# Characterisation of environmentally exposed cement-based stabilised/solidified industrial waste

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## Abstract

A metal-plating waste filter cake treated by stabilisation/solidification (S/S) using ordinary Portland cement (OPC) and pulverised fuel ash (PFA) has been characterised after exposure to the environment in SE England for approximately 10 years. The surface region ($\sim$5 cm) was severely degraded, extensively carbonated and had reduced acid neutralisation capacity (ANC) compared to bulk samples. Large 'plate-like' deposits of predominantly calcium hydroxide with a calcium carbonate upper layer were found close to, but below the surface of the exposed S/S waste. Calcium zinc hydroxide ($Ca(Zn(OH)_3)_2 \cdot 2H_2O$) was the major crystalline phase found in the S/S waste in the region below the calcium hydroxide plates (10–15 cm). Samples taken from the bulk of the environmentally exposed S/S waste, at a depth of approximately 0.5 m, were more amorphous, contained no readily identifiable crystalline phases and had negligible strength but retained high acid neutralisation capacity. Metal analysis of homogenised samples taken from different depths into the S/S waste indicated a reduction in the concentration of heavy metals, such as Zn, Fe and Cr, in the top 5 cm of the S/S waste and an increase in concentration of these metals in bulk samples. The majority of crystalline mineral phases detected in the 28-day samples were not identified in the 10-year-old samples.
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## 1. Introduction

Stabilisation/solidification (S/S) is used as a pre-landfill waste treatment technology that aims to make hazardous industrial wastes safe for disposal [1,2]. The process involves mixing hazardous wastes, either in the form of sludge, liquid or solid, into a cementitious

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binder system, and is most suitable for treating wastes that are predominantly inorganic, as these are considered to be more compatible with the types of cementitious binders normally used. However, a wide range of wastes, including many mixed inorganic/organic materials have now been treated using S/S technologies, particularly in the USA. The aim is to encapsulate and incorporate the waste in the binder system and produce a monolithic solid with structural integrity and long-term stability that exhibits minimal leaching.

S/S waste inhibits leaching of hazardous components by reducing waste/leachant contact and by forming a stable pH environment in which many heavy metals of environmental concern remain insoluble. A range of other mechanisms are also reported to occur for specific combinations of waste components and binders that may further inhibit leaching [3].

Information on the long-term performance and durability of S/S wastes is not readily available. The effects of freeze/thaw and wet/dry cycling, acid rain, sulphate attack, alkali-aggregate reaction, carbonation, microbial influenced degradation and leaching can all degrade cement-based materials. The same environmental factors that affect the durability of concrete must also be considered when evaluating the durability and permanence of cement-based S/S wastes [4].

S/S of industrial wastes produces materials that tend to be highly variable in composition and very weak compared to normal constructional concrete [5]. They are variable because the wastes they incorporate will normally vary in composition and the mixing processes used to produce S/S waste results in significant material inhomogeneity. The cementitious binders used are often a major cost in the process and therefore there has been an incentive to use the minimum amount of binders that enables the S/S waste to pass short-term regulatory requirements, such as those for early strength and waste component leaching. In addition, the waste will often adversely affect binder-setting reactions such as cement hydration, causing initial and potentially long-term interactions that can degrade physical properties [6–10].

The highly complex chemistry of S/S wastes, combined with poor physical properties makes predicting long-term performance and durability difficult, particularly as the nature of the stresses imposed on the material depend on the specific disposal/exposure conditions. However, this is a key research requirement and the aim of this work was therefore to investigate the long-term performance and properties by assessing the changes that have occurred to a specific S/S waste after exposure to the environment in SE England for approximately 10 years.

## 2. Field site design and material characterisation

### 2.1. Materials

A S/S waste exposure site was constructed in SE England in 1992 containing 2.5 m$^3$ cylindrical blocks of S/S waste. The cylinders were approximately 1.4 m in diameter and had a height of 1.6 m. They were cast in stainless steel containers housed in a purpose built structure that exposed the top circular surface of the cylinders to the environment [11].

The industrial waste used in the field site S/S waste was a filter cake obtained from a metal-plating company. Analysis data showed that this contained high levels of Zn (310 000 mg kg$^{-1}$ dry weight), Fe (135 000 mg kg$^{-1}$), Cu (315 mg kg$^{-1}$), Ni (106 mg kg$^{-1}$)

and Pb ($140\,\mathrm{mg\,kg^{-1}}$), although no information on the variability in composition was available. It was solidified at the exposure site using ordinary Portland cement (OPC, supplied by Blue Circle (UK) Ltd.) and Class 2 pulverised fuel ash (PFA, BS3892). The S/S waste had a mix design (weight percent) of 18% OPC, 18% PFA and 64% filter cake waste. This gave an overall solids content of 55% due to the water content contained within the filter cake, as no additional water was added to the mix. It should be noted that this was not a commercially produced S/S waste. However, the composition was chosen to be representative of the type of material believed to be produced by S/S waste plants operating in the UK during the late 1980s [11].

## 2.2. Field site S/S waste production

The S/S waste components were mixed using a 500 l planetary mixer. The cylindrical S/S waste blocks were prepared in 350 kg batches, with each batch being mixed, placed and compacted on-site. Typically around 13 batches were required to fill each container to approximately 2 cm below the top rim of the steel container. After filling, the top surface was covered with high-density polythene sheeting and the material allowed to cure for 28 days before being exposed to the environment.

## 2.3. Initial field site S/S waste characterisation data

Samples for unconfined compressive strength testing (10 cylinders, 50 mm diameter × 50 mm) were prepared from the S/S waste mixes used at the site and tested after 28 days. The mix composition used at the field site had a mean strength value of 871 kPa, indicating some binder setting but probable inhibition of normal cement hydration reactions.

Fractured compressive strength samples were used for the toxicity characterisation leaching procedure (TCLP) with leachates analysed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The TCLP leachate was found to contain Cr ($3.89\,\mathrm{mg\,l^{-1}}$), Fe ($2.08\,\mathrm{mg\,l^{-1}}$) and Zn ($1.48\,\mathrm{mg\,l^{-1}}$) and had a pH of 7.5.

Twenty eight-day samples were also analysed to identify major crystalline phases present by X-ray diffraction (XRD, Philips 2000 series) using copper K-alpha radiation on powdered ($<150\,\mu\mathrm{m}$) samples. The crystalline phases identified in the 28-day samples are given in Table 1 [11].

Table 1
Main crystalline phases present in 28-day S/S waste [11]

| 2$\theta°$ | D-spacing | Relative intensity | Mineral |
| --- | --- | --- | --- |
| 15.80 | 5.61 | 50 | Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot26H_2O$) |
| 24.91 | 3.58 | 60 | Vaterite ($CaCO_3$) |
| 26.66 | 3.35 | 74 | Silica ($SiO_2$) |
| 29.44 | 3.04 | 100 | Calcite ($CaCO_3$) |
| 31.07 | 2.88 | 61 | Alite ($3CaO\cdot SiO_2$) |
| 32.7 | 2.74 | 84 | Belite ($2CaO\cdot SiO_2$) |

## 3. Environmentally exposed S/S waste

### 3.1. Nature of exposure

The degradation of S/S waste will depend on the characteristics of the environmental exposure it experiences and an important feature of the exposure of the S/S waste studied in this work was the potential for rainwater to 'pond' on the top surface. This could collect to a maximum depth determined by the distance between the S/S waste surface and the rim of the steel container containing the material (∼2 cm). Any additional rainwater falling on the S/S waste would then overflow to a collecting tank. The S/S waste is therefore likely to have experienced extended periods of surface saturation, potentially combined with freeze/thaw cycling during certain periods of each year. The surface region will also have experienced stresses induced by drying and relatively high temperatures during summer months.

### 3.2. Surface region characterisation

Fig. 1 shows the effect of this type of exposure on the appearance of what was originally a monolithic S/S waste surface. It is evident that the surface has become severely degraded. After 10 years environmental exposure most of the top 2–3 cm of the S/S waste had a soil-like texture and was colonised by various types of algae, insects and other organisms. This region was also found to contain extensive plates of a hard material that varied in
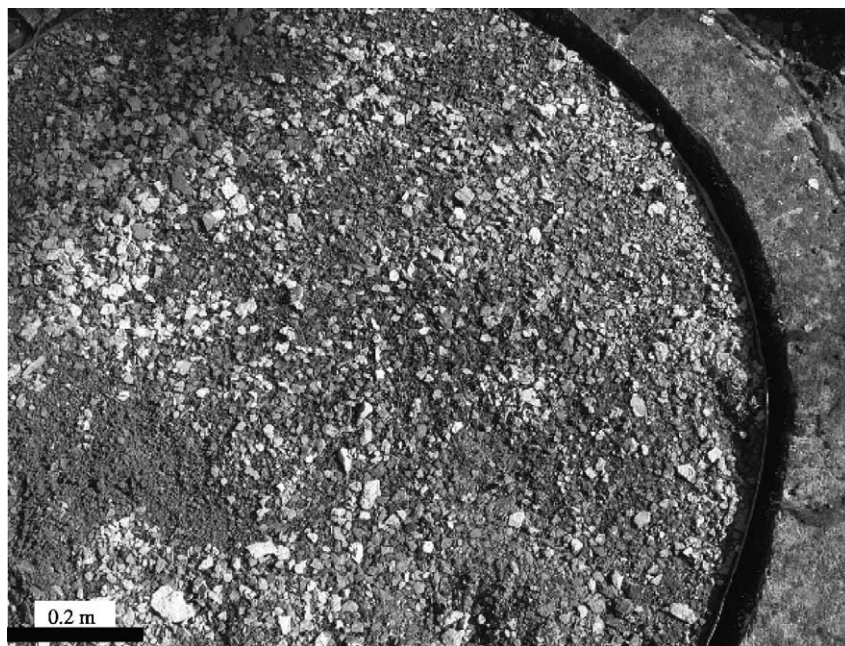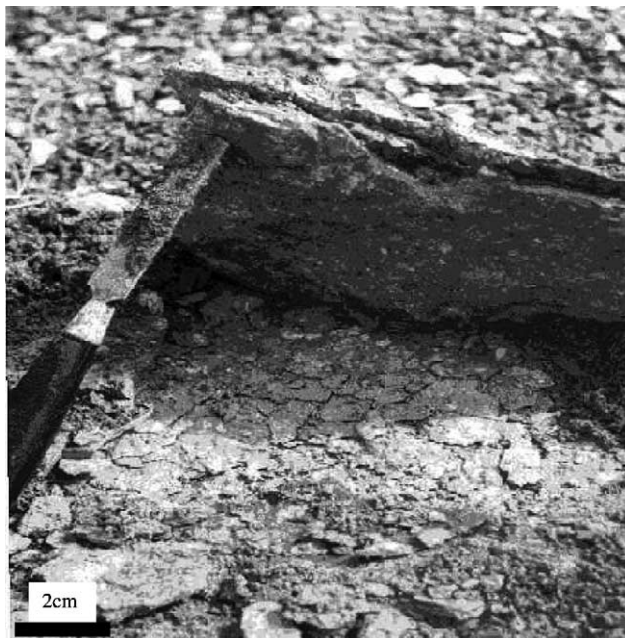


Fig. 1. Surface of environmentally exposed S/S waste.

Fig. 2. Plate-like deposits found close to the surface of environmentally exposed S/S waste.

thickness from a few millimetres up to about 2 cm. An example of this type of feature is shown in Fig. 2. These were generally fragmented, but present across the whole of the exposed S/S waste, normally close to (but just below) the surface and clearly had very different composition and properties from the original S/S waste. The plates consisted of two distinct regions, with the majority being a hard, stone-like grey material, with a more friable dark layer containing fine spherical cavities on the upper surface. In some regions, the grey material was observed to have encapsulated regions of S/S waste.

XRD has been used to characterise the crystalline phases present in this material using a Philips P1830 diffractometer system fitted with a PW1820 goniometer, and 40 kV, Cu Kα radiation. The current used was 40 mA and samples were scanned with a 0.02° 2θ step size.

### 3.3. Surface cores (0–15 cm)

It was impossible to take normal concrete cores from the 10-year-old S/S waste using wet diamond-cutting equipment because the material rapidly disintegrated into slurry. Three cores of material (denoted 1–3) were therefore hand excavated from the S/S waste at positions uniformly distributed across the diameter of the exposed S/S waste surface to a depth of approximately 15 cm, where the material appeared to be relatively homogenous.

The excavated material from each core, not including the hard plates of material, was divided into three equal portions, labelled 0–5, 5–10 and 10–15 cm. These were dried overnight at 105 °C, crushed in a mortar and pestle and passed through a 150 μm sieve to

produce homogenised samples. These were then digested [12] and analysed using ICP-AES (Perkin-Elmer Optima). The Optima ICP-AES has the capacity to use multiple emission lines simultaneously and has increased capability to resolve inter-element interference and background matrix effects.

XRD was used to identify the crystalline phases present in the homogenised samples using the same preparation and analysis techniques as for the plate material. Leaching characteristics of samples were assessed using the acid neutralisation capacity (ANC) test combined with leachate analysis to give metal release data as a function of leachate pH [13].

The ANC test was repeated in duplicate on the different portions of all three cores and involved the following stages

   i. Eleven acid washed centrifuge tubes were rinsed using distilled water.
  ii. 2.5 g of test sample was placed in each tube.
 iii. Appropriate quantities of nitric acid and distilled water were added to each tube to give a liquid to solids ratio of 10:1 and acid concentrations ranging from 0 to 12 meq./g.
 iv. The contents were manually shaken to ensure proper mixing.
  v. Tubes were then placed in the rotary extractor for 48 h, at room temperature.
 vi. Following removal of the tubes from the rotary extractor, the tubes were centrifuged for 10 min at 6000 rpm.
 vii. The pH of the supernatant in each tube was measured.
viii. The supernatant was decanted and filtered through a 0.45 μm filter paper into plastic tubes.
 ix. These tubes were acidified to 10% $HNO_3$ and refrigerated to preserve the samples for subsequent metals analysis.

ICP-AES was used to determine the leachate concentrations of Al, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Sr, Ti, V, Y and Zn following appropriate dilutions as required.

### 3.4. Characterisation of 'bulk' exposed S/S waste

In addition to characterisation of the top 0–15 cm 'surface' samples, S/S waste samples were taken from approximately 0.5 m below the exposed top surface. These are referred to as 'bulk' samples in later sections. The bulk samples were homogenised (dried, crushed and sieved) and analysed for chemical composition, crystalline phases and leaching using the same test procedures and analytical techniques as for surface samples.

## 4. Results

### 4.1. Analysis of major crystalline phases by XRD

XRD data for S/S waste taken from the surface region is shown in Fig. 3. Calcite ($CaCO_3$) was the major crystalline phase identified in the 0–5 cm sample. The 5–10 cm sample contained both calcite and monohydrocalcite ($CaCO_3 \cdot H_2O$), while the 10–15 cm sample

Counts/s



Fig. 3. XRD data for samples taken from the surface region of environmentally exposed S/S waste.

Table 2
Summary of XRD data indicating major crystalline phases present in S/S waste samples

| Material | Major crystalline phases identified |
|---|---|
| Environmentally exposed S/S waste (cm) | |
| 0–5 | Calcite ($CaCO_3$) |
| 5–10 | Calcite ($CaCO_3$) with monohydrocalcite ($CaCO_3 \cdot H_2O$) |
| 10–15 | Calcium zinc hydroxide ($CaZn(OH)_3)_2 \cdot 2H_2O$) (see Fig. 3) |
| Plate-like deposits in surface region (0–5 cm) | Portlandite ($Ca(OH)_2$), with some Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$, and calcium silicate ($Ca_3SiO_5$) with calcite ($CaCO_3$) rich surface (see Fig. 4) |
| Bulk sample from ∼0.5 m below surface | Predominantly amorphous with no clearly identifiable crystalline phases present (see Fig. 5). |

contained calcium zinc hydroxide ($Ca(Zn(OH)_3)_2 \cdot 2H_2O$) with no other crystalline phases being detected. In the presence of sufficient $Ca^{2+}$ ions in solution, zinc hydroxy anions $Zn(OH)_3^-$ and $Zn(OH)_4^{2-}$ can form ($Ca(Zn(OH)_3)_2 \cdot 2H_2O$). This is reported to have a key role in inhibiting cement hydration of Zn-containing cement systems as it completely coats cement grains, forming a barrier and preventing further reaction and setting [14].

XRD data of the plates of material found near the upper surface of the S/S waste is shown in Fig. 4. This indicates that these were massive deposits of predominantly portlandite ($Ca(OH)_2$) with some calcite, ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$) and calcium silicate ($Ca_3SiO_5$). The distinctively different much darker top surface layer of the plates was calcite ($CaCO_3$).

Representative XRD data of the bulk sample taken from approximately 0.5 m below the surface of the exposed S/S waste is shown in Fig. 5. It is very difficult to unambiguously identify any crystalline phases present in this material from this type of data. The low count rate indicates that this region is much more amorphous than other samples analysed and it appears to be very different from the material found nearer to the surface of the environmentally exposed S/S waste.

The results of XRD analysis of all the samples are summarised in Table 2. Apart from calcite and ettringite in the surface region, none of the crystalline phases found in the original 28-day samples were detected in the environmentally exposed S/S waste.

## 4.2. Chemical composition

The concentrations of hazardous metals found in the different samples of the S/S waste are given in Table 3. Each data is the average concentration obtained from analysis of three homogenised samples taken from each region.

There are significant concentrations of Zn, Fe and Cr in all the samples, although these are lower than expected based on the original composition of the metal-plating waste filter cake. The data indicates significantly reduced concentrations of these metals in the top 0–5 cm compared to analysis of samples taken from further into the S/S waste. It is possible that this results from the inherent variability in the composition of the S/S waste. However, the consistency of the data from the different cores suggests that this results from environmental
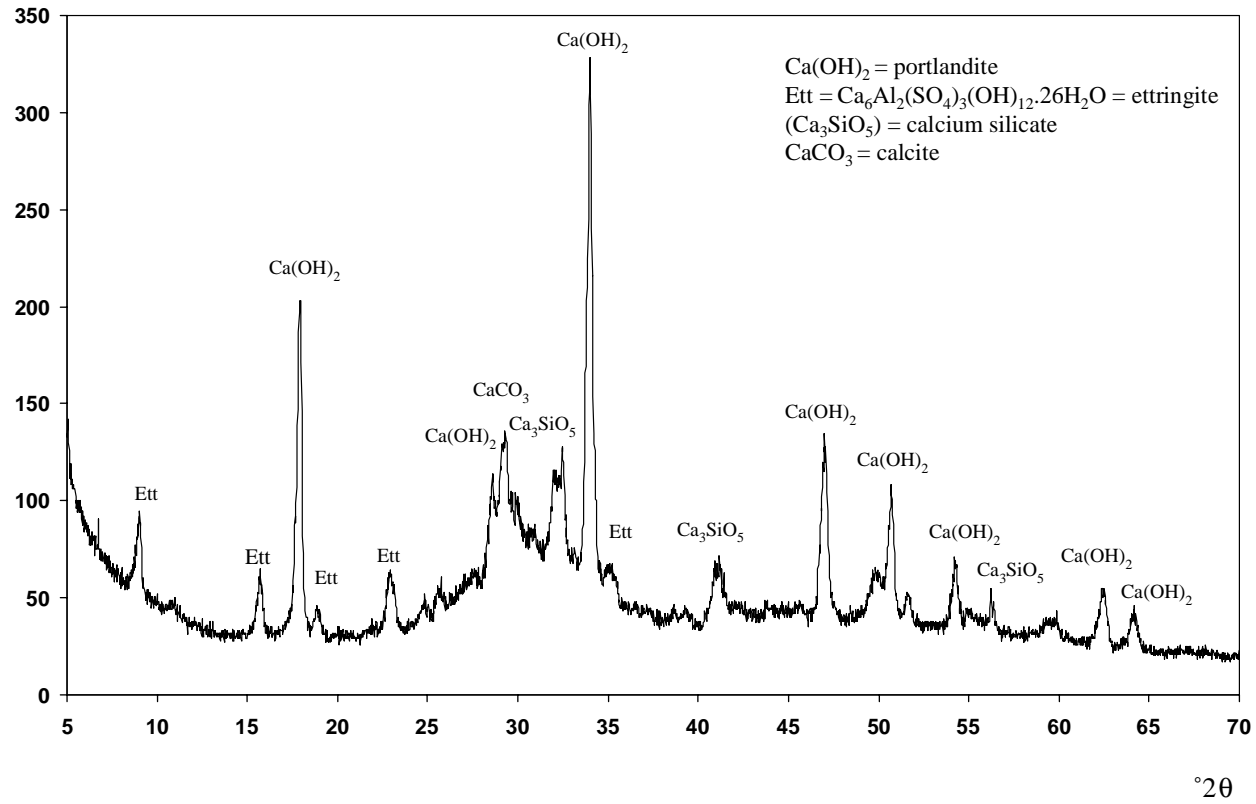
counts/s



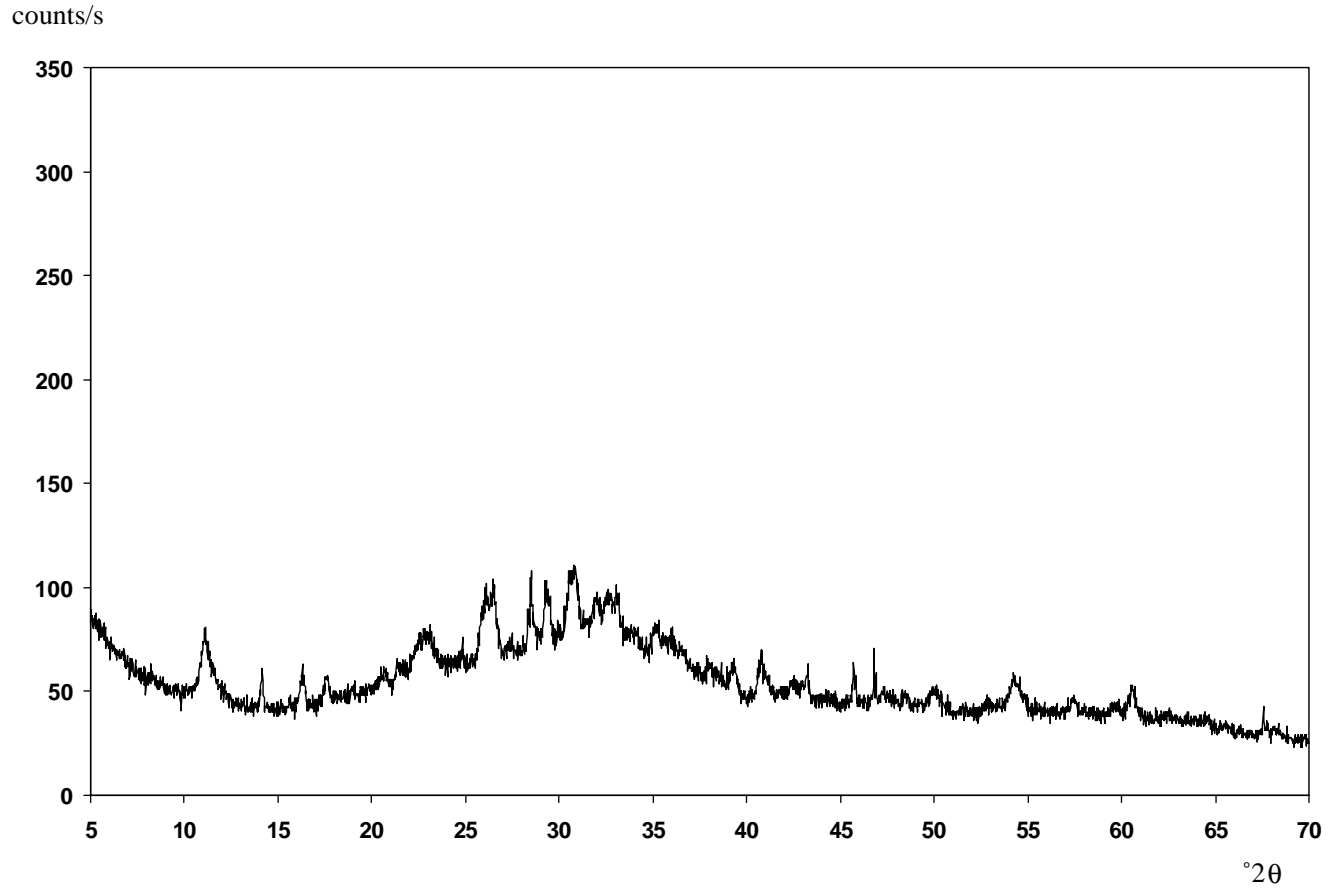Fig. 4. XRD data of the plate-like deposits found near the S/S waste surface.

counts/s



Fig. 5. XRD data for bulk S/S waste taken from ∼0.5 m below the surface.

Table 3
Concentrations[a] of hazardous metals in the 10-year-old environmentally exposed S/S waste (mg kg$^{-1}$)

| Metal | Top region exposed (0–5 cm) | | | Top region exposed (5–10 cm) | | | Top region exposed (10–15 cm) | | | Bulk (∼0.5 m) exposed |
|---|---|---|---|---|---|---|---|---|---|---|
| | Core 1 | Core 2 | Core 3 | Core 1 | Core 2 | Core 3 | Core 1 | Core 2 | Core 3 | |
| Cr | 2734 | 3676 | 4034 | 4750 | 4258 | 4460 | 4714 | 4606 | 4924 | 4880 |
| Cu | 72 | 108 | 108 | 118 | 102 | 124 | 108 | 104 | 130 | 138 |
| Fe | 21618 | 24132 | 30668 | 36592 | 27156 | 32304 | 33774 | 28460 | 35940 | 43220 |
| Ni | 58 | 76 | 80 | 78 | 74 | 82 | 74 | 76 | 84 | 74 |
| Pb | 64 | 80 | 84 | 88 | 84 | 84 | 90 | 86 | 90 | 98 |
| Zn | 26290 | 33426 | 37794 | 45068 | 37856 | 40710 | 41052 | 38870 | 43048 | 63126 |

[a] ICP-AES analysis data is average from digests of three homogenised samples.

exposure and ageing effects. The particularly high concentrations of Zn, Fe and Cr in the bulk samples may also result from inherent variability in the composition of the original S/S waste. However, this may also result from metal migration/leaching away from the surface into the bulk of the S/S waste.

### 4.3. Leaching properties

#### 4.3.1. Acid neutralisation capacity (ANC)

Representative ANC data for the different samples is presented in Fig. 6. The bulk samples of S/S waste had high ANC, particularly at low acid additions while the ANC of the 0–5 cm surface material is significantly reduced, indicating removal of alkali species results from
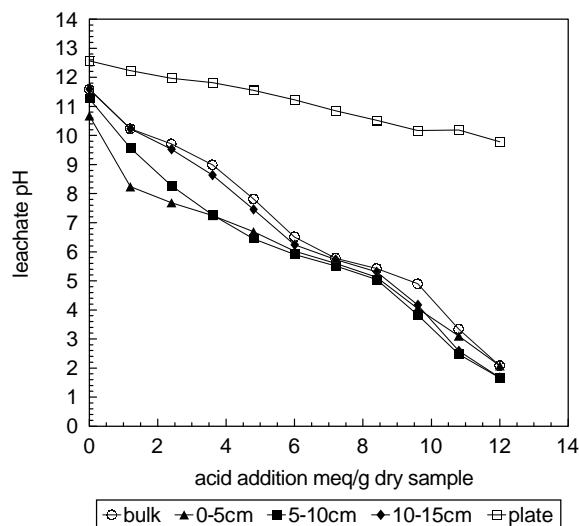


Fig. 6. Acid neutralisation capacity (ANC) data for different samples taken from environmentally exposed S/S waste.
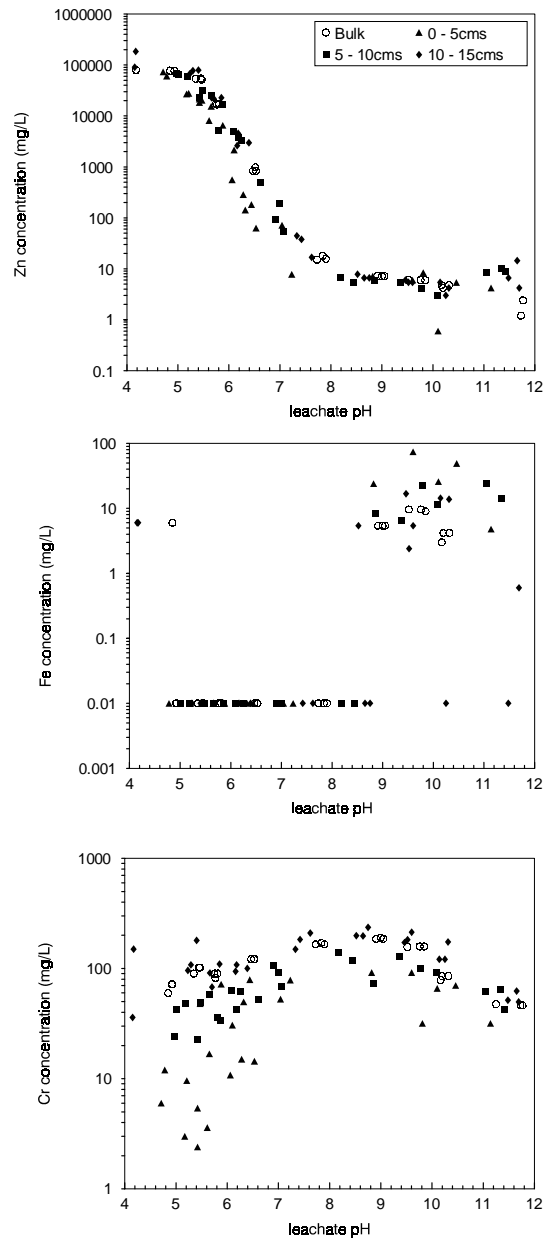
Fig. 7. Leaching data for: (a) Zn; (b) Fe; and (c) Cr for different samples of exposed S/S waste.
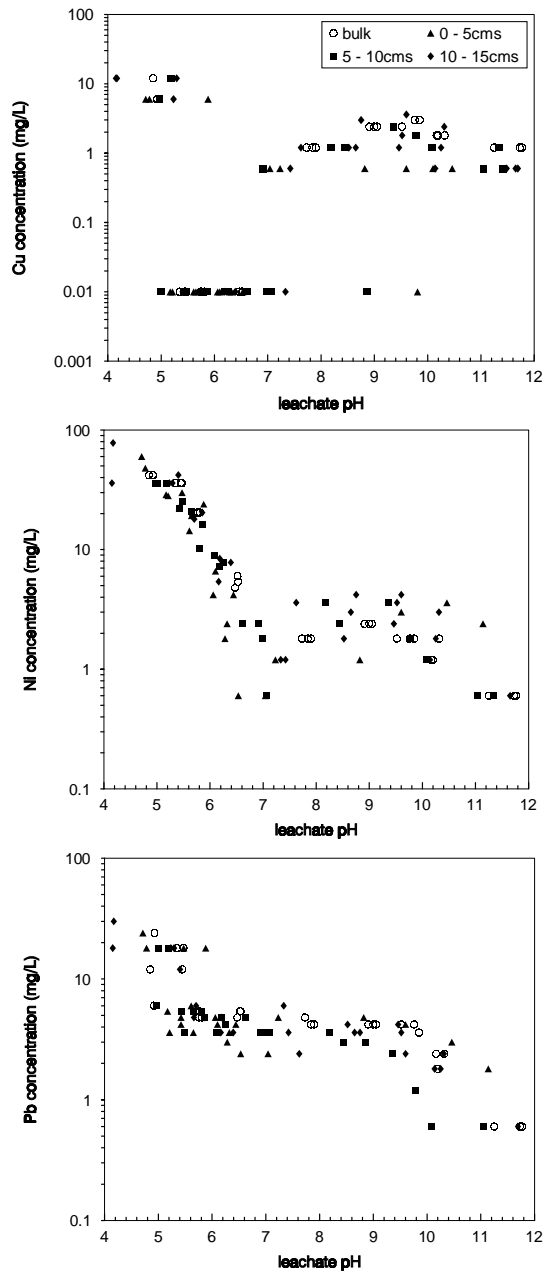
Fig. 8. Leaching data for: (a) Cu; (b) Ni; and (c) Pb for different samples of exposed S/S waste.

environmental exposure. The ANC appears to increase with depth into the S/S waste, with the 10–15 cm sample showing very similar behaviour to the bulk material.

Fig. 6 also includes ANC data for the predominantly portlandite ($Ca(OH)_2$) plate deposits that were found in the surface region (0–5 cm). This suggests an important effect of environmental exposure on the S/S waste is redistribution of ANC within the surface region resulting from leaching, migration and subsequent precipitation of alkali species and particularly $Ca(OH)_2$.

### 4.3.2. Metal leachate concentration data

Analysis of leachates produced by the ANC test provides metal release data as a function of leachate pH. Results for Zn, Fe, and Cr are given in Fig. 7, as these were the major heavy metals present in the field site S/S waste. Leaching data for Cu, Ni and Pb, the other heavy metal contaminants of concern are given in Fig. 8.

Zn release follows the type of leachate pH dependent behaviour previously reported for Zn-containing S/S wastes and other materials, with rapidly increasing concentrations leaching under acid conditions. However, the concentrations leached are generally high ($1–10\,mg\,l^{-1}$), even under pH conditions where minimum solubility of zinc hydroxide (pH $\sim 8.5–10$) would be expected. The behaviour of samples from different regions of the S/S waste were similar except that the 0–5 cm surface material leached significantly less Zn in the pH range 6–7.

High levels of Cr were leached from all samples, with concentrations typically ranging from 10 to $100\,mg\,l^{-1}$ over a wide pH range. The data shows a trend in leachate concentration at a specific leachate pH, with the surface (0–5 cm) samples leaching the least, the 5–10 cm samples consistently more, while the 10–15 cm and the bulk sample leach similar and high concentrations. Leaching from all the environmentally exposed samples is also much higher than expected based on the original TCLP results obtained on 28-day-old samples. These showed that under the TCLP test conditions Cr leached at concentration of $3.89\,mg\,l^{-1}$ at leachate pH 7.5, and although the different test conditions makes direct comparison of results problematic, increased leaching from the environmentally exposed and aged S/S waste is suggested.

Fe generally leached at concentrations in the range between 2 and $20\,mg\,l^{-1}$ at leachate pH levels above approximately 8.5. No significant leaching then occurred until the leachate pH was below 5. Similar leaching behaviour was observed for Cu. Under alkali conditions, Cu leaches at about $1\,mg\,l^{-1}$ and no leaching was observed between leachate pH values of approximately 7 and 5.5. There appears to be increased Cu leaching from the bulk and 10–15 cm samples compared to samples taken from near the surface.

Low concentrations of Ni leach under high pH, but leaching significantly increases as the pH falls below 6. Pb was found to leach between 1 and $10\,mg\,l^{-1}$ over a broad range of leachate pH conditions.

## 5. Discussion

This investigation has provided data on the composition, microstructure and leaching of a specific type of S/S waste after exposure to the environment for approximately 10 years.

The exposure may be regarded as a 'worst case' in which any landfill cover material has been removed, and under these conditions, the top exposed surface has experienced a range of stresses not necessarily encountered by properly landfilled S/S waste.

Exposure to the environment has significant effects on the properties of the S/S waste, particularly in the near surface region. The combination of wet/dry, freeze/thaw cycling and other environmentally induced stresses has caused extensive structural degradation to a significant depth. An unexpected feature of the exposed S/S waste was the precipitation of massive plates of $Ca(OH)_2$ that were found just below the surface. The formation of these is believed to result from the high concentrations of $Ca^{2+}$ and $OH^-$ and other ions leached into the pore water when the structurally degraded S/S material becomes saturated. As the surface region dries from the top exposed surface, moisture movements combined with evaporation increases pore water ion concentrations, resulting in formation of the plate-like deposits of $Ca(OH)_2$ observed. This leaching and re-precipitation of readily soluble alkali metals and associated anions causes redistribution of acid neutralisation capacity in the top region of the exposed S/S waste. Above the plates the S/S waste is extensively carbonated and as a result the acid neutralisation capacity is significantly reduced. The plates have extremely high ANC due to the $Ca(OH)_2$ and below these the S/S waste composition is largely dominated by hydroxide species that produce high and stable ANC conditions.

The reduction in concentration of heavy metals found near the surface of the S/S waste indicates that significant mobility and redistribution of elements has occurred. The reduced ANC from the effects of repeated saturation and carbonation of the surface region means that metal leaching can occur, with metals removed from the S/S waste system in leachate overflowing from the surface. Alternatively solubilised metals may migrate further into the bulk of S/S waste possibly due to gravitational sedimentation. This is supported by the apparent increase in metal concentrations observed in the bulk samples.

The data also indicates significant differences between the original 28-day laboratory cured S/S waste samples and the environmentally exposed S/S waste. The absence of alite, belite and portlandite from the bulk exposed S/S waste, given that the binder used was a blended cement (50% OPC and 50% PFA), might suggest complete hydration had occurred. However, this would be associated with the development of significant mechanical strength in the 10-year-old S/S waste. The fact that the material rapidly disintegrated into slurry when cores were taken using diamond-cutting equipment indicates insignificant strength development. This is due to the long-term inhibiting effects of metals and particularly Zn and Pb present in the waste filter cake on cement and PFA hydration reactions.

Performance testing of S/S wastes is normally completed on laboratory prepared samples cured for 28 days. This is based on the assumption that the material tested is representative of S/S waste after disposal. The results from this study show that this assumption is not necessarily correct. The S/S waste material after 10 years environmental exposure was very different from that present in 28-day laboratory cured test samples. The leaching properties of material tested after 28 days also seem to be very different from leaching determined on environmentally exposed S/S waste. This raises the issue of the relevance of data obtained from 28-day laboratory prepared and cured samples to long-term S/S waste performance.

While understanding the long-term performance, durability and degradation mechanisms in S/S wastes remains a key research need, it is clear that each type of S/S waste and disposal site combination represents a relatively unique scenario. When this is combined with the

inherent inhomogeneity associated with S/S wastes deriving unambiguous data and generic conclusions from investigations of S/S waste after disposal becomes highly problematic.

## 6. Conclusions

A metal-plating filter cake waste treated by stabilisation/solidification using ordinary Portland cement and pulverised fuel ash has been characterised after exposure to the environment in SE England for approximately 10 years. The major conclusions from the work are

1. The surface region of the S/S waste, to a depth of ∼5 cm, was severely degraded and had a soil-like consistency. It was extensively carbonated and had reduced acid neutralisation capacity compared to bulk samples.
2. Large 'plate-like' deposits of calcium hydroxide with a calcium carbonate rich top surface had formed close to, but generally below the environmentally exposed S/S waste surface.
3. S/S waste taken from the bulk of the environmentally exposed material was complex, appeared to be amorphous and retained high acid neutralisation capacity. Calcite was the only crystalline phase detected in both exposed and the 28-day samples.
4. Metal analysis of homogenised samples from different depths into the S/S waste indicates significant reduction in the concentration of Zn, Fe, Pb and Cr in the surface region.
5. Deriving conclusions on the performance and behaviour of S/S waste by post-disposal analysis is problematic. This is due to the lack of adequate characterisation data that normally exists on the original S/S material and the inherent variability and inhomogeneity of S/S wastes. Microstructural changes observed are also specific to the particular S/S waste and disposal scenario investigated.

## Acknowledgements

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