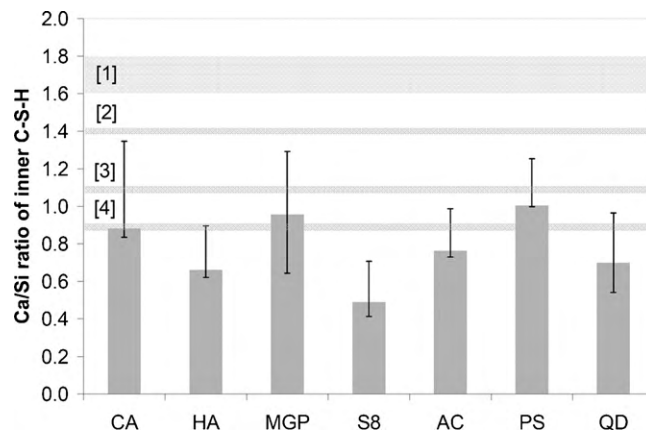


Fig. 6. Average elemental Ca/Si ratios of the inner C-S-H gel from the *S/S* sites (error bars interquartile range). [1] Portland cement [19,20]; [2], [3], [4] fly ash blended cements [19,18,21].

3.4. Contaminant immobilisation

S/S treatment does not eliminate the contaminants, which remain immobilized within the product. The cementitious component of the *S/S*-treated soil responsible for encapsulation, is therefore extremely important for contaminant retention over time. However, longer-term chemical reactions and waste binder-interferences may also occur in the *S/S* soils, and may also reduce the mobilization of contaminants into the environment [2]. To assess these two key aspects of *S/S*, post remediation monitor-

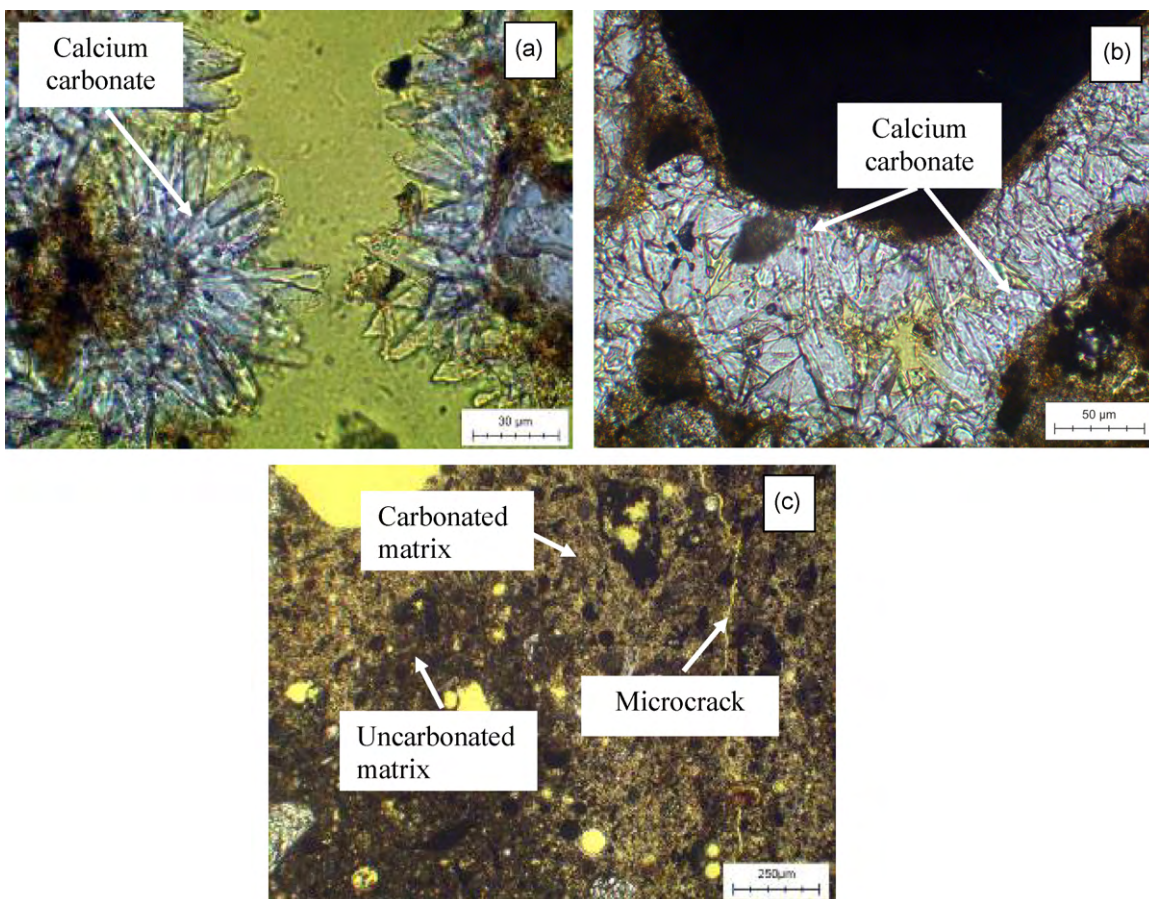


Fig. 7. Transmitted light photomicrographs of well crystallized calcium carbonate infilling large pore spaces in the *S/S* soil; a) plane polarised light 'dog's tooth' crystals of calcite; b) interlocking crystals of calcite; c) carbonated matrix around microcrack (PS).

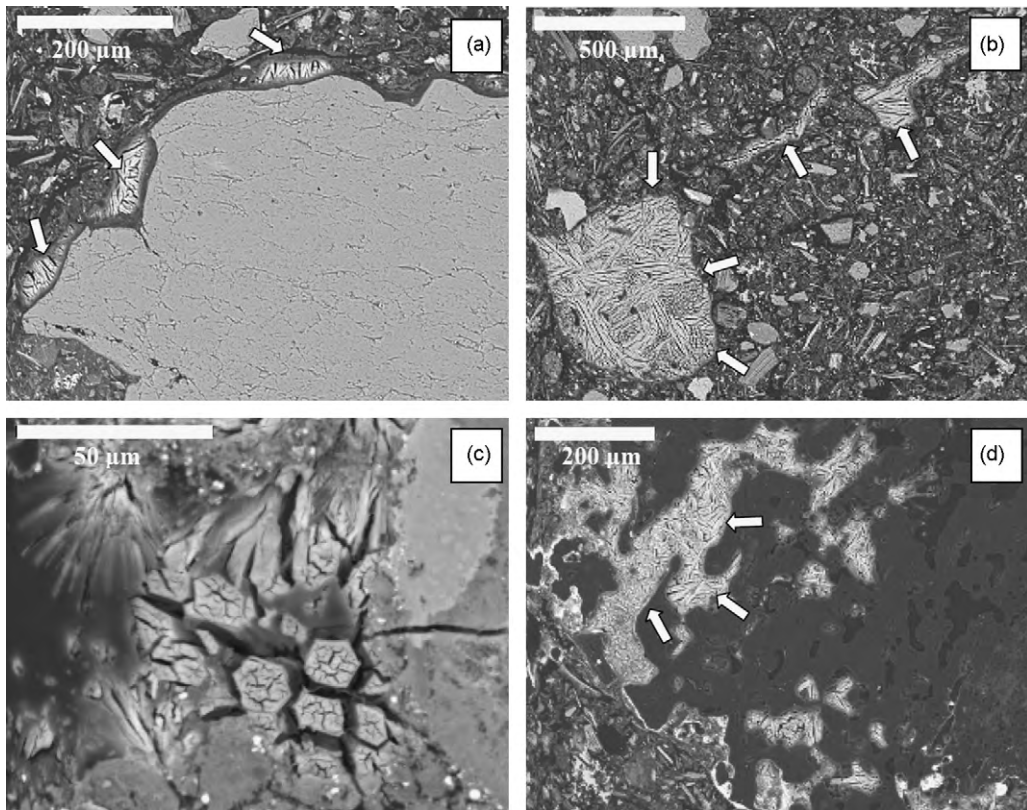


Fig. 8. Backscattered electron images of ettringite formed at: a) the interfacial regions of an aggregate particle (QD site); b) in voids and microcracks (QD site); c) within the matrix (HA site); d) in interconnected voids within a carbon rich/unburnt coal particle (QD site).

ing can be carried out on contaminant release, both within and outside of the treated area [4]. Monitoring depends greatly on the specific contaminants and the conditions prevailing on site; and may include pH, redox, conductivity and contaminant concentration in groundwater. Occasionally, archived samples are tested over time for e.g. unconfined compressive strength (UCS), permeability, California Bearing ratio (CBR) and leaching. The results obtained can be compared against target values set at the time of remediation, and thus can give an indication of the current performance of the cement-treated soil. However, this degree of scrutiny is quite uncommon in practice, hence

the lack of strong data relating to field performance of the S/S soils.

The contaminants encountered in the soils were inorganic (lead, zinc, chromium, copper, arsenic) and organic compounds (PAH, PCB, TPH), as indicated in Table 1. The results presented in Table 3 were obtained using different leaching tests, as indicated. It must be noted that, for a number of sites, there are no enforceable limits for metal leaching, but benchmarks for comparing leaching concentrations from the S/S soils. For the US sites (AC, PS, S8, QD and MGP), often the EPA's Maximum Contaminant Level (MCL) for drinking water is used as a benchmark. However, the MCL value is usually

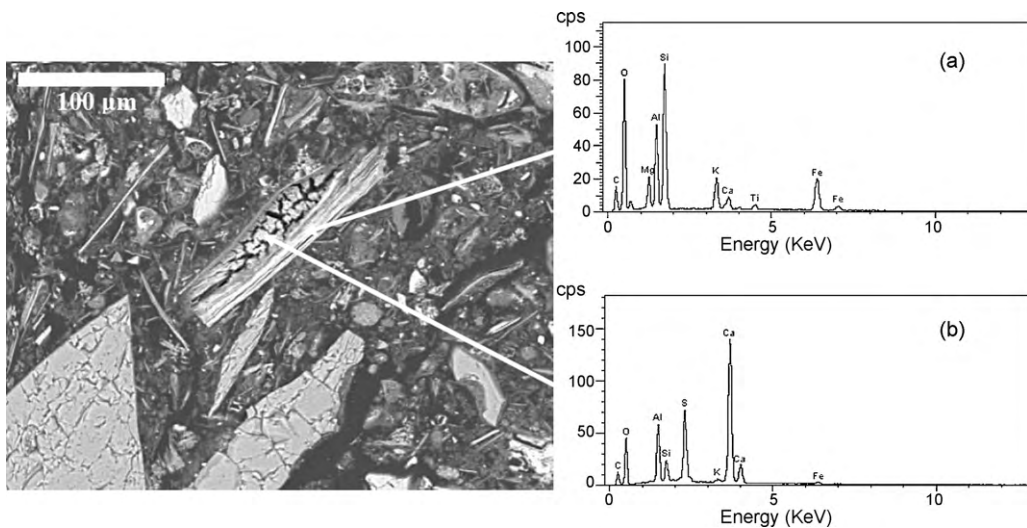


Fig. 9. Ettringite formed between mica lamellae and the corresponding EDS spectra: a) unaltered mica; b) ettringite.

Table 3
Contaminant leaching from the S/S soils up to 16 years following remediation.

Contaminant	Leaching concentration in mg/l (target values in brackets)							
	AC ^a	PS ^a	S8 ^a	QD ^a	MGP ^a	AP ^b	HA ^c	CA ^d
Lead		0.029 (0.050) ^e	0.04 (0.015) ^e			<0.008 (0.015)	0.060 (0.025)	
Arsenic	<0.002 (0.050)	0.006 (0.050) ^e	<0.002 (0.050) ^e				0.060 (0.010)	
Copper						0.06 (5)	0.07 (2)	
Zinc						0.01 (5)	0.01 (0.8)	
TPH				bdl (0.001)				0.5 (0.8)
PAH	0 ^f (0.01)				0.37 (10)			0.2 (0.1)
Dioxin	3.6 (30 × 10 ⁻³)							
PCP	0.16 (0.2)							

^a USEPA SPLP 1312.

^b DIN 38414-S4.

^c NRA.

^d NEN 7375.

^e The State Drinking Water Standards were lowered since the remediation to 0.010 mg/l for arsenic and 0.015 mg/l for lead.

^f Benzo α pyrene (BaP) equivalent.

bdl signifies values below detection limit.

viewed as a target to be achieved at a point of compliance i.e. monitoring well, at the site boundary, rather than a leaching limit for the metal release from the S/S soil. Therefore the drinking water standards are an indirect means of monitoring the performance of the soil stabilization remedy and its long-term impact on groundwater. Remediation target values equal to drinking water quality limits are over specified, due to the numerous factors which contribute to dilution, dispersion and retardation of the contaminant release. The contaminant concentration in groundwater is determined by the permeability of the S/S matrix, the rate of diffusive release of contaminants to infiltrating water, retardation in the vadose and additional hydrogeological factors i.e. aquifer thickness and hydraulic gradient. Therefore higher levels are allowed in the SPLP extract so long as the levels will be reduced to the MCL at the site boundary or other points of compliance. In Table 3, the benchmark values are presented in italics.

The AP, HA and CA soils did not have enforceable leaching limits, but guideline values derived from Drinking Water Limits [30,31] and site specific risk assessments, respectively.

All contaminants throughout the S/S soils have complied with the guideline or target values, except for lead at S8, as shown in Table 3. The SPLP results for S8 showed that three out of five samples leached lead above the drinking water action level of 0.015 mg/l. However, due to the soil heterogeneity, the USEPA allowed for 20% of samples to be twice the limit, and for 10% of samples to exceed by a factor of 5, provided that the average of all samples tested met the limit [32]. Also, a recent report by the US EPA on the S8 soil performance indicated that despite the lead leaching following the SPLP test, the groundwater concentrations have not reached the action level. Therefore, the treatment is still protective of the groundwater, which was identified as the main risk of contamination [32].

4. Conclusions

This work examined the long-term performance of S/S soils up to 16 years old. The eight Superfund and private sites in the UK and USA were characterized and the main conclusions were:

- The S/S materials retrieved, in general, met their original acceptance criteria for physical performance. The S/S soils were shown to behave like cement-bound materials rather than concretes, as indicated by the physical characteristics and mineralogical and microstructural observations.
- A number of potential key risk indicators for the performance of S/S soils were identified i.e. carbonate minerals, sulfate bearing minerals (ettringite and gypsum) and weathered minerals. Carbonation and ettringite growth are regarded as deleterious

processes affecting concrete. The observations made suggest that their role in S/S soils is largely inconsequential.

- The release of contaminants from the S/S soils was within the specified limits. This suggests that the contaminants are likely to be adequately immobilised over an extended period of time.

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