TRANSPORT RESEARCH LABORATORY

The effects of age on cement stabilised/ solidified contaminated materials

Prepared for Quality Services, Civil Engineering Division, Highways Agency

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Scope of the project

The Highways Agency (HA) is concerned about the cost and resources required to construct highways across areas of contaminated land. Many innovative methods of processing contaminated land have been developed in recent years. These methods are generally not yet being applied to highways, although a highway is one of the least sensitive end uses for contaminated land. The Transport Research Laboratory (TRL) was therefore commissioned by HA in September 1995 to establish which existing methods of processing contaminated land are economically and environmentally acceptable for highway works. The work described in this report forms part of this larger project, and deals with the use of stabilisation/ solidification techniques to treat contaminated materials.

Summary

At an early stage in the project the use of solidification/ stabilisation (S/S) techniques using inorganic cementitious agents was identified as a potentially important method for highway schemes. Experimental work at TRL has concentrated on the use of lime as a cementing agent for fine-grained materials such as sludges. There was a need for case studies utilising other materials to provide a balanced picture, particularly of the long-term durability of the treated material. An opportunity to investigate some slabs of metallic slag stabilised using cement and the proprietary agent Geodur arose through contact with Portsmouth University. The slabs had been placed in 1994 and left open to the weather as part of the CIRIA demonstration and case study programme on the remediation of contaminated land (Jardine and Johnson, 2000). A considerable amount of mechanical and environmental test data had been obtained at the time of placement and over the following 15 months, and this was kindly made available by CIRIA and VHE Construction plc, the owners of the site.

A total of six materials had been treated by the S/S process, comprising two soil/slag mixtures, pulverised fuel ash (as a control), two metal-working slags and a contaminated soil. Part of one of the soil/slag mixtures was later excavated and crushed to a grading suitable for Type 1 sub-base. The materials were sampled in March 1998, 1216 days after they were placed. Mechanical and environmental tests were carried out to repeat the tests which had been carried out in the CIRIA programme. Further tests were conducted to determine the permeability of the stabilised materials and to assess the suitability of the crushed material for use in highway applications.

The results show that for all materials the unconfined compressive strength increased with time. The weakest material was the contaminated soil, which had a significant clay content. All the other materials had average strengths at 1216 days of greater than 9 N/mm^2, and the 28 day strengths exceeded the waste regulatory authorities requirements for S/S materials. However, the early strength (< 7 days) of the materials was low. This suggests that the organic and inorganic contaminants within the materials delayed the initial hydration reactions in the cement. This low early strength could limit the use of these materials as cement-bound materials in road construction.

The crushed soil/slag S/S material was shown to be suitable for highway earthwork Class 1 and 6 applications, as Type 1 sub-base and as an aggregate in a cement-bound material.

The materials were analysed to determine their total chemical content and subjected to the NRA leaching test method. Analysis of the leachate showed metals Cd, Hg, Ni, and Zn were effectively immobilised, as were low concentrations of hydrocarbons. Metal contaminants Al, Cr, Pb and organic contaminants phenols and total polycyclic aromatic hydrocarbons (TPAHs) leached from one or more of the treated materials. In some cases, the concentrations were higher in leachate from the S/S materials than in leachate from the untreated materials. This echoes the findings of work on materials treated with lime (Reid and Brookes, 1999). The results were compared with environmental quality standards and published dilution factors, using the system proposed in Baldwin *et al.*(1997). This showed that four of the S/S materials were classed in Group 1, for which there are no restrictions to use in highways based on potential to affect water quality. The other two S/S materials were classed in Group 2, for which some restrictions to use in highways may be required based on potential to affect water quality.

The trial was not designed specifically to assess the suitability of the S/S materials for use in highway applications. Nevertheless, it has provided valuable data on the properties and behaviour of contaminated materials treated with inorganic cementitious agents, which can be used to assess their potential for a number of applications. In particular, this work has shown that the strength of the S/S materials does not decrease over the time period investigated and there is no indication of any significant release of contaminants. Water samples from drains on the site were of high quality, indicating no significant leaching is occurring.

1 Introduction

A number of environmental issues are now having a financial impact on the construction industry. The environmental costs of waste disposal by landfill were addressed on the 1st October 1996 by the introduction of a new environmental tax. Quarrying of primary aggregate is considered as a likely target for environmental taxation. The use of environmental taxation has and will provide financial incentives for a more sustainable use of resources by the construction industry. Increasing demand on land resources for development has resulted in UK government policy requiring the greater utilisation of derelict and contaminated land. To meet these recent market requirements, new technologies for the remediation of contaminated land and the recycling of construction materials are emerging.

One available technology is the stabilisation and solidification (S/S) of contaminated land and industrial by-products, using cement and additives. Chemical contaminants treated by S/S technology may be both chemically altered (stabilised) and physically bound within a cement matrix (solidified). S/S technology has been applied to remediate a number of contaminated sites in the UK. However the use of S/S technology in the UK has been on a small scale compared to use in the USA. It is estimated that 30% of contaminated sites in the USA have been remediated using S/S technology (Jones and Hopkins, 1997).

The future use of S/S technology in the UK is predicted to increase due to implementation of the EC directive on landfill, which will ban the land filling of liquid hazardous waste by 2002 (Ends Report, 1998). S/S is considered to be a possible solution to this problem as the process uses established technologies and is cost effective. The process is also applicable to contaminated land as it avoids the creation of concentrated waste residues with associated high transportation / disposal costs and improves the ground engineering properties of the remediated site.

Several issues have been identified that may have contributed to the slow acceptance of S/S technology in the UK. These issues are:

- Concerns of owners and developers that contaminants remain on a site and may affect future changes in the use of the site.
- The effects of age and weathering on the integrity of S/S materials are not well documented.
- As S/S technology uses cement, a non-renewable resource, the resulting S/S material should be utilised in a sustainable application.

This project was initiated to investigate two of the above issues: namely the effects of age and weathering on the chemical and physical integrity of S/S materials and the potential for reuse of S/S materials in applications such as road construction.

A trial was conducted using six contaminated soil and industrial by-product samples treated with a proprietary additive, Geodur. Geodur is a S/S technology available from VHE Construction plc. The first phase of the trial was organised and managed as part of the CIRIA demonstration and case study programme on the remediation of contaminated land. The CIRIA trial subjected exposed slabs of S/S materials to natural weathering and leaching over an 18 month period.

Core samples of the treated materials were taken at time intervals and tested for changes in strength and the leaching of contaminants. The results of the 18 month trial are compiled in CIRIA Funders Report RP489/2 (Jardine and Johnson, 2000). This phase of the S/S material trial is termed Phase 1. The data collated from Phase 1 of the trial has been released by CIRIA for inclusion in this report.

The second phase of testing commenced during March 1998. Phase 2 of the trial conducted testing on 40 month old S/S material samples collected from the trial site. Phase 2 of testing was undertaken by a joint collaboration between VHE Construction plc, University of Portsmouth and the Transport Research Laboratory (TRL). This second phase of the trial arose independently of the CIRIA trial. Funding for Phase 2 testing of the S/S material was provided by the Highways Agency, via TRL research project E199D/HG: Processing contaminated land. S/S is a technique that could find widespread application for the treatment of contaminated materials during highway construction schemes.

This report details the findings of the physical and chemical tests conducted on the six materials and compares the results to available environmental quality standards, highway specifications for materials in road construction and previous research findings on the use of untreated industrial by-products in road construction.

As with many S/S technologies the Geodur additives used in the S/S process are subject to commercial confidentiality. Therefore the report the does not investigate how the Geodur additives performed or the effectiveness of the S/S process compared to Portland cement alone. This report suggests how a cement based S/S technology may perform over time, when exposed to natural weathering.

2 Aims and objectives

The primary aim of this study was to assess the variation in physical and geochemical properties of cement S/S materials with time.

Two secondary objectives were identified as:

a To understand the leaching behaviour of the materials and identify mechanisms for the leaching of contaminants.

b To identify potential applications for the reuse of S/S materials for highway construction.

3 A review of stabilisation/solidification technology research

The use of cement, lime and pozzolanic materials (termed binders) to stabilise and solidify hazardous waste has a long history, particularly in the USA where use of binders dates back to the mid 1960's. The USA Resource Conservation and Recovery Act 1976 enforced the use of

S/S technology to stabilise and solidify liquid hazardous waste before landfill disposal and prohibited selected wastes from disposal to landfill altogether (Wiles, 1987). Cement technology is also accepted as an effective method to stabilise / solidify US industrial and municipal waste (Lo, 1996). Published research and guidance on the application of S/S technology is predominantly American.

3.1 Terminology used by S/S technology

The terminology used in S/S literature has been defined by Wiles (1987) in a review of S/S technology. S/S technology is defined as two separate processes:

- Stabilisation results in chemical changes in a contaminated material that produce chemically more stable components i.e. by precipitation of soluble metal ions out of solution. This process may not result in an improvement of the physical properties of the material.
- Solidification results in reduced fluidity and friability of a contaminated material, preventing access by transport processes to contaminants contained in the solid product.

The processes may be applied separately, but most commonly the technology operates by joint application of the two processes. The review by Wiles proposes categorisation of S/S processes by the type of binder used. Cement, lime, pozzolans, gypsum and silicates are referred to as inorganic binders. Organic binders reported include epoxy, polyesters, asphalt / bitumen, polyolefins and urea-formaldehyde.

A review of S/S technology has been produced by the UK Construction Industry Research and Information Association (CIRIA, 1995). This report gives a general overview of S/S technology but is based mainly on experience from North America.

3.2 Binders used by S/S technology

There is a wealth of literature available on laboratory scale research into the effectiveness of a wide range of S/S mixes, used to treat inorganic and organic contaminants. The majority of binders described in the literature are inorganic. The predominant binders used are pulverised fuel ash (PFA), ferrous slag, lime and cement or a combination (Madrid et al, 1997). As the contaminated soils and industry by-products examined by this trial were treated using a Portland cement based treatment, this review will be restricted to Portland cement binder.

3.3 The cement binder S/S process

The use of a cement binder to stabilise and solidify contaminated material is based on the hydration reactions of four principal compounds found in Portland cement. The four compounds are tricalcium silicate (C_3S in cement notation) the main constituent of Portland cement, dicalcium silicate (C_2S), tricalcium aluminate (C_3A) and calcium aluminoferrite (C_2AF). Gypsum may also be an added constituent (Odler, 1998).

On mixing, water from the untreated contaminated material (or added water) chemically reacts with the anhydrous Portland cement to form calcium silicate hydrate (C-S-H), calcium hydroxide and aluminate compounds. This reaction is termed hydration. The hydration of cement liberates heat due to exothermic chemical reactions. The release of heat fluctuates with time. Within minutes of the water and cement mixing, a short lived heat peak is generated followed by a dormant period. A second, main exothermic peak is evolved after a few hours. The rate of heat release then slows down over a few days to very low levels (Odler, 1998).

The cement hydration products initially set and then harden to form a cement paste that binds the liquid and solid components of the contaminated material. The solid phases in a contaminated material act as an aggregate and are bound by the hardened cement paste to form a low strength concrete.

3.4 Mechanisms for treatment of contaminants by S/S technology

Chemical contaminants treated by S/S technology may be both chemically altered (stabilised) and physically bound within the cement matrix (solidified). There are a number of mechanisms by which contaminants can be held in the cement matrix, see Figure 1. A contaminant may be incorporated into the chemical structure of a hydration product or form inclusions within the hydration products. Precipitation and sorption onto the high surface area of a hydration product or cement grain may also occur. Contaminants, particularly organic compounds such as phenol may be soluble in water and can be held in the free pore water of the hardened cement paste. The pore water fraction contains the most easily leached contaminants retained by a S/S material (Cocke, 1990).

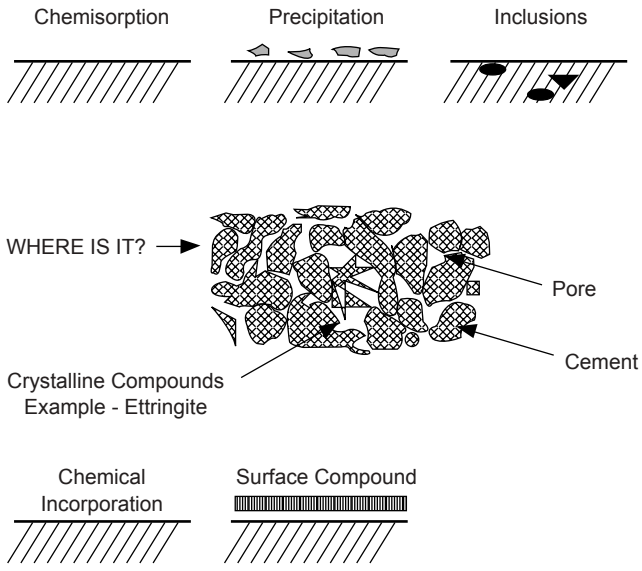


Figure 1 Stabilisation/solidification mechanisms (after Cocke 1990)

3.5 Factors affecting the physical properties of a S/S material

A key factor controlling the physical characteristics of a hardened cement paste is the water / cement (w/c) ratio. The initial starting w/c ratio of a cement mix controls the eventual porosity and strength of hardened cement. Porosity has been shown to increase with increasing initial w/c ratio. In a water saturated, hardened cement paste the volume of pores is equal to the volume of free water (Odler, 1998). A w/c ratio of above 0.48 has been considered detrimental to the final porosity and strength of a S/S material (Means et al, 1995). However the w/c ratio is not the only factor that influences the structure and porosity of a S/S treated material.

3.5.1 Cement/aggregate interface

The structure of a Portland cement / aggregate concrete differs from that of a hardened cement paste. A complex transitional zone exists between the hardened cement paste and the surface of an aggregate clast. The aggregate surface is coated with a film of calcium hydroxide with rod shaped C-S-H gel particles covering the calcium hydroxide film. This assemblage is called the duplex film. The hardened cement paste in the vicinity of the duplex film is more porous than the bulk of the cement paste (Odler, 1998). The chemical properties of the aggregate are also predicted to influence the microstructure of the cement paste / aggregate interface (Tasong, 1998).

It can be seen that the physical properties and strength found in a hardened cement paste, produced at a known w/c ratio cannot be extrapolated to a heterogeneous, contaminated material treated by a S/S technology. The actual w/c ratio used during the S/S process will be dependent on criteria such as the required workability of the pre-set S/S material (Glasser, 1992). S/S treated contaminated soils with high w/c ratios have been shown to develop higher strength due to better mixing than S/S materials with lower w/c ratios (Evans and Al-Tabba, 1997).

3.5.2 Interference to Portland cement hydration by additives and contaminants

It has long been known that the properties of fresh and hardened cement can be altered by the addition of certain chemical additives to a cement / water system (Young, 1972). These additives can be used to retard or accelerate the setting and hydration of Portland cement. There are three categories of additive that may affect the cement / water system:

- Surfactants affect the cement / water system immediately upon addition by influencing the surface tension of the water and by absorbing onto the anhydrous cement grain surfaces.
- Set controlling additives affect the cement hydration reaction after a time elapse of minutes or hours.
- Mineral additives affect the rheology of the fresh concrete.

Organic additives such as lignosulphonic acids, citric acids and gluconic acids are mostly used as plasticisers and water reducing agents. These additives lessen the interparticulate attraction between cement grains allowing greater dispersion, thereby reducing the amount of hydrating water required. This results in a lower w/c ratio and hence higher strength of the cement paste. The controlled effects of chemicals on the hydration of cement have been used as a basis for understanding the effects produced by contaminants on the strength of a S/S material (Pollard et al, 1991).

3.5.3 The effect of contaminants on cement hydration

The use of chemical additives can be beneficial to the final properties of a hardened cement paste. However the presence of organic and inorganic contaminants often has a deleterious effect on the hydration of Portland cement (Akhter et al, 1990). The strength of cement is largely dependent on the formation of hydrated C-S-H. Organic and inorganic contaminants can effect the hydration process and reduce the amount of C-S-H formed, resulting in reduced strength of the hardened cement paste. To date the effects of individual contaminants on cement hydration are poorly understood (Pollard et al, 1991).

3.5.3.1 Inorganic contaminants

The effects of metals on the hydration of anhydrous cement are dependent on the individual metallic element and its compounds present. This contrast has been shown by Cartledge et al (1990), in a study of lead and cadmium hydroxide. The two hydroxides were mixed with Portland cement and hydrated. An uncontaminated Portland cement was used as a control. The study indicated that cadmium hydroxide formed a nucleation site for the precipitation of C-S-H and calcium hydroxide which formed an impervious coating. However lead hydroxide was believed to form an impervious coating around the anhydrous cement grains that retarded hydration. During hydration, changes in pH caused the lead hydroxide to dissolve and then reprecipitate as salts on the surface of the hydrated cement grains. These exposed salts were more soluble than the lead hydroxide and were easily leached (Cartledge et al, 1990).

Using a scanning electron microscope to observe a copper oxide and tricalcium aluminate (C_3A) system, Lin and Huang (1996) also showed how copper oxide was microencapsulated by the hydration product of C_3A. This may indicate that copper oxide acted as a nucleation site for C_3A hydration products.

3.5.3.2 Organic contaminants

As with metal contaminants the individual type of organic contaminant can have markedly different effects on the hydration of Portland cement. A review produced by Pollard et al (1991) of the mechanisms and processes occurring within cement based S/S systems contaminated by organics, concluded that minor amounts of certain organic contaminants can produce significant structural changes to the properties of hydrating cement. The review also found that non-polar organics did not hinder strength development in cement systems, but the need for more research on the effects of chlorinated hydrocarbons was

suggested. Hydroxy, (alcohols, sugars) carboxylate (acids) and amine functionalities were expected to impair cement hydration. The effects of organic contaminants on a S/S material may also be specific to the binder used. Increasing concentrations of organic contaminants in a S/S material have been shown to have less effect on the long term strength of a S/S material bound with Portland cement than materials bound with cement / PFA and lime / PFA binders (Cullinane and Bricka, 1989).

3.6 Summary of research to-date on S/S technology

S/S technology is used primarily in the USA to treat industrial and municipal wastes. The cement based S/S process operates by chemically and physically locking contaminants into a cement bound matrix. The most commonly used binders for S/S treatments are those of the inorganic group. The hydration of cement comprises a complex series of reactions that may be greatly affected by the presence of organic and inorganic contaminants. The interaction of multiple contaminants with the hydrating cement system and the effects of age on S/S materials are currently poorly understood and little researched.

4 History of the S/S material trial and summary of equipment, process and sites used

A trial of the Geodur S/S treatment process and resulting S/S material arose during the remediation of a contaminated land site at Wath Manvers, South Yorkshire. The Wath Manvers site had had a complex history as a former railway marshalling yard and as a site for metal recycling / smelting plants. Industrial by-products including nickel electrodes used in the manufacture of oil products were brought from the USA to the site for recycling. Concentrated copper solutions were also known to have leaked into the ground over a long period of time. The ground conditions consisted of a coarse grained contaminated soil and slag as made ground to a depth of 1.5m. The Wath Manvers site was remediated by removal of the contaminated material to landfill. The remediation was undertaken by VHE Construction plc as a contractor (Jardine and Johnson, 2000).

VHE Construction plc holds the UK licence for the Geodur stabilisation / solidification system from the technology providers, the Swiss company Geodur CIS. The Geodur system uses additives and cement to stabilise and solidify contaminants. This ex-situ process is suitable for treatment of both contaminated soils and industrial by-products. A combination of factors led to the initiation of the Geodur system trial:

- Commencement of the CIRIA demonstration and case study programme on the remediation of contaminated land.
- Close proximity of the Wath Manvers remediation project to land owned by VHE Holdings plc.
- Availability of the Geodur process from VHE Construction plc.

The Geodur system trial was organised and managed as part of the CIRIA Demonstration and Case Study Programme on the Remediation Of Contaminated Land. This first phase of the trial occurred between December 1994 and July 1996 and is termed Phase 1. The data collated from Phase 1 of the trial were released by CIRIA for inclusion in this report.

A second phase of testing (termed Phase 2) of the S/S material was undertaken between January 1998 and February 1999 by collaboration between VHE Construction plc, University of Portsmouth and the Transport Research Laboratory (TRL). Phase 2 of testing commenced during March 1998 and used the same analytical laboratory and methods as Phase 1 in order to reduce variability between data sets.

4.1 Materials treated

Phase 1 of the trial comprised the collection of six contaminated soil and industry by-products at the Wath Manvers site. The six materials are described in Jardine and Johnson (2000). Sieve analysis data of the untreated materials are provided in Jardine and Johnson.

Materials A and B
Two contaminated soil / slag made ground materials from the former metal recycling and processing plant, at Wath Manvers Yorkshire. Soil / slag material described as a clayey silty sand containing ash particles and slag ranging in clast size from sand to gravel. The physical characteristics of the two materials were the same, although material A was considered to be more contaminated than material B.

Material C
A lagoon conditioned pulverised fuel ash (PFA) from a coal burning power station. Light grey in colour.

Material D
A lead / zinc slag from smelting. Black glassy grit with sand sized particles.

Material E
A vertical retort metal smelting residue. A black ash formed into weakly cemented briquettes.

Material F
A contaminated gasworks soil / made ground. The material was described as a dark brown silty clay with debris including plastic bags, bricks and electrical cables, with an acrid odour and an oily appearance.

Materials A and B were passed through a 63mm screen to remove over sized lumps.

4.2 S/S treatment of the contanminated materials

The two most important aspects of any S/S treatment process are the binder mix design and the physical mixing of the contaminated material with the binder. Formulation of the mix design, i.e. ratio of contaminated material to binder, binder composition and water / cement ratio is critical to achieve the optimum performance of a S/S material. The standard practice for mix design is to produce a number of trial mixes and establish the most effective mix for a contaminated material (CIRIA, 1995). Due to the short time available for the trial this procedure

was not carried out. The mix design used was formulated on site, in conjunction with a Geodur representative present during the trial. The general trial mix design for all six contaminated materials treated is shown in Table 1.

Table 1 Mix design for the six S/S materials

S/S material constituent	*Percentage by weight*
Contaminated material A-F	54
Limestone aggregate	18
Portland cement	10
Water	17.85
Geodur Tracelok	0.15
Water /cement ratio	1.8 : 1

There are two notable points about the mix design used. Limestone aggregate was added to the contaminated materials to improve the consistency and engineering properties of the pre-set mix. The use of primary aggregate in the mix design of a commercial scale process would not be desirable. Addition of primary aggregate leads to increased treatment costs, negative environmental effects and an increased volume of the S/S treated material. However the addition of industrial by-products or coarse grained contaminated soils from other locations may produce a superior strength S/S material.

Secondly the moisture content of the untreated contaminated materials ranged from 20% for material C to 3% for material D. The mix design required 18% water content. Additional water was added to contaminated materials with < 18% moisture content. The moisture contents specified by the mix design produced an apparently high maximum w/c ratio of 2.0 for material C and 1.8 for materials A, B, D, E and F. However not all water occurring in a contaminated material is freely available for hydration of the anhydrous cement grains. For example, water in a contaminated soil may be bound within the clay and gypsum minerals present. Therefore the actual w/c ratios of the six S/S materials may have been lower than apparent from the design mix, depending on the sorption of water by the untreated material constituents.

4.3 Processing of the contaminated material

The physical mixing of a contaminated material with a binder can be achieved by either in-situ or ex-situ processes. An ex-situ mixing process was used for Phase 1 of the trial. The equipment used was a mobile Belmix 50 batching plant (Plate 1) with a screw auger, continuous feed mixer. The plant operated at a production rate of 56.8 tonnes / hour. Jardine and Johnson (2000) concluded that the mix design and quality of the final S/S material produced was limited by the batching plant used. The treatment process took place between the 7th and 15th December 1994, on hard standing at the Wath Manvers remediation site. The pre-set treated material was then transported by truck to a second site where the S/S material slabs were cast and could be left, undisturbed for an indefinite time period.

4.4 Construction of S/S material slabs

An objective of the Phase 1 trial was to demonstrate that S/S material could be reused as a construction material. In order to test the long term suitability of the S/S materials as construction materials, exposed ground slabs were constructed (Figure 2, Plate 2). The material processed at Wath Manvers was immediately transported to land owned

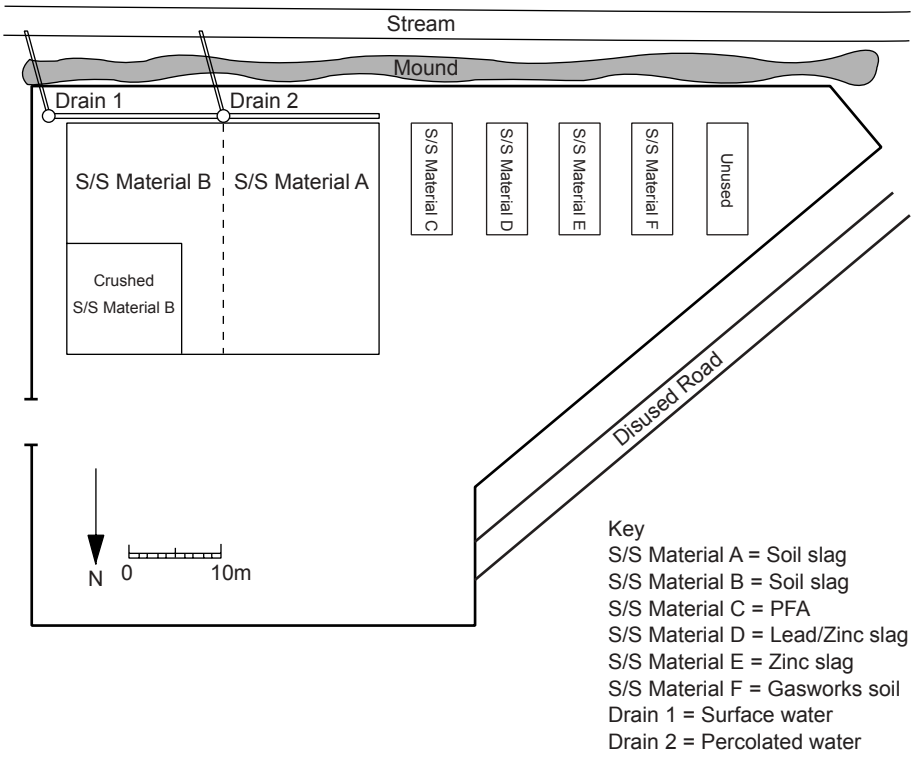


Figure 2 Site layout of slabs at Wombwell

by VHE Holdings plc at Wombwell, Yorkshire. On arrival at the Wombwell site the S/S materials were spread over the prepared ground using a tracked shovel and compacted using a vibrating roller.

The slab area at Wombwell had been prepared before the trial commenced. The site was graded to a low angle for drainage and the slab areas were excavated and lined with Visqueen™ polyethylene membrane. A main slab 35m x 25m x 0.3m thick was constructed from S/S materials A and B. Four slabs between 8 and 12m x 4m x 0.3m thick were constructed from S/S materials C to F. A series of drainage channels for percolated leachate were constructed under the main slab prior to lining with the membrane. The percolated leachate channels drained into a sump (Figure 2) at the side of the slab. Additional drainage for surface runoff was provided after the main slab had been constructed. Surface water was allowed to drain from the main slab into a drainage ditch before collection in a second sump, as shown by Figure 2.

The slabs were left uncovered to cure under prevailing weather conditions. After 1 year a quarter of the main slab was broken, crushed and compacted to test the suitability of the material as an aggregate. The section of slab crushed and compacted consisted of material B.

4.5 Description of the S/S material slabs at Phase 2 sampling

4.5.1 Materials A and B

The S/S material slabs were inspected on the 24th March 1998 prior to the Phase 2 core sampling. The surfaces of the six trial slabs showed varying degrees of degradation. However no vertical fractures within the S/S materials were observed. The surface of the main slab, constructed from materials A and B, was covered with moss and a layer of loose clasts up to 10mm in size. The clasts consisted of S/S material, brick, natural flint and stone. A soil horizon 15-50mm thick was found below the layer of clasts. When the soil was scraped away the surface of the S/S material was exposed. The depth of the soil layer varied across the slab. The southern end of the slab area, toward the stream had no soil layer and the clasts lay directly on the S/S material surface. The clast / soil layer is believed to originate from the disturbed surface left by the tracked shovel used to compact treated material (see Plate 2). On the second day of sampling (26th March 1998), overnight rain had left deep surface water puddles on areas of the main slab.

4.5.2 Material C

The surface of the material C slab was in good order, with areas of small, horizontally fractured plates of material between 10-20mm thick. The slab surface around core number 20 was fractured at a depth of 10mm. When stood on, the plate compressed and the fracture could be seen to close. A phenolphthalein test within the core hole showed the material C to be carbonated above the fracture. See Plate 6.

4.5.3 Material D

The surface of material D slab was covered in 5mm thick platelets, 30-40mm in diameter and with a fine soil like regolith, up to 40mm deep. The degradation of the surface appeared to be due to weathering. The surface of the slab was cleared by hand using a shovel to expose the unweathered surface of the S/S material. A calcareous like precipitate from the S/S material had formed on the Visqueen sheet at one corner of the slab.

4.5.4 Material E

The surface of material E slab showed the same surface degradation as material D. The plates were larger up to 200mm in diameter with soil regolith.

4.5.5 Material F

The surface of material F slab showed the least degradation of all six trial slabs. The surface was hard and had no surface debris. However at depth, areas of the slab material showed varying degrees of solidification. At the southern end of the trial slab solidification diminished with depth, the base of one core had the texture of a clay with aggregate.

4.6 Physical and chemical sampling of the S/S material slabs

4.6.1 Phase 1 – treated and untreated material samples

The untreated contaminated feed stock was sampled at hourly intervals during processing.

24 x 100mm test cubes were made from pre set material A.

12 x 100mm test cubes were made from pre set materials B, C, E and F. No cubes of material D were made because the processed material did not set for a considerable time.

An unknown quantity of 150mm diameter cores were taken from each S/S material slab for testing at 3, 6 and 15 month intervals.

Two water samples were taken from the adjacent stream, one water sample upstream and one water sample down stream of the trial site.

A single soil sample was taken from the prepared ground at Wombwell.

4.6.2 Phase 2 - treated material samples

41 x 100mm diameter cores were taken from the S/S material slabs between the 25th and 26th of March 1998. See Figure 3 for the location of each sample. The cores were taken with an electrical powered drilling rig using a diamond tipped cutter. Water was used as a drilling fluid (Plate 3). Each core was labelled and wrapped in bubble wrap immediately following recovery to avoid damage (Plate 4).

Water samples were taken from the surface run off sump and from the sump for the percolated leachate from the base of the main slab.

A sample of the calcareous like precipitate was taken from the corner of material D slab.

100kg of crushed material B was taken to test the material for suitability in road construction.

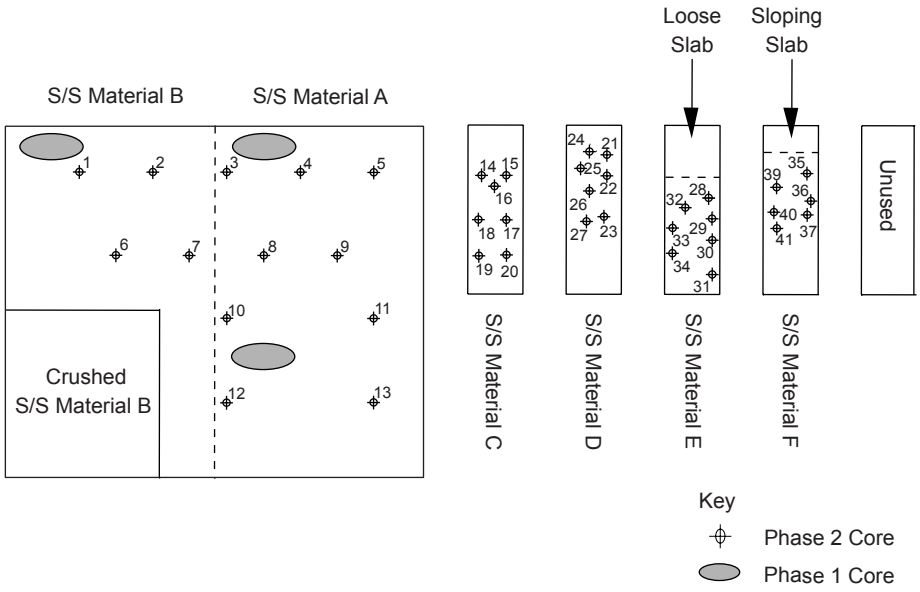


Figure 3 Location of core samples

5 Materials testing

The objective of Phase 1 was to demonstrate and assess the performance of a commercially available S/S technology. Several physical and chemical testing methods were chosen to meet this objective. The aim of Phase 2 was to assess variations in the physical and geochemical properties of the S/S materials with time and assess the suitability of S/S material as a construction material. In order to produce consistent data the same testing methods were used for Phase 2 as for Phase 1. The physical and chemical testing methods used are detailed in the following sections.

5.1 Physical testing methods

Unconfined compressive strength tests were used to determine the cube and core compressive strengths. All cubes and core strength values are reported in N/mm^2.

5.1.1 Cube strength

All the cubes of S/S material were tested during Phase 1. Cube compressive strength testing was conducted by South Yorkshire Laboratory, Sheffield. The testing was not conducted to BS standards.

5.1.2 Core unconfined compressive strength

The unconfined compressive strength of the cores taken during both Phase 1 and 2 were determined in accordance with BS1881: Part 120: 1983. The estimated in-situ cube strength was calculated in accordance with this standard. Compressive strength testing of Phase 1 samples was conducted by SGS United Kingdom Ltd, Sheffield. Phase 2 samples were tested at the Department of Civil Engineering, University of Portsmouth.

5.1.3 Permeability

Permeability testing of the six S/S materials was conducted during Phase 2. Permeability of the core samples was determined under constant head conditions in a triaxial cell. The tests were conducted in accordance with BS1377: Part 6: clause 6: 1990 by LTG Laboratories.

5.1.4 Porosity

The porosity of the six S/S materials was determined by caliper and saturation methods, using the International Society for Rock Mechanics suggested methods (1985).

5.1.5 Aggregate testing of crushed material B

Crushed material B was subjected to a number of tests required to establish whether a material will comply with criteria set out in the Specification for Highways Works.

The tests comprised:

- A particle size distribution test was conducted to BS 812: Part 103: 1985.
- Ten percent fines value test (TFV) was conducted to BS 812: Part 111: 1990. The TFV test was conducted on previously soaked material between 10-14mm in size. On examination of this washed material it was observed that many of the clasts in this size range were limestone. In order to find the true TFV of material B the sample was visually inspected and limestone clasts were removed.
- Determination of the plastic limit of material B passing a 425mm sieve was conducted to BS 1377: Part 2:1990.
- Determination of magnesium sulphate soundness value (MSSV) was carried out to BS 812: Part121: 1989. The MSSV test was conducted on a 20-28 mm size fraction sample of crushed Material B.
- Determination of water absorption was carried out to BS 812: Part 2: 1989.

5.2 Chemical testing methods

All chemical tests were conducted by Law Laboratories Ltd part of the Monitor Group. The chemical testing regime consisted of two parts:

1 Total chemical content of the samples.

2 Leachate testing.

5.2.1 Methodology of the leachate test

The leachate tests used was the NRA leachate test (Lewin et al, 1994).

NRA leachate test method.

The material was lightly crushed below 5mm.

A sieve analysis of the crushed sample was then conducted.

100g of the <5mm sample was placed in a flask with 1000ml of distilled, de-ionised water (10% w/v) and shaken end over end for 24 hours.

6 Results of physical testing

6.1 Unconfined compressive strength of the S/S materials

The compressive strength results for materials A, B, C, E and F are shown in Figures 4 to 8. These figures show the combined cube strength and estimated in-situ cube strength data, over time for each material.

The estimated in-situ cube strength data were derived using the appropriate equation given in BS 1881:Part 120:1983, from the core compressive strength data. The BS method states that in-situ cube strength data cannot be equated to standard cube strengths. However the lack of aged cube samples necessitated the use of the estimated in-situ cube data, to allow a plot of material compressive strength with time.

Unconfined compressive strength testing of the cube samples (Phase 1 of the trial) showed the early strength (<120 hours) of materials A, B, E, and F to be below the calibration range (i.e. less than about 2 N/mm^2) of the compression machine used. Materials A, B and E took between 7 and 10 days to develop strength within the calibrated range. Material F took 28 days to develop strength within the calibrated range. The low early strength of the S/S materials may have been measurable using test equipment capable of measuring strengths of 50 kN/m^2 upwards. There are no data available for the early strength of materials C and D.

Test cubes of material D were not produced during Phase 1 of the trial as the treated material did not set for a number of days. Table 2 shows the estimated in-situ cube strength values over time for material D core samples, obtained during Phases 1 and 2.

It can be seen from Figures 4 to 8 and Table 2 that all six materials, produced by the Portland cement based S/S process, cured and achieved measurable unconfined compressive strength. Materials B, C, E and F appear to be still gaining in compressive strength after 1216 days (40 months).

Table 2 Estimated in-situ cube strengths of material D core samples

Material D cores	*Age in days*	*Strength N/mm^2*
Phase 1	91	12.4
Phase 1	182	15.8
Phase 1	456	17.8
Phase 2	1216	9.7
Phase 2	1216	12.9
Phase 2	1216	16.8

6.2 Long-term strength

Table 3 shows the long term compressive strengths developed by the six S/S materials. Single core samples of materials B and D achieved the highest maximum strength (16.8N/mm^2) at 1216 days for all six materials tested. The weakest material at 1216 days was F with a maximum strength of 5.6 N/mm^2. The mean compressive strength of each S/S material at 1216 days is shown in Table 3. The mean compressive strength was calculated from at least 3 core samples.

Table 3 Estimated in-situ cube strengths of the six materials at 1216 days

	Maximum estimated in-situ cube strength N/mm^2	*Minimum estimated in-situ cube strength N/mm^2*	*Mean estimated in-situ cube strength N/mm^2*	*Standard deviation ± N/mm^2*	*Standard deviation ± % mean*
Material A	10.6	6.6	9.1	1.9	20.9
Material B	16.8	8.6	12.7	4.1	32.3
Material C	15.9	9.3	11.6	3.7	31.6
Material D	16.8	9.7	13.1	3.5	26.7
Material E	14.1	3.8	9.4	5.2	55.3
Material F	5.6	2.6	4.0	1.5	37.5

All six S/S materials tested show a range of compressive strength values for a given time. The range of long term compressive strength for each S/S material at 1216 days was expressed using standard deviation. The use of standard deviation gives an indication of the range of compressive strength that 68% of samples from a S/S material may be expected to reach. The standard deviation for each S/S material is shown as a value in N/mm^2 and as a percentage of the mean in Table 3. It can be seen that the three materials D, B and C with the highest mean compressive strength, have lower standard deviation percentages than two weaker materials, E and F. However material A had low strength and the lowest standard deviation percentage.

6.2.1 Maximum long term strength development

Single core samples of materials A and D reached highest compressive strengths of 11.8 N/mm^2 at 91 days and 17.8N/mm^2 at 456 days respectively, for each material. The 17.8N/mm^2 at 456 days achieved by material D was the highest compressive strength of all the materials tested. Although none of the nine material A samples tested after 91 days exceeded 12 N/mm^2, it was not considered possible to determine whether materials A and D had reached maximum compressive strength before 1216 days, due to the limited compressive strength data for each S/S material.

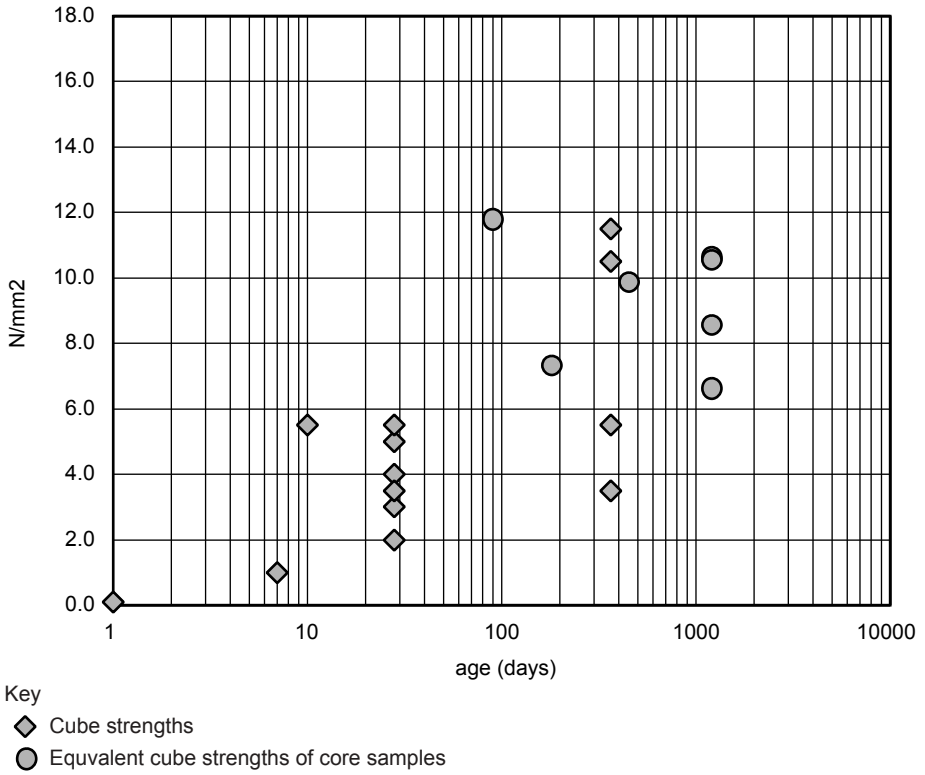


Figure 4 Material A compressive strength with time

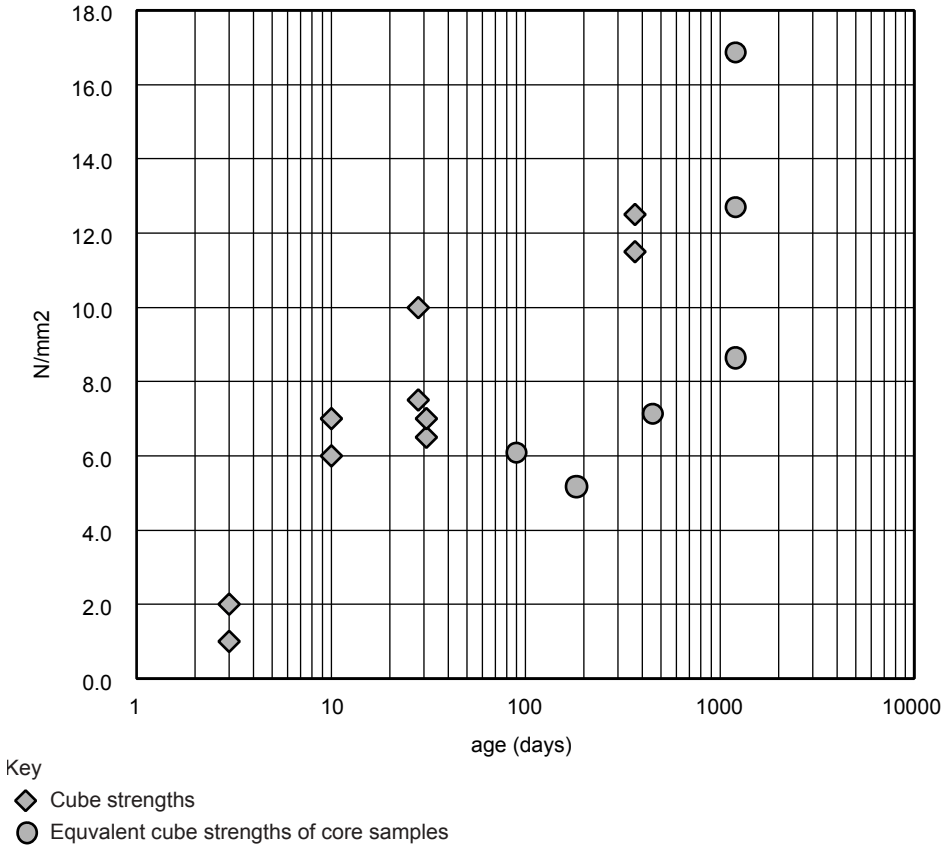


Figure 5 Material B compressive strength with time

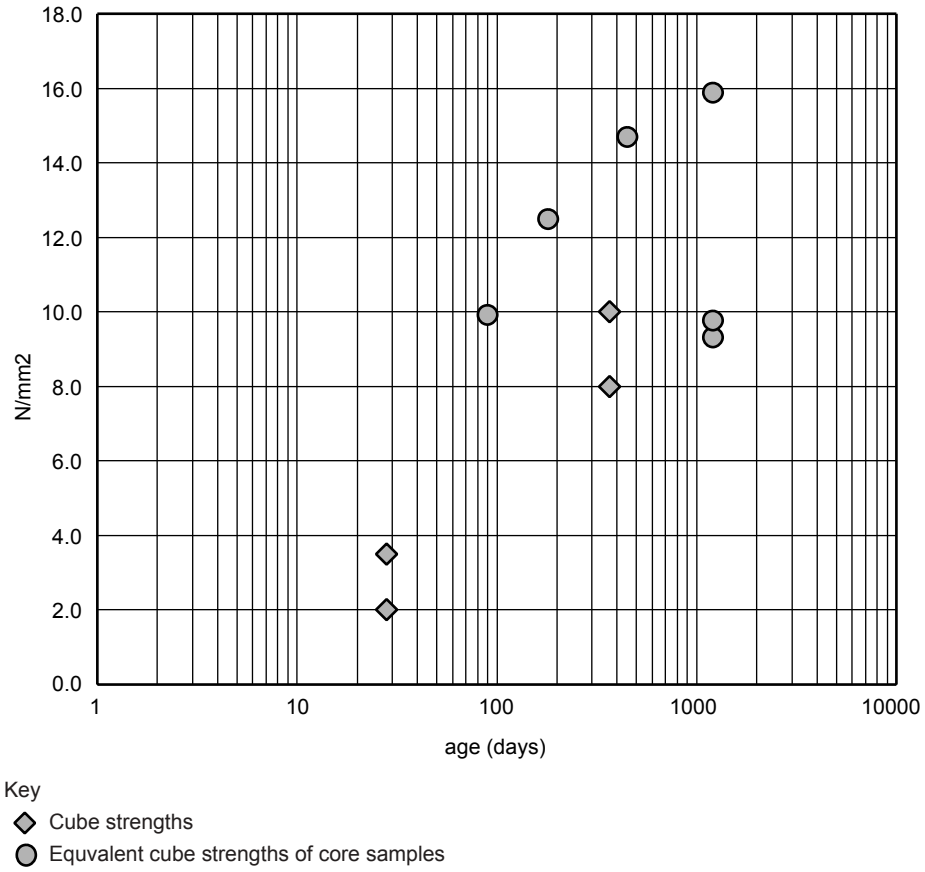


Figure 6 Material C compressive strength with time

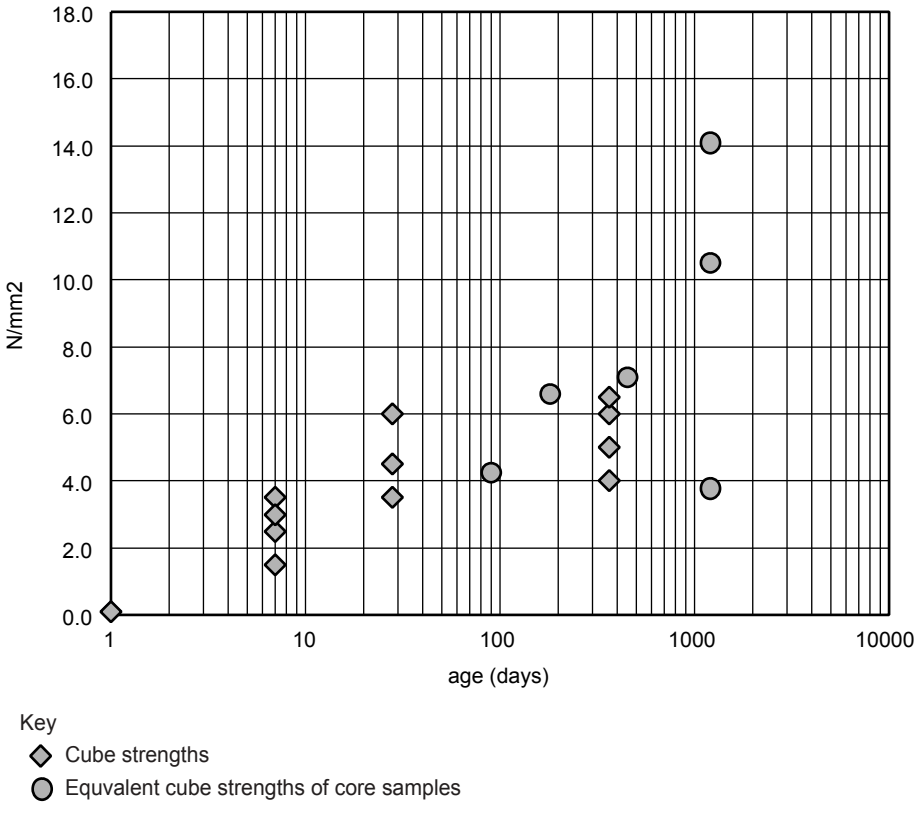


Figure 7 Material E compressive strength with time

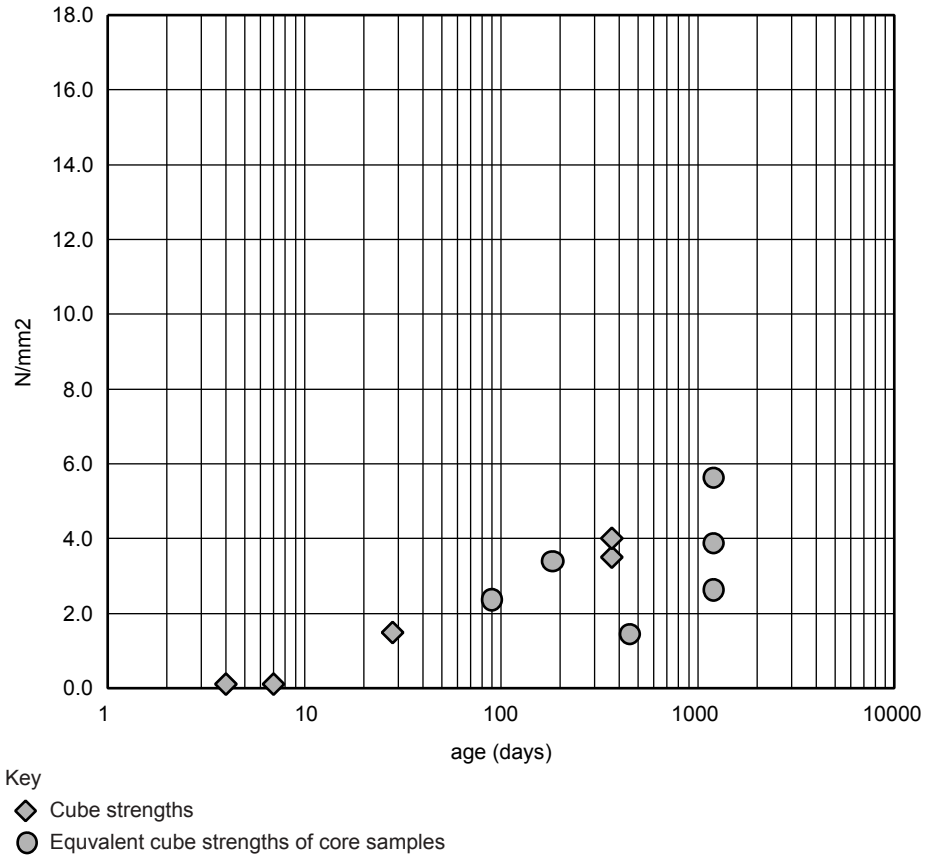


Figure 8 Material F compressive strength with time

6.3 Porosity/permeability tests

The results of porosity and permeability testing on the six S/S materials are shown in Table 4. Material E had the highest porosity but also the lowest permeability. This indicates that a S/S material may be porous but if the pores are occluded or unconnected, then permeability can be low. Material C was the most permeable and had the second highest porosity.

Table 4 Porosity/coefficient of permeability of materials A-F

Material	*Porosity (%)*	*Coefficient of Permeability (m/s)*
A	23.4	1.7×10^{-9}
B	24.7	1.4×10^{-9}
C	31.6	2.3×10^{-7}
D	22.9	4.0×10^{-8}
E	33.7	8.1×10^{-10}
F	25.9	4.0×10^{-8}

Porosity of concrete is known to be dependent on the w/c ratio of the design mix and the w/c ratio of all six S/S materials was equal. The porosity of the S/S materials may have been controlled by the sorption of water by the ash or minerals present within the untreated contaminated material. The high porosity of material C may be attributed to the greater amount of free water available for reaction with the Portland cement. The glassy spheres of PFA do not absorb water, the water is retained as a film around the sphere and any water not used in the cement hydration is free to form the pore water phase (Dhir et al, 1988) resulting in higher porosity.

6.4 Aggregate test results

Results of the tests conducted to assess the suitability of a crushed material B sample as an unbound material in road pavements are given below. The particle size distribution (PSD) of the crushed sample is shown in Figure 9, with the envelope of Type 1 sub-base superimposed for comparison.

The crushed material B sample tested in accordance with BS 812: Part 111: 1990 as a soaked aggregate had a ten percent fines value of 65kN.

The crushed material B passing the 425mm sieve was tested in accordance with BS 1377: Part 2 1990 and found to be non plastic (as defined by BS 1377:Part 1:1990). The plastic limit of the material could not be determined by rolling.

The crushed material B with a 20-28mm size fraction sample was tested in accordance with BS 812: Part 121: 1989. The material had a magnesium sulphate soundness value (MSSV) of 95.

The crushed material B with a 20-28mm size fraction sample was tested in accordance with BS 812: Part 2: 1985. The material had a water absorption value of 10 %.

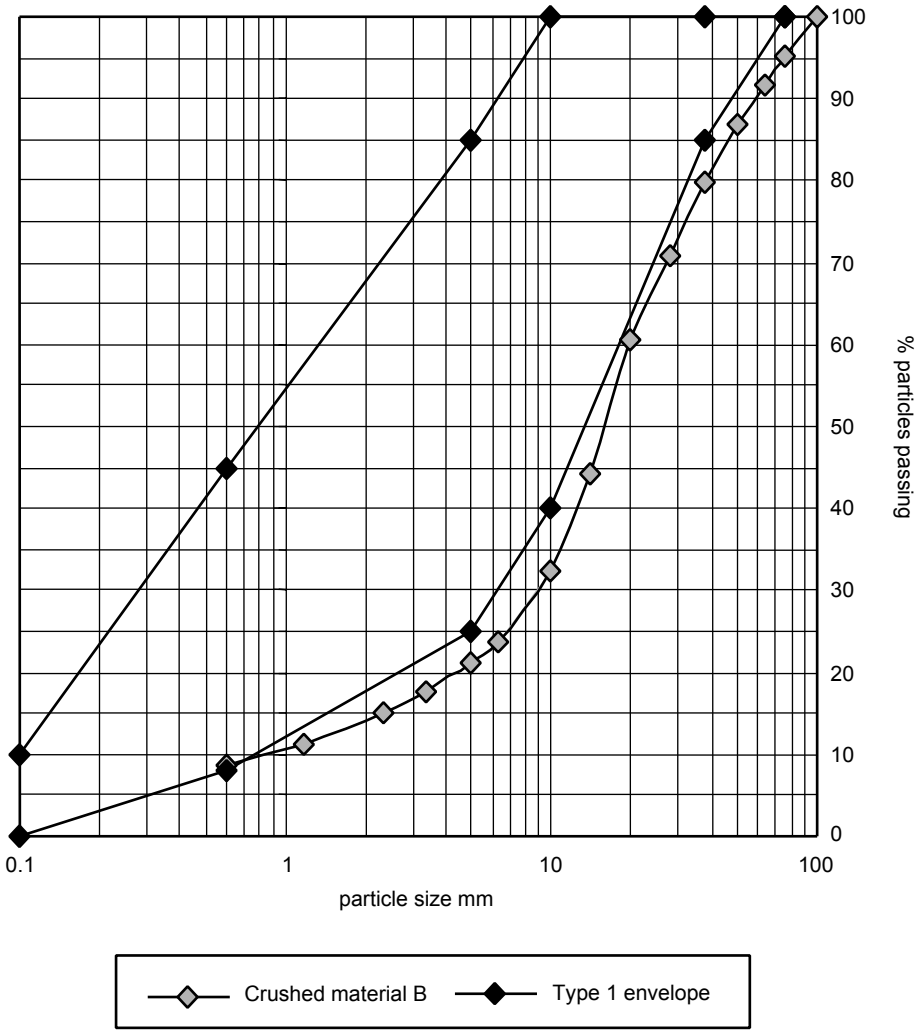


Figure 9 Particle size distribution of crushed material B and sub-base Type 1 material

7 The reuse of S/S material as a secondary aggregate in road construction

The potential for reuse of industrial by-products in road construction has been recognised for many years. The development of industry by-product reuse in road construction has been limited by the availability of comparatively cheap, primary aggregates. The requirement for a more sustainable use of primary aggregates and the use of environmental taxation to reduce the volume of construction waste, is now leading to the greater reuse of secondary aggregates. Although the UK road construction programme has declined recently the use of secondary aggregates in the future maintenance of roads may be considerable (Sherwood, 1995).

7.1 Specifications

The mandatory requirements for road construction are given in the Manual of Contract Documents for Highway Works, Volume1: Specification for Highways Works (1998) (MCHW 1). A review of this national specification concluded that it is not unduly restrictive and allows a wide range of materials to be used in highway construction (Dudgeon, 1999). The evaluation of secondary materials for use in highway construction should be carried out by comparing the properties of the secondary material with the properties of satisfactory materials already in use (Sherwood, 1995).

The tests specified in the MCHW 1 and detailed below were conducted on the crushed material B, to determine its physical suitability as a material in road construction.

Grading

The PSD of the crushed material B (Figure 9) met the grading requirements for:

a General Granular Fill, Class 1A material.

b Selected Granular Material, Class 6 materials 6B, 6E, 6F2, 6G.

Other Class 6 applications could be suitable if the PSD gradings were met by accurate crushing and appropriate screening of the S/S material.

Plasticity

The fine fraction <425 mm of material B was found to be non plastic.

The Ten percent fines value (TFV).

The TFV test gave a value of 65kN for material B.

Magnesium sulphate soundness value.

The material had a magnesium sulphate soundness value (MSSV) of 95.

7.2 Series 600 earthworks

The crushed B material data was compared to Series 600 Earthworks, MCHW 1. The specification for Class 1, 6, and 9 were reviewed. Applications within Classes 1 and 6 for which crushed concrete may be used were considered as potentially suitable applications for crushed material B. Fills 6A, 6C 6D and 6F3 are termed unsuitable for crushed concrete in the specifications. The crushed material B was not suitable as Fills 6I-J due to the high pH of the S/S material. The following classes within the MCHW 1 were found suitable for crushed material B:

General Granular Fill Class 1A material.

Selected Granular Fill Class 6 materials.

- 6B Starter layer
- 6E For stabilisation with cement to form capping
- 6F2 Capping
- 6G Gabion filling

On the addition of cement, class 6E material becomes class 9A.

7.3 Series 800 road pavements — unbound materials

A higher grade application for S/S material would be as a sub-base material. Two specifications are outlined for Granular Sub-base Materials Type 1 and Type 2. The preferred material for highway construction is Type 1. The crushed material B passed all tests required.

- The material was non plastic.
- The material had a TFV >50kN.
- The material had a MSSV > 75.

The crushed B material did not meet the exact requirements for the Type 1 grading envelope (Figure 9), but with controlled crushing the material would pass the grading test.

7.4 Series 1000 road pavements — concrete and cement bound materials

The crushed material B described above was produced from the aged main slab. Production of various fill materials by a crushing method requires a delay for curing of the S/S material before crushing can commence. An alternative application for a pre-set S/S material is as a Cement Bound Material (CBM). CBM can be used for roadbase or sub-base layers in highway construction.

Untreated contaminated materials were processed at the Wath Manvers remediation site and then transported to a second site where the S/S material slabs were cast, demonstrating the flexibility of the S/S process. Treated S/S materials can be handled by plant, transported and laid, prior to setting of the material, with no significant detrimental effects to the resulting S/S material.

The specifications for four CBMs are given in Series 1000. The materials permitted in a CBM are specified as having a coefficient of uniformity greater than 5 and as having low plastic (< 20%) and liquid limits (<45%). A ten percent fines value of greater than 50 is specified for materials to be included in CBM 2. Suitable crushed concrete may be used as an aggregate in all classes of CBM. Cured cubes formed from CBM 1 and 2 should be tested in accordance with BS 1924: Part 2: 1990 (including a seven day immersion followed by strength test). Either in-situ or ex-situ processes may be used to mix selected aggregate / soil with cement binder to form a CBM material.

7.4.1 Suitability of pre-set S/S material as a cement bound material

The above requirements for a material to be used in a CBM indicate that a physically suitable, untreated contaminated soil / industrial by-product, may theoretically be used in a CBM. However a limiting factor for the use of a pre set S/S material as a CBM material appears to be the early 7 day compressive strength required. The lowest acceptable 7 day strength is 4.5 N/mm^2 for CBM 1. The delayed early compressive strength seen in the six S/S materials tested would seem to prevent the use of a pre-set S/S material as a CBM. The meeting of the CBM 1 early strength standard may be a goal for future research on mix design.

7.4.2 Suitability of an aged S/S material in a cement bound material

The tests conducted on the crushed material B, show that an aged S/S material is suitable for use as an aggregate in a CBM. 40mm particles are the largest size of aggregate permissible in a CBM. The large size of S/S material particles allowed in a CBM and the addition of further cement in production of a CBM would further enhance the stabilisation / solidification process.

7.5 Summary

The crushed S/S material B has been shown to be suitable for:

- Earthwork Class 1 and 6 applications.
- Sub-base Type 1 material.
- As an aggregate in a cement bound material.

8 Discussion of the physical characteristics of the S/S materials

8.1 Strength requirements for a S/S material

S/S technology has been applied to a small number of contaminated sites in the UK. Two documented UK sites remediated using S/S technology are: Ardeer landfill,

Scotland (Barker and Esnault, 1996) and a former chemical works at West Drayton, England (Al-Tabba and Evans, 1998, Al-Tabba et al, 1998). Both sites were remediated using in-situ (auger mixing) S/S technology and differing mix designs. The Ardeer landfill site S/S treatment used a cement, lime and PFA mix design. The West Drayton S/S treatment used a cement / modified organoclay grout.

Although the two applications above used in-situ S/S processes and different mix designs to this trial, they provide a benchmark for compressive strength requirements of S/S materials. The strength requirement of the Ardeer landfill S/S material was > 0.2 N/mm^2 at 28 days, however the actual strengths achieved appeared to be up to 2.0 N/mm^2 (Barker and Esnault, 1996). The West Drayton S/S material was designed to meet a standard of 0.35 N/mm^2 at 28 days (Al-Tabba et al, 1998). The strengths achieved were again higher, between 1.0 and 1.5 N/mm^2. UK guideline strength requirements for solidified S/S materials, set by local waste regulatory authorities (WRAs) now superseded by the Environment Agency, are reported as a typical 28 day strength of 0.7 N/mm^2, but the criterion may be as low as 0.35 N/mm^2 (Hills and Pollard, 1997).

No strength requirements were specified for the six materials laid at Wombwell. However the 28 day strengths of all six materials exceed the 28 day strength requirements set by the former WRAs and also exceeded the final strengths of the Ardeer and West Drayton S/S materials. The compressive strength of all six materials continued to increase up to 90 days. Four of the six materials (B, C, E and F) showed continuing strength development up to1216 days.

The regulatory strength requirements set for S/S materials are very low compared to the strengths developed by the Ardeer and West Drayton sites and the six S/S materials tested. Most economic and environmental benefit is gained if a waste is treated with the minimum quantity of cement / additives required to achieve the desired degree of contaminant containment. A low strength requirement can be set because strength is not an indicator of effective contaminant containment (Means et al, 1995). Methods such as leachate testing are a better indicator of effective contaminant containment within a S/S material than strength. However, another physical performance factor that must be considered when evaluating a S/S material is its durability.

8.2 Durability

Although a low strength S/S material may be effective at containing contaminants in the short term, long term degradation of a S/S material will result in the release of contaminants. Both strength and durability (particularly under freezing conditions) of a S/S material, have been shown to improve with increasing amounts of cement (Evans and Al-Tabba, 1997; Al-Tabba et al, 1998).

Durability testing was not conducted on the six S/S materials. However visual inspection of the slab surfaces showed that materials D and E appeared to be more susceptible to weathering than materials C and F. This indicates that although strength is important, it is not the only factor controlling durability. Permeability, porosity, pore size and moisture content of a S/S material are major factors controlling durability of concrete (Eglinton, 1998).

8.3 Additional factors affecting S/S material strength

8.3.1 The effect of particle size distribution on the strength of a S/S material

The particle size distribution (PSD) of simulated clean made ground and natural sand / gravel has been shown to be a controlling factor on the 28 day strength of a S/S material (Evans and Al-Tabba, 1997). Made ground with its higher percentage of fine (<63mm) particles had a lower strength compared to a coarser grained sand / gravel. The presence of contaminants in both materials further reduced the strength of the S/S materials by 50%.

The PSDs of untreated materials A, B, D and E showed them to comprise of predominantly sand sized particles, and so the PSD effect on the strength of the four materials was considered to be minimal. The low strengths of material F are believed to result from the high clay content of the untreated material. The low shear strength of clay reduces the compressive strength of a S/S material. A high clay content also results in poor mixing of the cement and additives. The PSD effect on the strength of material C does not occur as a PFA / PC concrete has been shown to reach greater long term strength than a PC concrete (Dhir et al, 1998).

8.3.2 Identification of detrimental effects of contaminants on strength

If the ratio of contaminated material to cement of S/S materials is equal and the same mixing / construction processes used in their treatment, three major factors control the strength of an individual S/S material.

- Water / cement ratio.
- The particle size distribution of the untreated material.
- The type and concentration of contaminants within the untreated material.

As the w/c ratios of all six S/S materials tested were equal and the possible PSD effects on strength regarded as minimal for all but material F, the dominant factor affecting strength may be regarded as the type and concentration of the contaminants present.

8.4 Strength developed by material C

A variety of heavy metal contaminants are bound on to and within the glassy spheres which comprise PFA. Pozzolanic forms of PFA are commonly used as partial cement replacements and also increase the sulphate resistance of concrete. PFA is known to have a beneficial chemical effect on the hydration of Portland cement (PC) (Dhir et al, 1988).

The long term strength development of PFA concrete is also well known. Dhir et al (1998) report that up to 45 % of PC can be replaced with PFA without affecting the 28 day strength of a concrete. At normal w/c ratios the 28 day strength of a PFA concrete will be lower than a PC concrete. By 56 days the PFA concrete will be stronger than the PC concrete. Considerable strength development over 10 years is reported for some PFA concretes (BCA, 1995).

Detrimental effects of contaminants contained in PFA on a PFA / PC material have been shown to be minimal (Dhir et al, 1988). Therefore material C was predicted to achieve the highest compressive strength of the six materials treated. This hypothesis was tested by comparing the unconfined compressive strength of each S/S material with that of material C.

8.5 Comparison of material A, B, C and E compressive strengths

8.5.1 Less than 5 day strengths

The low early strengths (< 120 hours) of all the S/S materials for which there are data, indicates that the concentrations of organic and inorganic contaminants present within the materials delayed the initial PC hydration reactions.

8.5.2 28 day strengths

The 28 day strength data for material C was limited to two test cube unconfined compressive strengths of 2.0N/mm^2 and 3.5N/mm^2. The low 28 day strength of the material C cubes was attributed to a high carbon content of the untreated PFA. Materials A and E developed similar 28 day strength to that of material C (Figures 4, 6 and 7). Material B appeared to developed greater 28 day strength than material C (Figures 5 and 6). No data were available for material D.

8.6 Long term strength of the materials

8.6.1 Materials stronger than material C

The 91 day and mean long term (1216 day) strengths achieved by material C (Figure 5) were within the strength range of 7.0-12.0N/mm^2 predicted for a 10:1 PFA / PC material at 90 days. The mean long term strengths of materials B and D were slightly greater than those achieved by material C (Table 3).

The delayed setting of material D for several weeks and the reduced 28 day strength development of material C due to high carbon content, did not appear to greatly affect the long term strength of either material. The eventual (10 year) strength of material C may exceed the maximum strengths of materials B and D, due to continued pozzolanic reactions within material C.

8.6.2 Materials weaker than material C

Materials A, E and F showed lower compressive strength than material C at 91 and 1216 days. Materials E and F may still be gaining in strength but will fail to exceed the maximum long term strength of material C. As discussed in section 8.3.1, the low long term strength of material F was due to its high clay content.

The 28 day strength of material E was shown by comparison with material C to be relatively unaffected by its contaminant content. However the 91 day and long term strengths of material E were lower than those of material C. Therefore the long term strength of material E may have been controlled by the type and concentration of contaminants present within the material.

Material A appeared to have developed relatively consistent long term strength by 91 days. The physical characteristics and treatment of materials A and B were virtually identical. Material A showed a reduced long term strength when compared to material B which may have been caused by the slightly higher concentrations of organic and inorganic contaminants present in material A.

8.7 Summary

The 28 day unconfined compressive strengths achieved by all six S/S materials exceeded the former WRA requirements for S/S materials and were greater than documented site examples. PFA based material C was predicted to achieve the highest compressive strength. Materials B and D, contained significant concentrations of inorganic contamination, and developed slightly greater mean long term compressive strength than the control material C. Contamination appeared to control the long term strength of materials A and E. The strength of material F was considered to be controlled by the particle size distribution of the untreated material.

This trial has shown that contamination may cause time dependent effects on the early and long term compressive strength of S/S materials. However the effects vary greatly between S/S materials produced from similar contaminated materials. Treatment process factors such as particle size distribution of the untreated material may have a greater effect on compressive strength than low levels of contamination.

9 Chemical results and discussion

9.1 Total chemical contents of the six S/S materials

The mean total chemical content (TCC) of metals present in each S/S material are shown in Table 5. The mean TCC concentrations of Al, As, Cd, Cr, Cu, Hg, Ni, Pb and Zn were calculated from Phase 1 and Phase 2 data. B, Ca, Fe, K, Mg, Mn, Na and Se mean TCC concentrations were calculated from Phase 2 data. Table 6 shows the mean TCC of organic compounds present in each S/S material. Total organic carbon (TOC); benzene, toluene, ethyl benzene and xylene (BTEX); and polychlorinated biphenyls (PCBs) TCC were calculated from Phase 2 data. The mean TCC of all other organic compounds including total polycyclic aromatic hydrocarbons (TPAHs) were calculated using Phase 1 and Phase 2 data. Table 7 shows the mean TCC of anions present in each S/S material. The mean TCC of fluoride was calculated from Phase 2 data. All other anion mean TCC concentrations were calculated from Phase 1 and Phase 2 data.

9.2 Comparison of TCC data with ICRCL guidelines

The mean TCC data from the six materials were compared with the Interdepartmental Committee on the Redevelopment of Contaminated Land (ICRCL) guideline values (DoE, 1987). The ICRCL values are the current UK guidelines for the remediation of contaminated land. The ICRCL guidelines are divided into several categories according to the end use of the land and the form of the contaminants. As a S/S material would not be reused in a

Table 5 Total chemical content of metals present in the six S/S materials

Mean total chemical content mg/kg	*As*	*Al*	*B*	*Ca*	*Cd*	*Cr*	*Cu*	*Fe*	*Hg*	*K*	*Mg*	*Mn*	*Na*	*Ni*	*Pb*	*Se*	*Zn*
Material A*	23	23300	766	157750	286	237	2682	21100	1.4	2025	36850	1860	330	2644	347	27	530
Material B†	18	12223	119	140400	96	91	1346	15832	0.6	1294	37880	1011	280	1041	1084	13	574
Material C	9	9632	114	126000	9	30	173	11053	<0.1	2387	28900	390	696	144	91	2	150
Material D	3454	24250	2173	117000	66	74	4643	127333	<0.1	3393	8680	4680	116333	470	13212	<1	42000
Material E	791	13950	1243	121000	36	34	1622	70000	<0.1	1980	31100	5930	33650	208	7650	<1	14400
Material F	99	10000	168	145000	4	38	525	35650	0.2	1295	38550	954	728	193	988	2	3235
ICRCL values parks, buildings	40				15	1000	130		20					70	2000	6	300

** excluding 90 day data.*
†including crushed material B data.

Table 6 Total chemical content of organic compounds present in the six S/S materials

Mean total chemical content mg/kg	*Total organic carbon*	*Toluene extractable matter*	*Hydro-carbons by infra red*	*PCBs*	*TPAH*	*BTEX*	*Hydro-carbons as mineral oil*	*Total phenols*
Material A*	299	2860	1585	0.13	52	<1	<20	4.26
Material B†	152	6329	2765	<0.01	37	<1	<20	3.41
Material C	43	463	283	N/D	N/D	<1	<20	0.52
Material D	13	2292	1145	N/D	6	<1	<20	1.48
Material E	8	1023	633	N/D	4.5	<1	<20	0.65
Material F	290	1990	735	N/D	44	<1	<20	5.32
ICRCL values parks, buildings					1000			5

** excluding 90 day data.*
† including crushed material B data.
N/D - Not Detected

Table 7 pH, moisture content and total chemical content of anions present in the six S/S materials

Mean total chemical content mg/kg	*Moisture content at 105°C w/w %*	*pH*	SO_4	*Water soluble sulphate (g/l)*	S_2	*Cl*	*F*	*Total CN*
Material A*	4.3	11.0	7072	0.06	23	40	2.10	0.56
Material B†	4.5	11.2	7211	0.05	26	26	0.88	0.83
Material C	5.2	11.6	6616	0.01	<20	29	0.40	<0.50
Material D	3.0	11.8	6124	0.05	26	17	0.70	<0.50
Material E	7.6	11.5	9900	2.63	24	39	0.84	0.53
Material F	5.3	11.5	18785	0.10	49	31	0.70	52.8
ICRCL values parks, buildings		<5	2000					250

** excluding 90 day data.*
† including sub base material.

domestic garden, the TCC values are compared to the ICRCL parks / buildings threshold values. This category is appropriate for materials used in road construction. However a simple comparison of contaminant concentrations present within a material with guideline values does not constitute a risk assessment of a material. Other factors such as leachability are of greater importance.

All the S/S materials except material C exceeded the ICRCL threshold value for at least three metal contaminants. Material C only exceeded the ICRCL threshold value for phyto-toxic metals. Of the six materials, material D contained the highest concentration of metal contaminants Al, As, Cu, K, Na, Pb and Zn. Material A contained the highest concentrations of Cd, Cr, and Ni. Material E contained a similar suite of metals to material D but the metals were present in lower concentrations. All six materials exceeded the ICRCL threshold value for sulphate, see Table 7. The materials also had a high pH value as expected for cement based S/S materials.

Only material F had a mean TCC concentration of an organic compound (total phenols) that exceeded the ICRCL threshold value, see Table 6. Of the six S/S materials, material A had the highest TOC content, however the TOC concentration was relatively low. Material A also contained the highest concentration of total polycyclic aromatic hydrocarbons (TPAHs) and was the only material to contain a measurable concentration of PCBs. Material B contained the highest concentration of toluene extractable matter (TEM) and concentrations of hydrocarbons detected by infra-red spectrometry (HC by IR). Material C contained the lowest concentration of organic contaminants.

In summary five S/S materials had mean TCC concentrations of several metal contaminants, potentially harmful to health, that exceeded the ICRCL threshold values for open spaces and buildings. Material D contained the highest concentration of As, Pb and phyto-toxic metal contaminants. Material A contained the highest concentration of Cd, Cr and Hg and also contained the highest concentrations of organic contaminants. The concentrations of organic contaminants present in the six S/S materials were low compared to ICRCL threshold values.

9.3 Total chemical content of untreated materials A-F

The untreated materials TCC data were produced during Phase 1 testing. The TCC of the untreated materials showed higher contaminant concentrations than those found in the S/S materials. The reduced contaminant concentrations found in the S/S materials were due to dilution by addition of cement and aggregate. Based on Phase 1 TCC data for untreated materials, Al was the only metal observed to increase in concentration in the S/S material. This increase was due to the addition of cement which contains a high concentration of Al.

9.4 XRF analysis of material D carbonate precipitate

On inspection of the trial slabs, a white, fine grained carbonate type material containing small grains of material D was found to have precipitated on the Visqueen membrane at the edge of material D slab (Plate 5). Major element analysis (Table 8) and trace element analysis (Table 9) of a precipitate from slab D are provided. The results are expressed as oxide wt %. The material had a loss on ignition of 32.7 % at 1000°C. The total wt % reported was 90% because the material contained borate

Table 8 Major element concentrations of carbonate precipitate from slab D

Major element	SiO_2	TiO_2	Al_2O	Fe_2O_3	*MnO*	*MgO*	*CaO*	Na_2O	K_2O	P_2O_5
As wt %	14.91	0.19	4.25	11.39	0.32	1.06	56.26	1.17	0.31	0.12

Table 9 Selected trace element concentrations from slab D carbonate precipitate

Trace element	*Sr*	*Zr*	*Nb*	*Pb*	*Zn*	*Ni*	*Cr*	*V*	*Ba*	*MnO*	*TiO*	*As*
mg/kg	926	18	6	6583	10280	257	209	54	792	2176	1475	792

which is not measurable by XRF. The high weight % of CaO indicates that this material is carbonate based. Comparison of the precipitate and material D total chemical content data showed an increased concentration of Cr in the precipitate. This finding indicates that Cr may have been mobile during the delayed setting of material D.

9.5 Carbonation

Two trial slabs, materials C and F, showed evidence of conventional carbonation reactions. Phenolphthalein testing of a material C core hole, immediately on removal of the core showed that a carbonation rim had formed to a depth of 10mm (Plate 6). Carbonation reactions occur between calcium bearing phases and carbon dioxide resulting in the formation of calcium carbonate and water (Walton et al 1997).

Several beneficial effects due to carbonation of S/S materials have been identified. Hydration of cement used as a binding agent in the S/S process may be strongly impaired by organic and inorganic contaminants. However the carbonation of S/S materials with retarded cement hydration has been shown to increase setting and strength development (Hills and Pollard, 1997). The immobilisation of inorganic contaminants within S/S materials has also been shown to be improved by carbonate alteration of hydrated silicates (Lange et al, 1996).

Carbonation reactions cause changes to both the physical and chemical characteristics of a S/S material. The formation of calcite results in reduced porosity as calcite fills more pore space, thus lowering the permeability of carbonate altered material (Walton et al 1997). The contrast between the hard, sound surface and the poorly cured clayey material found at the base of certain cores from the material F trial slab, may be attributed to carbonation of the upper slab material.

9.6 Leachate testing

Clause 78A of the Environmental Protection Act 1990 defines contaminated land as land that has the potential to cause significant harm to human health or the environment or pollution of controlled waters. For a site to pose significant harm or threaten water resource pollution, the source - pathway - receptor model must be applied. If no pathway is available for contaminants to reach a receptor then a site with contaminant concentrations above current guideline levels cannot be considered as harmful (Nancarrow, 1998). Using S/S technology to remediate a contaminated material does not destroy contaminants, it stops or restricts pathway processes transporting contaminants to a receptor. Leaching of contaminants to surface and ground water is a key pathway from a source to a receptor. The effectiveness of S/S technology to remediate a contaminated site must be assessed by leachate testing (Means et al, 1995).

9.7 Processes controlling leaching

Leaching can be defined as the mobilisation and transportation of components from a material. Two processes, diffusion and dissolution control the mobilisation of S/S material components. Transportation of mobilised components through a S/S material may occur by diffusion or advective flow. The transportation mechanism is determined by the permeability of a S/S material. Transportation of mobilised components occurs by diffusion when a S/S material has a coefficient of permeability lower than 1×10^{-9}m/s (Means et al, 1995).

Permeability testing of the six S/S materials showed material E to have permeability equal to or lower than 1×10^{-9}m/s. Materials A, B, C, D and F had a coefficient of permeability higher than 1×10^{-9}m/s. Theoretically, advective flow would control the leaching rate of components from the latter group of materials.

9.8 Evaluation of the NRA leachate test

The NRA leachate test is a dissolution based method, requiring the crushing of a sample to <5mm, thereby increasing the surface area of a material in contact with the leachant. This method ensures that leachable contaminants are rapidly extracted from a contaminated material. By using a dissolution based leaching method, solidification (a fundamental factor in the effectiveness of a S/S material) is disregarded. Therefore the data gained from the NRA method should be viewed as providing a conservative estimate of the effectiveness of a S/S treatment.

9.9 Evaluation of leachate data

The leachate data produced from testing a S/S material may be evaluated against environmental quality standards (EQS). EQS values are derived according to the quality and use of a water resource that a leachate may enter. Usually EQS values are based on drinking water or groundwater standards. The comparison of leachate contaminant concentrations with EQS values may indicate the effectiveness of the S/S treatment, but EQS values should not be used as the pass / fail criteria for a S/S material (Baldwin et al, 1997). Leachate from a S/S material entering the soil will be affected by a number of processes such as: dilution with soil pore water; precipitation / solution reactions; and natural attenuation of the contaminants. The concentration of contaminant that eventually reaches a water resource may be considerably less than the original leachate concentration. Site specific assessment of the potential for a S/S material leachate to pollute a water resource should be made using a tiered methodology developed for the Environment Agency (1996c).

9.9.1 NRA leachate testing of five untreated materials

The leachate data for untreated materials (UTM) A and B subjected to the NRA leachate test, are reported in Jardine and Johnson (2000). No leachate data are available for UTM D. UTM leachate data for specific contaminants are included in many of the following figures. The inclusion of these data enabled a comparison of untreated and treated S/S material leachate concentrations. The effects of S/S treatment on the increased or decreased leachability of a contaminant could then be evaluated.

Comparison of the UTM NRA leachate results showed that all materials tested exceeded the EQS value for Al. UTMs A, B, and F exceeded the EQS value for Cd, a list 1

metal banned from discharge to water resources. UTM F exceeded the EQS values for Cu, Ni, Zn and sulphate. UTM E exceeded the EQS value for Pb.

9.9.2 NRA leachate testing of the six materials

Figures 10 to 24 show the concentrations of certain inorganic contaminants leached from the six S/S materials subjected to the NRA leachate test method. Included in the figures are the detection limit (DL) of the analytical equipment used, the leachate concentration from the UTMs and an EQS reference value (Environment Agency, 1996b).

9.9.3 Mean leachate pH of the six S/S materials.

The mean leachate pH of the six S/S materials ranged from 11.5 for material E to 12 for material B. The high pH environment of a S/S material will tend to suppress the solubility of metal cations, however amphoteric elements such as Al, Cr, and Pb are soluble at low and high pHs (Gill, 1992). In strongly basic solutions such as a S/S material leachate these amphoteric elements may form soluble anionic complexes (Glasser, 1994). The strongly basic pH of the leachates produced by the six S/S materials indicates that amphoteric metals present in the leachates may be in anionic form rather than as colloidal particles.

The leachate pH of five materials A, B, C, D, and F remained relatively constant with time, with the pH of most leachate samples fluctuating by ± 0.5. Material E appeared to show a decline in leachate pH over 1216 days from 12 to 11. Leachate concentrations of chloride and sulphate from the six S/S materials remained constant throughout the trial. Material F showed a considerable decrease in the concentration of sulphate leached from the treated material compared to the concentration of sulphate leached from the UTM.

9.10 Inorganic contaminants effectively contained by the six S/S materials

The NRA leachate test data from the six S/S materials were compared with EQS values derived from drinking water standards (Environment Agency, 1996b). Comparison showed that metals Cd, Cu, Hg, Ni, and Zn, were effectively immobilised below EQS values for all six materials throughout the trial period. The leachate concentrations of the five metals were below EQS values even though high average concentrations (42000 mg/kg of Zn in material D and 2600 mg/kg of Ni in material A) were present in certain S/S materials. Cu was leached at concentrations above the UTM values from five of the six materials, however the leachate concentrations of Cu were always below the EQS value.

The effective stabilisation of Cd, Hg, Ni, and Zn was also seen by comparison of the treated and untreated material leachate data. Cd was leached by four of the five UTMs for which there were data, but was not seen to leach from any of the six S/S materials. High leachate concentrations of Al, Cu, Pb, Ni, and Zn from UTM F were considered to be due to the low pH (3.5) of the leachate. All five metals leached from the UTM F were stabilised effectively in material F.

9.11 Leached inorganic contaminants

9.11.1 Material A

It can be seen from Figures 10 to 12, that Al, Pb, and Cr were leached from material A. The concentrations of the metal contaminants leached from the material changed with time. The concentration of Al exceeded the UTM value and the EQS value (0.2 mg/l) throughout the trial period. The leaching of Pb from material A showed an initially similar pattern of leaching behaviour. However by 456 days, leachate concentrations of Pb reduced, remaining below the EQS value for the duration of the trial period. Leachate concentrations of Cr showed a variable range over the trial period, fluctuating from below the detection limit of the analytical equipment to exceeding the 0.05mg/l EQS value.

9.11.2 Material B

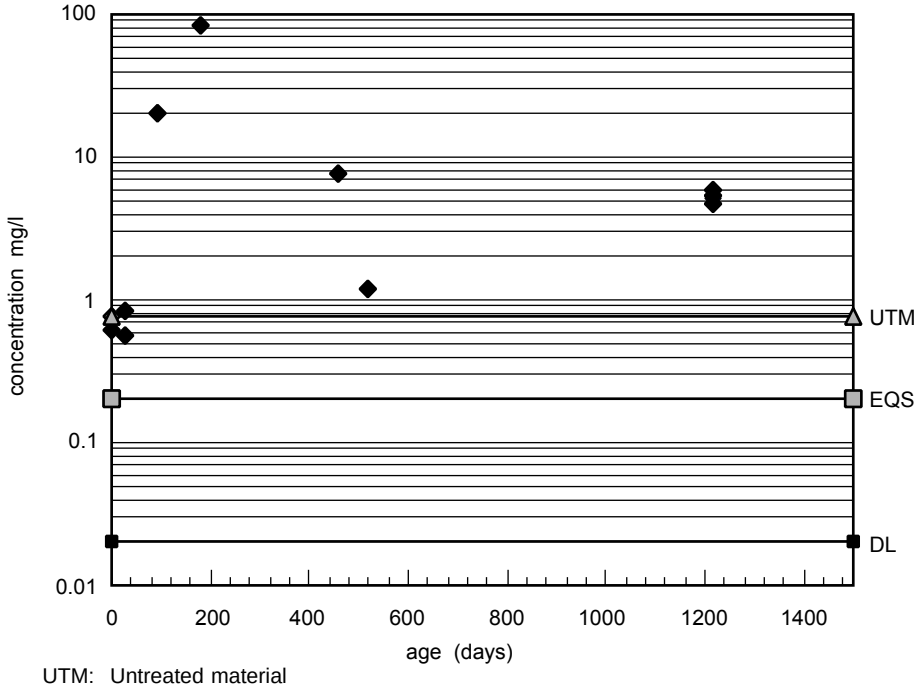
Material B leachate contained significant concentrations of Al, Pb, and Cr (Figures 13 to 15), the same metal contaminants detected in material A leachate. Material B also showed similar metal leaching patterns with time as material A. The leachate concentrations of Al exceeded the EQS and UTM values throughout the trial period. The material B leachate concentrations of Pb varied with time and were consistently above the UTM value throughout the trial period. Material B leachate concentrations of Cr were variable exceeding the EQS and UTM values at times.

9.11.3 Material C

The metals Al and Cr were leached in significant quantities from material C. The UTM leachate concentrations of Al significantly exceeded the 0.2 mg/l EQS value (Figure 16). After an initial peak at 91 days, the material C leachate concentration of Al remained above the EQS value at a consistent concentration of between 2 and 5 mg/l for the remainder of the trial period. Figure 17 shows that the concentration of Cr leached from material C increased with time, indicating a possible relation between the age of the sample and the concentration of Cr leached. At 1216 days the concentration of Cr was almost twice the EQS value.

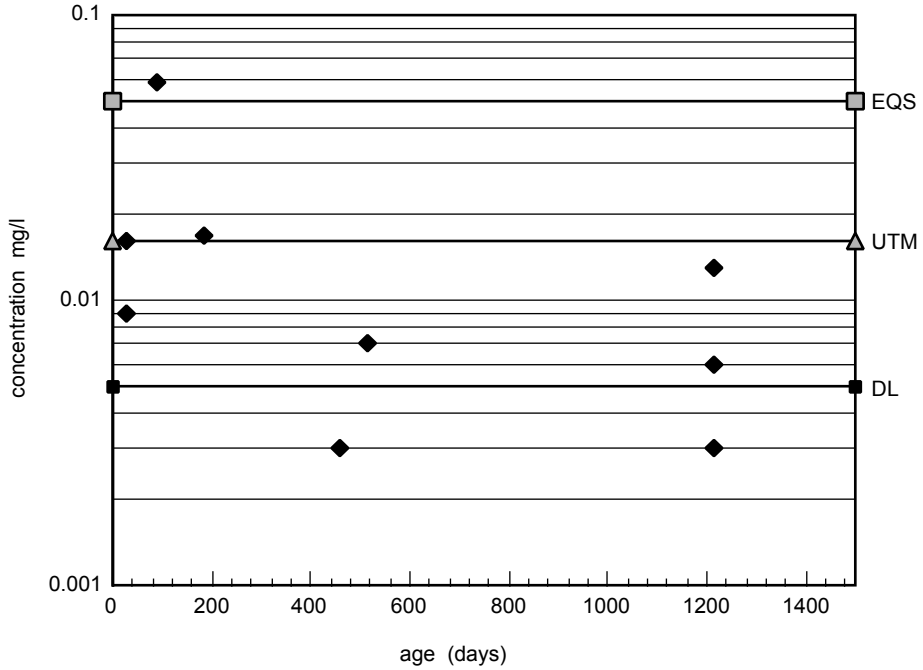
9.11.4 Material D

The metals Al and Pb (Figure 18), leached in significant concentrations from material D. The early leaching behaviour of material D could not be established as no test cubes were made and leaching tests were not conducted on the UTM. Material D leachate concentrations of Al peaked at 91 days (20mg/l) and then decreased with time. At 1216 days Al concentrations were below the 0.2 mg/l EQS value and detection limit. Leachate concentrations of Pb increased with time to exceed the EQS value at 182 days. The leachate concentration of Pb continued to remain high and a material D leachate sample contained the highest Pb concentration (36 mg/l at 1216 days) of all six S/S materials. Cr was not leached at any time from material D by the NRA leachate test method.



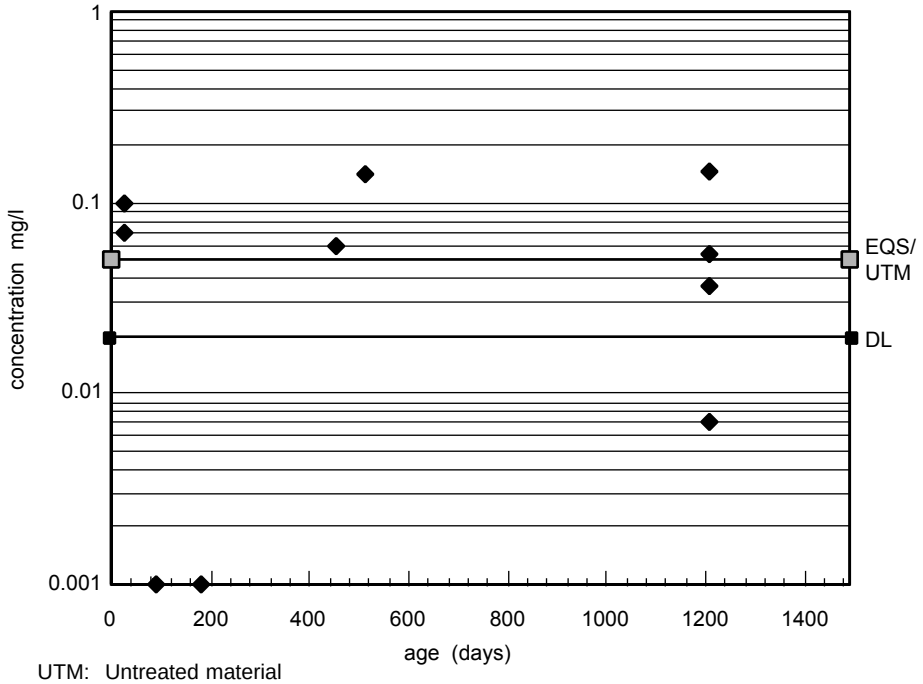
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 10 A1 concentrations leached with time from material A



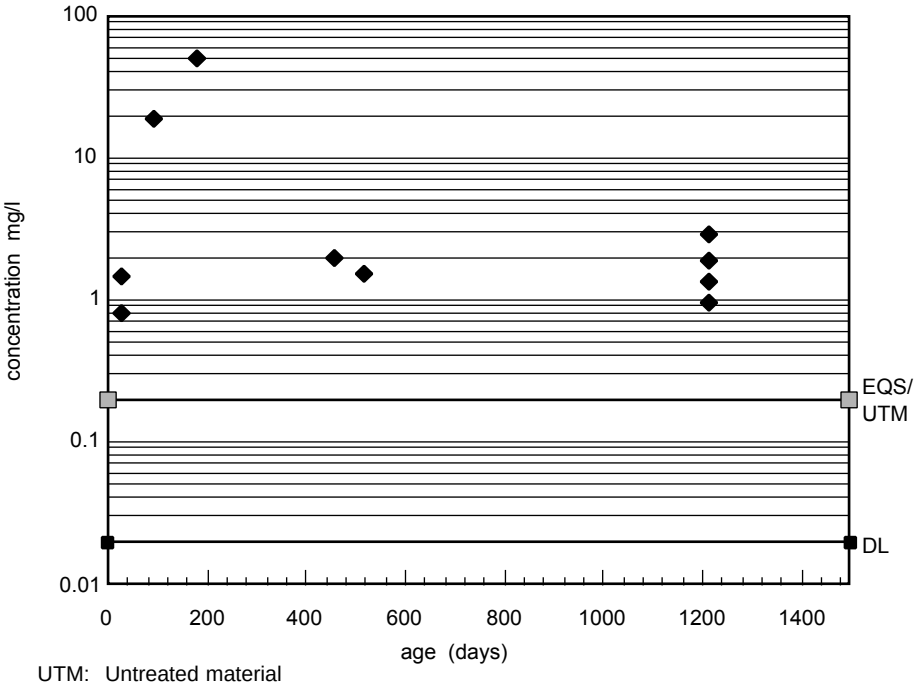
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 11 Pb concentrations leached with time from material A



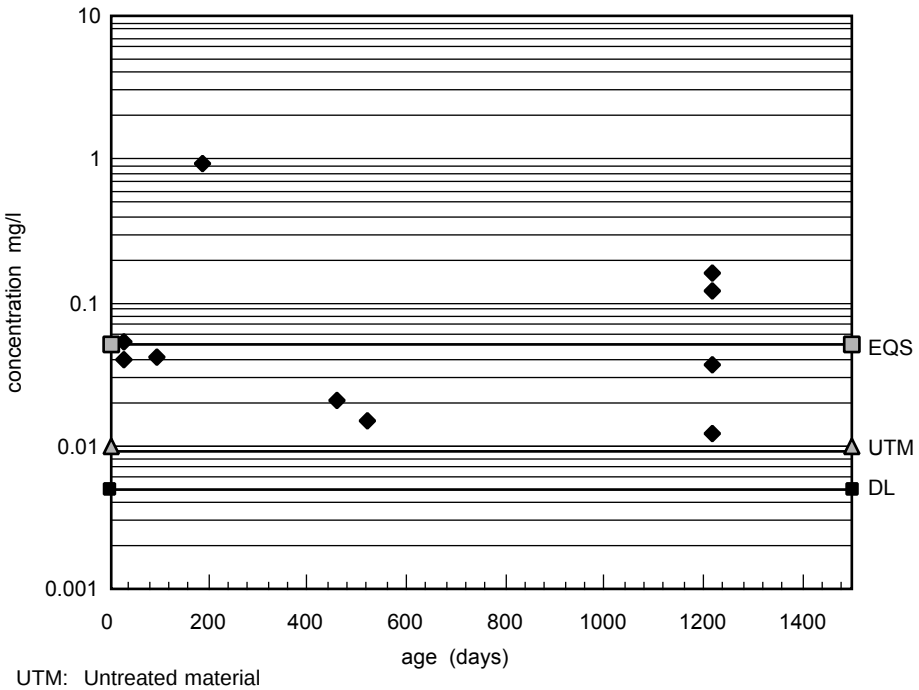
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 12 Cr concentrations leached with time from material A



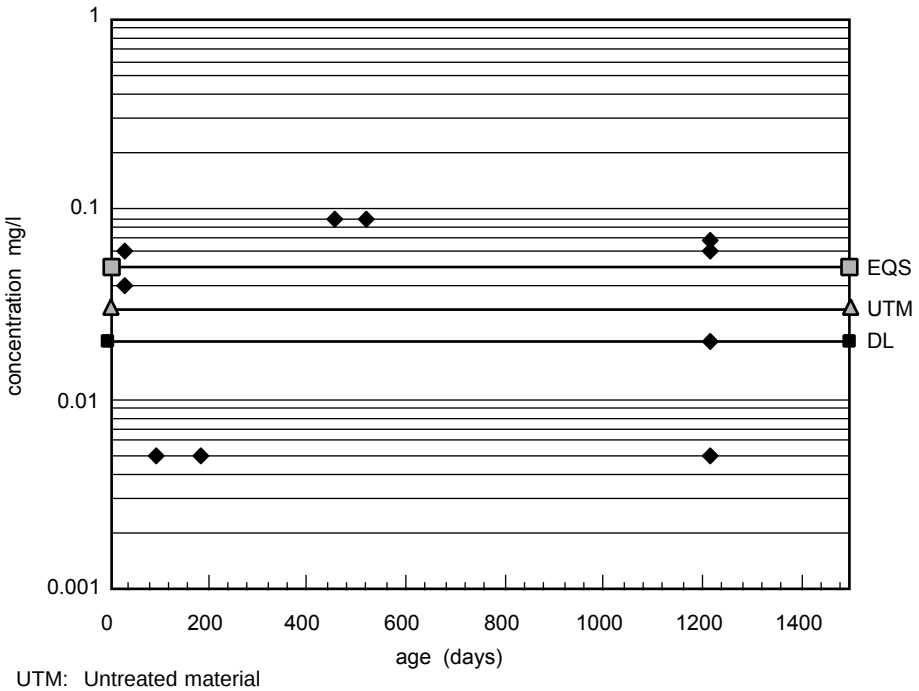
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 13 A1 concentrations leached with time from material B



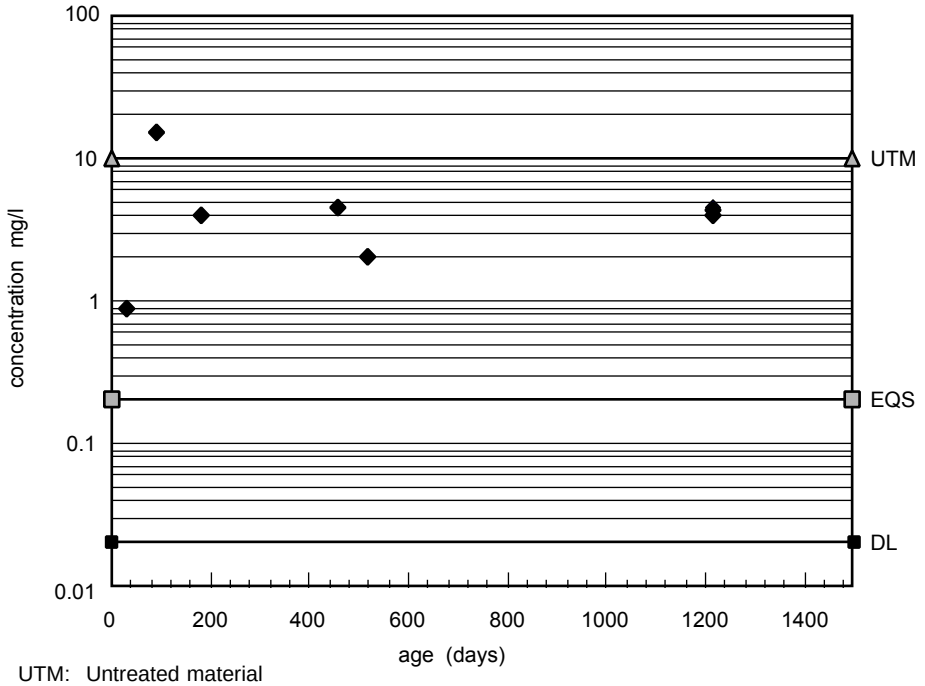
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 14 Pb concentrations leached with time from material B



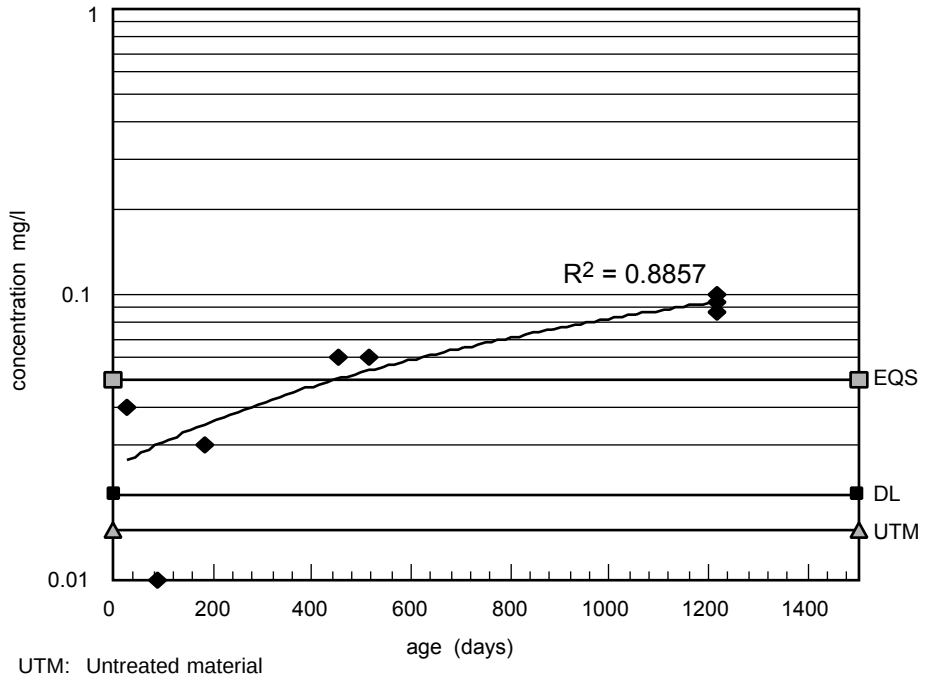
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 15 Cr concentrations leached with time from material B



UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 16 A1 concentrations leached with time from material C



UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 17 Cr concentrations leached with time from material C

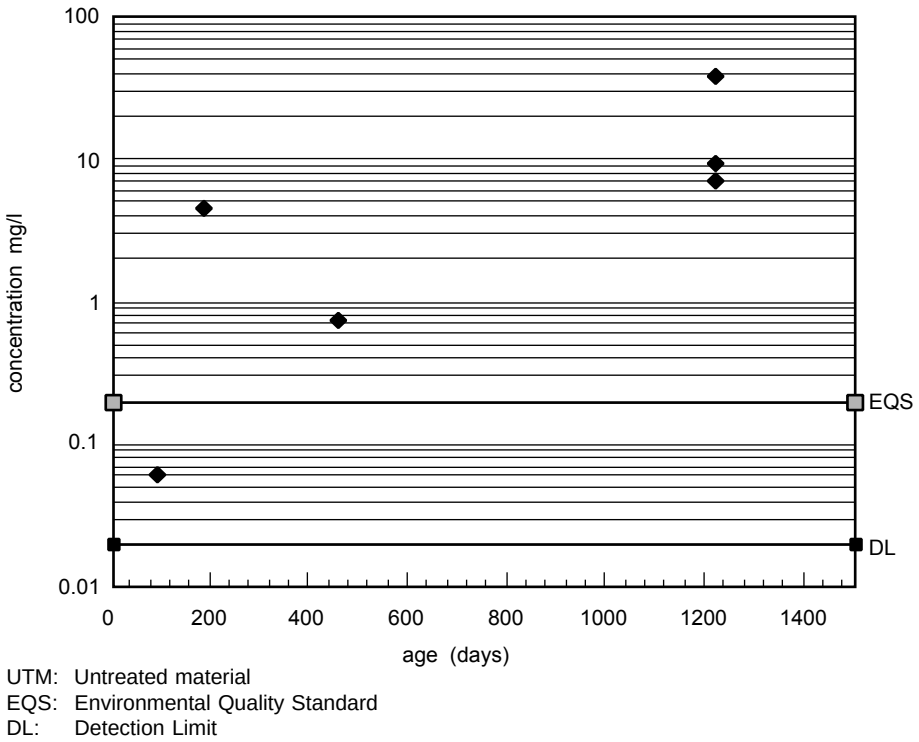


Figure 18 Pb concentrations leached with time from material D

9.11.5 Material E

Figures 19, 20 and 21 show the concentrations of metals As, Pb and Cr leached from material E. The leachate concentration of As was below the detection limit prior to 91 days but exceeded the UTM value by 182 days. However the leachate concentration of As did not exceeded the EQS value during the trial period. The material E leachate concentration of Al exceeded both the EQS and UTM values during the entire test period. The leaching of Pb from material E (Figure 20) produced a different leaching pattern compared to the other S/S materials. Leachate concentrations of Pb were constantly below the UTM. At 1216 days the concentration of Cr had decreased to below the EQS and UTM values.

9.11.6 Material F

Figures 22 and 23 show that significant concentrations of Al and Pb were leached from material F. The material F leachate concentration of Al (Figure 22) exceeded the 0.2mg/l EQS value throughout the trial period, but remained below the UTM value throughout the same period. Leachate concentrations of Pb from material F (Figure. 23) ranged from below the detection limit to equal to the 0.05 mg/l EQS value. Complex cyanide was continually leached from material F above the UTM value throughout the trial period. Prior to treatment complex CN did not leach from the UTM. The increased leachability is considered to be due to the higher pH environment within the S/S material causing dissolution of the CN species. Iron-cyanide complexes only adsorb to oxide minerals at pH <6 (Shefchek et al, 1995).

9.12 Summary

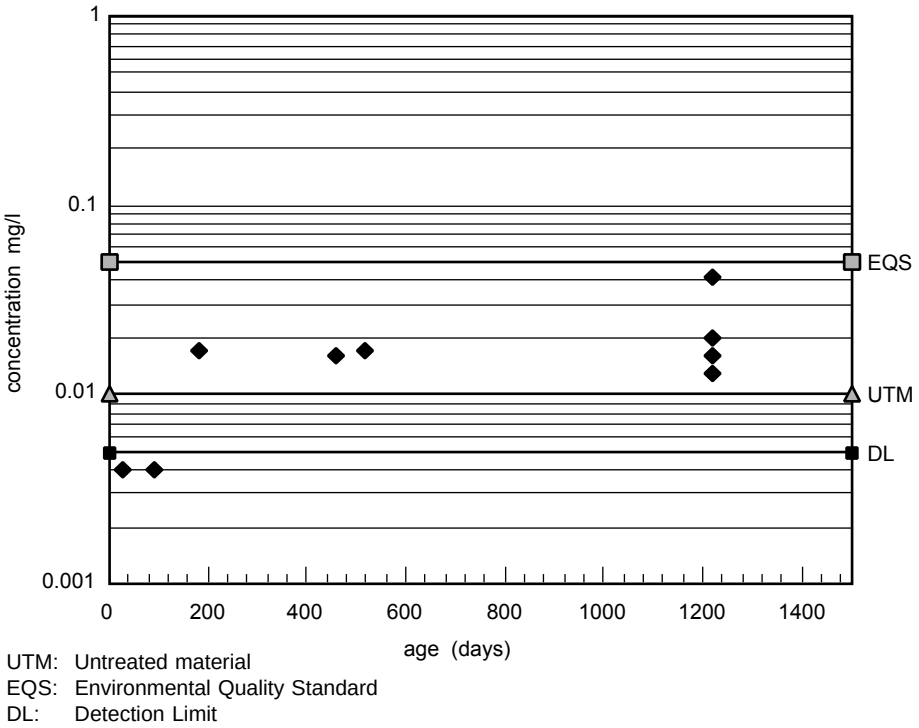
Al was leached above its EQS value by all UTM leachate samples. All six S/S materials also exceeded the EQS value for Al at some time during the trial period. In S/S materials A and B, the leachate concentrations of Al exceeded the UTM values. By 1216 days, all leachate samples from five of the six materials (excluding material D) still exceeded the EQS value for Al.

Pb was leached at concentrations above EQS values by one or more samples of materials A, B, D, and E at some period during the trial. By 1216 days only materials B and D had leachate sample concentrations of Pb exceeding EQS values.

Cr was leached at concentrations above EQS values by one or more samples of materials A, B, C, and E at some period during the trial. No UTM leachate concentration of Cr exceeded the EQS value. Therefore the S/S treatment of the trial materials increased the leachability of Cr.

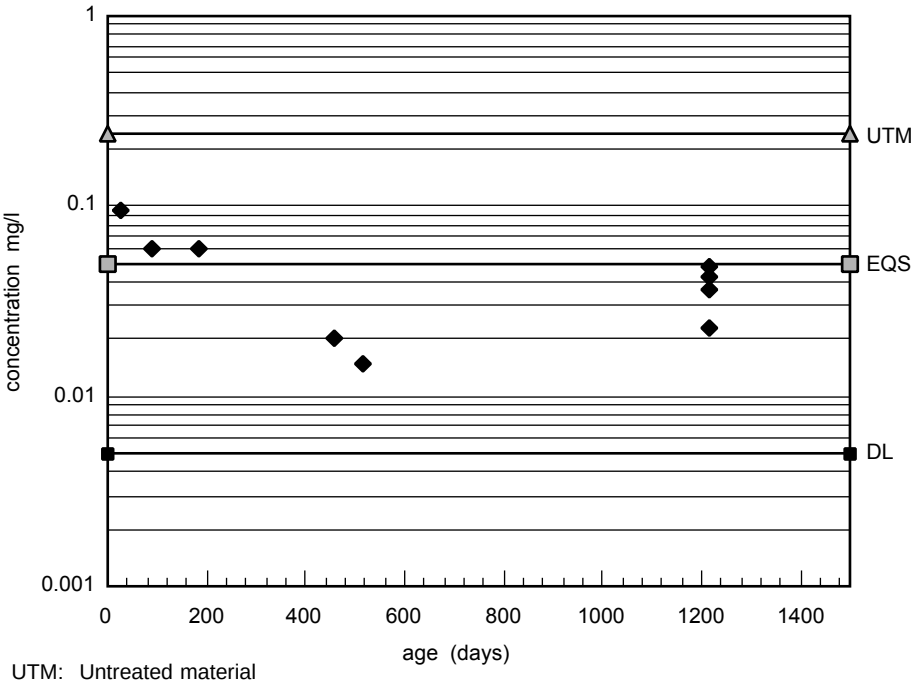
9.13 Leaching of other components

Leachate concentrations of components K, Fe and Ca were measured from only the 1216 day material samples. Leachate concentrations of K were found to exceed EQS values for all six materials. The EQS value for Ca was exceeded by leachate from materials A and F. The EQS value for Fe was exceeded by leachate from material F. These three components are not considered as substances dangerous to human health (Baldwin et al, 1997), but may have to be taken into consideration when assessing the impact of a leachate on aquatic life.



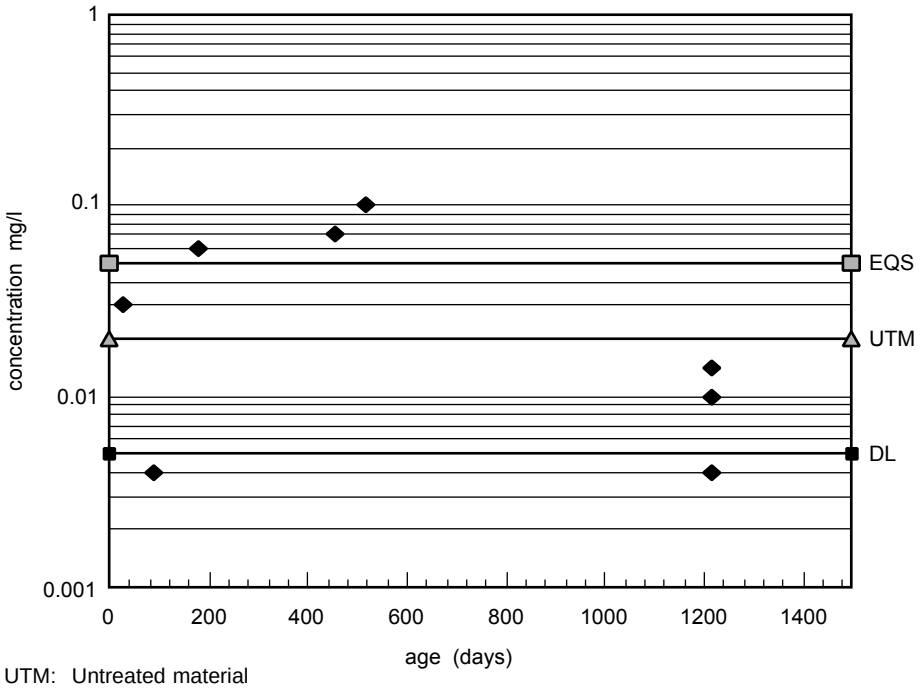
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 19 As concentrations leached with time from material E



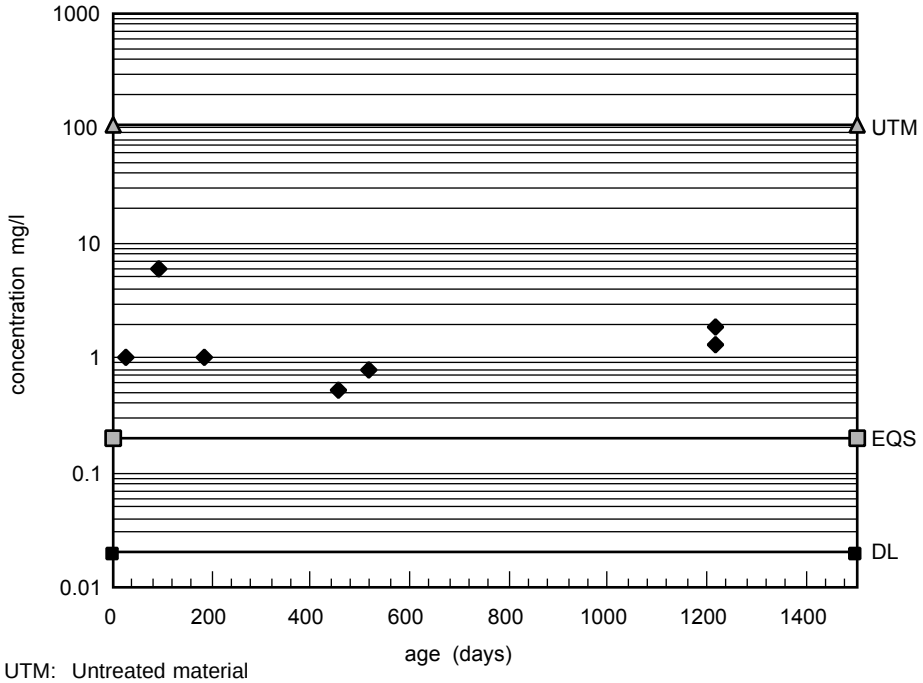
UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 20 Pb concentrations leached with time from material E



UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 21 Cr concentrations leached with time from material E



UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 22 A1 concentrations leached with time from material F

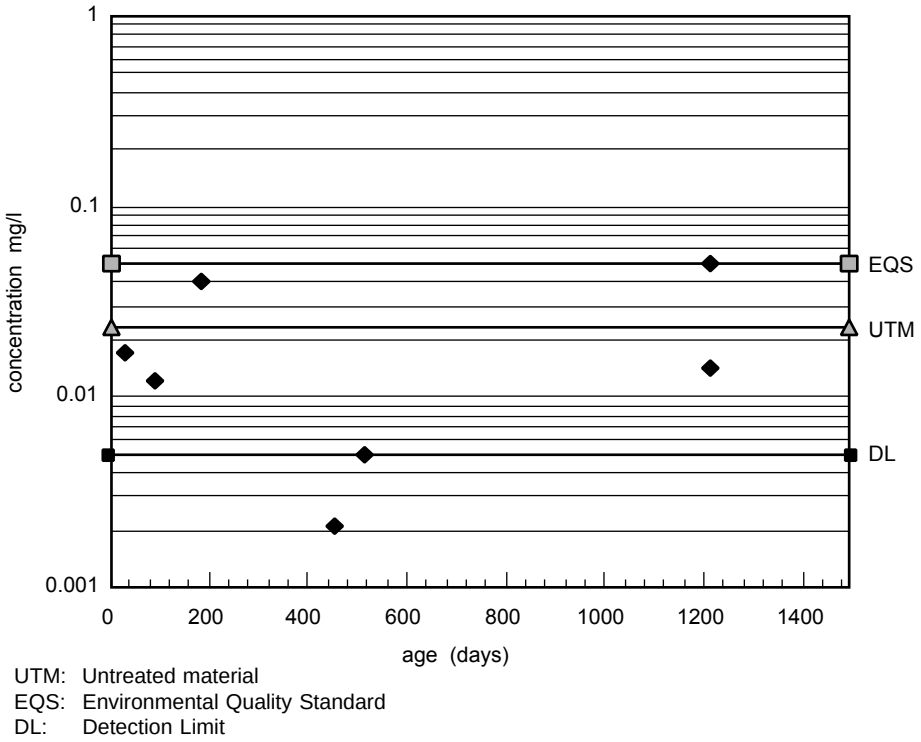


Figure 23 Pb concentrations leached with time from material F

9.14 Inorganic contaminants leached at 1216 days

The mean concentration of individual inorganic contaminants leached from the six materials at 1216 days, are shown in Tables 10 to 14. The mean concentrations were based on at least 3 samples, excluding material F for which only two leachate samples were tested. Standard deviation (Stdev) and Coefficient of variation (CV) were used to indicate the concentration range of an individual ionic contaminant for each material. CV is the standard deviation expressed as a percentage of the mean.

It is clear from these data that the coefficient of variation (CV) of a specific inorganic contaminant may be large e.g. ± 99% of the mean concentration for Cr leached from material A. The CV of inorganic contaminants is also seen to be large between the six materials. Cr (± 7-99%) and Pb (± 29-95%) showed the greatest range with Al (± 9-48%) and Cu (± 27-66%) having a smaller, but still considerable range.

Materials of similar composition such as A and B, D and E, showed wide ranges in the mean concentrations of individual metals leached. No relation is seen between the type of contaminated material treated and the mean concentration of inorganic contaminants leached from a S/S material. This finding clearly indicates the need for an adequate number of S/S material samples to be tested, in order to gain an accurate evaluation of S/S material performance.

9.15 Organic contaminant behaviour

9.15.1 Untreated material leachate concentrations

The analysis of UTM leachate showed no organic contaminant to be leached above the EQS values. However the UTM leachates were not analysed for TPAHs.

9.15.2 Organic contaminants immobilised by S/S treatment

The organic contaminant groups analysed for were petroleum and diesel range hydrocarbons, TPAHs, PCBs and total phenols. Hydrocarbons and PCBs were not found to have leached from any of the six S/S materials. All Phase 1 leachate samples tested had concentrations of hydrocarbons below the 0.25mg/l detection limit. However the detection limit of 0.25mg/l was above the hydrocarbon EQS value (0.01mg/l) for all Phase 1 and a number of the Phase 2 samples tested. Eight samples analysed during Phase 2 using a lower detection limit method of 10mg/l were all below the EQS value for hydrocarbons.

9.15.3 Organic contaminants leached

Figures 24 to 26 show the concentrations of total phenol (including chlorinated forms of phenol) leached from materials A, B and F. As in the previous section the figures show the DL, UTM materials leachate concentration and an EQS reference value. The EQS used for total phenols was the maximum concentration allowed by the World Health Organisation drinking water quality guidelines (Environment Agency, 1996b). However, nearly all the samples were above the UK drinking water limit of 0.5µg/l.

Total phenols were found to leach in significant concentrations from three materials A, B and F. Only leachate concentrations from materials A and F exceeded the EQS value for total phenol. Material F leachate concentrations of total phenol increased with time, showing a logarithmic relation (Figure 26). The leaching of phenol from the S/S materials appears to be greater than from the UTM.

Table 10 Statistical data for Al concentrations leached from S/S materials at 1216 days

Age 1216 days	*Material A*	*Material B*	*Material C*	*Material D*	*Material E*	*Material F*
Contaminant	Al	Al	Al	Al	Al	Al
EQS value (mg/l)	0.2	0.2	0.2	0.2	0.2	0.2
Mean NRA leachate conc (mg/l)	5.2	1.8	4.3	0	4.0	1.6
Stdev (± mg/l)	0.48	0.84	0.32	0	1.01	0.40
CV (± %)	9	48	7	N/A	24	25

Table 11 Statistical data for Cu concentrations leached from S/S materials at 1216 days

Age 1216 days	*Material A*	*Material B*	*Material D*	*Material F*
Contaminant	Cu	Cu	Cu	Cu
EQS value (mg/l)	3.0	3.0	3.0	3.0
Mean NRA leachate conc (mg/l)	0.48	0.22	0.04	0.15
Stdev (± mg/l)	0.14	0.06	0.03	0.07
CV (± %)	28	27	66	47

Table 12 Statistical data for Cr concentrations leached from S/S materials at 1216 days

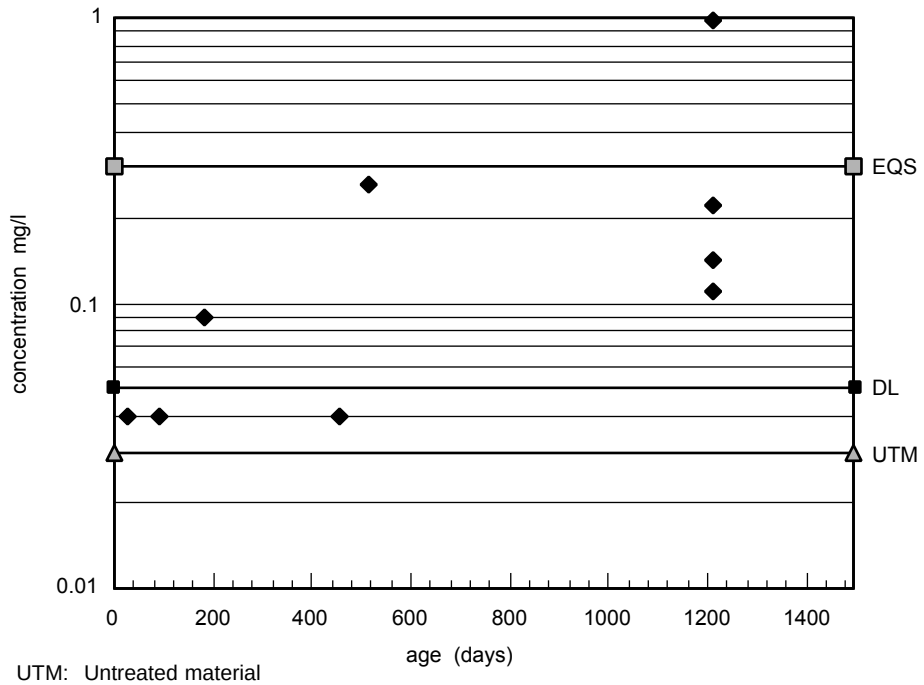
Age 1216 days	*Material A*	*Material B*	*Material C*	*Material E*
Contaminant	Cr	Cr	Cr	Cr
EQS value (mg/l)	0.05	0.05	0.05	0.05
Mean NRA leachate conc (mg/l)	0.06	0.04	0.09	0.01
Stdev (± mg/l)	0.06	0.03	0.01	0.01
CV (± %)	99	80	7	61

Table 13 Statistical data for Pb concentrations leached from S/S materials at 1216 days

Age 1216 days	*Material A*	*Material B*	*Material D*	*Material E*	*Material F*
Contaminant	Pb	Pb	Pb	Pb	Pb
EQS value (mg/l)	0.05	0.05	0.05	0.05	0.05
Mean NRA leachate conc (mg/l)	0.01	0.08	18.04	0.04	0.03
Stdev (± mg/l)	0.01	0.07	17.15	0.01	0.02
CV (± %)	Below DL	85	95	29	79

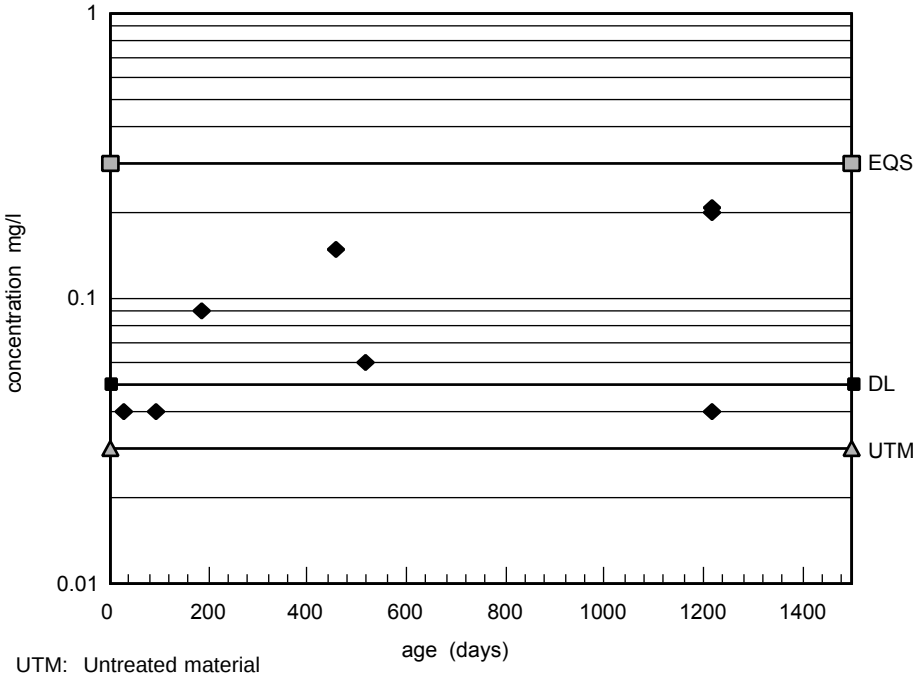
Table 14 Statistical data for As and CN anion concentrations leached from S/S materials at 1216 days Age 1216 days

	Material E	*Material F*
Contaminant	As	CN
EQS value (mg/l)	0.05	0.05
Mean NRA leachate conc (mg/l)	0.02	2.05
Stdev (± mg/l)	0.01	0.15
CV (± %)	58	7



UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 24 Total phenol concentrations leached with time from material A



UTM: Untreated material
EQS: Environmental Quality Standard
DL: Detection Limit

Figure 25 Total phenol concentrations leached with time from material B

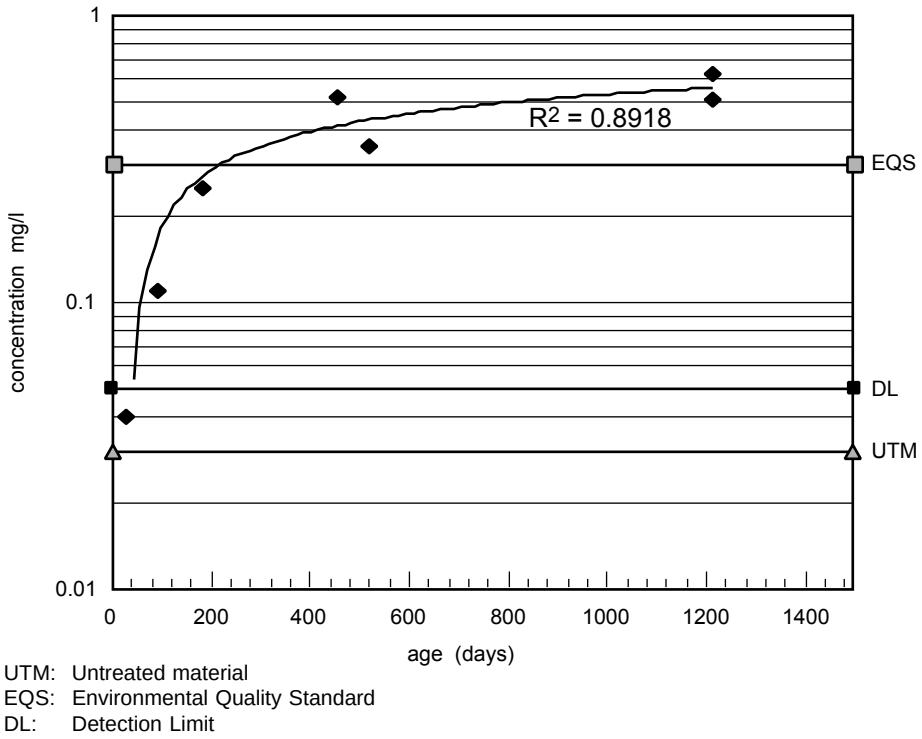


Figure 26 Total phenol concentrations leached with time from material F

However the sampling methods used for the UTMs were not detailed in Phase 1. Degradation of organic contaminants occurs rapidly and appropriate sampling methods must be used. The lack of phenol leaching from the UTMs compared with the treated samples may be due to the sampling and analytical procedures used during the two phases of testing.

The concentrations of TPAH leached from materials A, B, and F are shown in Table 15. The concentrations leached were higher than the EQS value of 0.2µg/l derived from drinking water standards. Leachate concentrations of TPAH from material A were higher than materials B and F, which leached similar concentrations of TPAH.

Table 15 NRA method leachate concentrations of TPAH from materials A, B and F

Material	*Core number*	*Age in days*	*TPAH (µg/l)*
A	—	91	<10
A	—	182	19
A	Core 4	1216	86
A	Core 9	1216	44
A	Core11	1216	52
A	Core13	1216	45
B	—	28	16
B	—	28	12
B	—	91	<10
B	—	182	15
B	Core 1	1216	13
B	Core 2	1216	14
B	Core 6	1216	10
B	Core 7	1216	13
B	sub-base	1216	9
F	—	91	<10
F	—	182	14
F	Core 35	1216	19
F	Core 41	1216	13

10 Comparison of findings with CIRIA Report 167

CIRIA Report 167 *Use of industrial by-products in road construction - water quality effects* by Baldwin et al (1997), examined the use of unbound industrial by-products in road construction. The report used a leachate test (comparable with the NRA leachate test method) to produce an evaluation of the pollution potential of a number of industrial by-products. The report concluded that many unbound industrial by-products could be used in road construction with no detrimental effects to water resources. The report recommended that leachate tests be used to categorise secondary aggregate materials into groups. The report also recommended that the grouping for a secondary aggregate should be based on the degree of dilution required to reduce the concentration of a contaminant in a leachate, to below the EQS value of a water resource.

Three material groups were identified:

Group1 *No restrictions to use based on potential to affect water quality.*

Leachate contains no EEC directive 76/464/EEC, List 1 substances over EQS limits. No EEC directive 76/464/EEC, List 2 substances

requiring more than 10x dilution to meet EQS values. No other species requiring more than 100x dilution to meet EQS values

Group 2 *May require some restrictions to use based on potential to affect water quality.*

Leachate contains no EEC directive 76/464/EEC, List 1 substances over EQS limits. No EEC directive 76/464/EEC, List 2 substances requiring more than 1000x dilution to meet EQS values.

Group 3 *Will need some restrictions to use based on potential to affect water quality.*

Leachate contains no EEC directive 76/464/EEC, List 1 substances requiring more than 1000x dilution to meet EQS values. No EEC directive 76/464/EEC, List 2 substances requiring more than 1000x dilution to meet EQS values.

Comparison of the organic and inorganic leachate data from the six materials produced the following reuse classifications. The groupings were found to be dependent on the age of the S/S material.

Group 1 (at 1216 days) Materials B , C, E and F*

Group 2 (at 1216 days) Materials A and D

* *All Group 1 materials were, at some period during the first 500 days of the trial classified as Group 2 materials.*

The short term effect of S/S treatment on the six contaminated materials was to increase the leachate concentrations of some components above their UTM leachate concentrations and the EQS values. The short term increase in leachate concentrations gave the S/S materials a Group 2 classification. However, the effects of ageing on the S/S materials were to reduce the potential effect of the S/S materials on water resources. Four out of six S/S materials met the criteria for Group 1, the lowest risk group.

CIRIA report 167 speculated that addition of binding agents may alter the leaching characteristics of unbound industrial by-products. This assumption was correct for PFA which was given a Group 2 classification by the CIRIA report. The PFA treated by this trial to form material C would be classified as a Group 1 type material on the basis of the tests at 1216 days.

10.1 Comparison of leachate data with a crushed concrete secondary aggregate

It has been recommended that assessment of any secondary aggregate should made by comparison with materials already used in road construction (Baldwin et al 1997). The reuse of crushed concrete as a secondary aggregate in road construction is a common practice (Sherwood, 1995).

In order to test the chemical suitability for reuse of the six materials, leachate data from a crushed concrete and the six materials were compared. A sample of commercial crushed concrete, graded to meet the Type 1 sub-base particle size distribution, was subjected to the Phase 2 chemical testing. The leachate concentrations were compared with those from the six S/S materials. It should be noted that crushed concrete is a variable material and the leachate test results will depend on the composition of the cement and aggregate used and the service conditions of the concrete prior to crushing.

Comparison of alkali metal concentrations leached from both materials showed that excluding material F, Fe was leached in similar concentrations from the crushed concrete and five S/S materials. The leachate concentration of K from the crushed concrete was below EQS values whereas all six S/S materials exceeded the K EQS value. However more extensive leachate testing of crushed concrete has shown leaching of K between 1-10 times the EQS value (Baldwin et al, 1997). The leachate concentration of Ca from the crushed concrete was just below the EQS value. Similar Ca concentrations were leached from four materials B, C, D, and E, with Ca leachate concentrations below EQS values.

Concentrations of Al leached from five S/S materials exceeded the EQS value. However the Al leachate concentration from the crushed concrete also exceeded the EQS value by a smaller but significant concentration. This result shows that Al leaching from S/S materials should not necessarily preclude reuse of the S/S materials in road construction. No other organic (PAHs and total phenol) or inorganic (As, Pb, Cu, Cr and CN) contaminant, found in certain S/S material leachate samples, was leached from the crushed concrete at concentrations exceeding the EQS value.

10.2 Comparison of site drain water with drinking water limits

The actual environmental impacts of the leachate from materials A and B should be seen in the surface run off and percolated water collected in the drains along the side of the main slab at the trial site (Figure 2). Generally the drain water was of high quality, see Table 16. However the water samples from both Drain 1 and Drain 2 contained concentrations of K and Se that exceeded UK drinking water limits. No data are available on the organic content

Table 16 Concentrations of inorganic contaminants in drain water samples

Contaminants in drain water samples (mg/l)	*Drain 1*	*Drain 2*	*UK drinking water limits*
As	<0.005	<0.005	0.05
Al	<0.02	0.03	0.2
B	0.57	0.39	2
Ca	32.5	34.7	250
Cd	<0.0005	<0.0005	0.005
Cr	0.017	0.025	0.05
Cu	0.04	<0.02	3
Fe	<0.02	<0.02	0.2
Hg	<0.0005	<0.0005	0.001
K	15.3	40.8	12
Mg	3.92	2.51	50
Mn	<0.02	<0.02	0.05
Na	9.75	22.9	150
Ni	<0.02	<0.02	0.05
Pb	<0.005	<0.005	0.05
Se	0.03	0.02	0.01
Zn	<0.02	<0.02	0.1
Total CN	<0.05	<0.05	0.05
pH	8.1	8.3	—
Conductivity µs/cm 20°C	300	450	—

of the drain waters. However as the organic contents of materials A and B were low compared to ICRCL values, the concentrations of organic contaminants in the drain waters are likely to be negligible.

10.3 Environmental impact of highways

The environmental effects of reusing S/S materials in highway construction should be viewed in context with other pollution issues associated with highways. A number of motorway discharges have been identified, including:

- Sediment particles of 63µm have been shown to transport a high proportion of sorped contaminants such as metals, hydrocarbons, salts and nutrients.
- Hydrocarbons consisting of petroleum, fuel oil, lubricating oil and hydraulic fluids have been shown to be present. Concentrations of 2-20µg/l of TPAH contaminants sorbed to sediment particles are reported.
- Cd, Cu, Fe, Pb and Zn are the common metals found in motorway discharges.
- Other discharges containing salts and minerals from de-icing applications, herbicides and pesticides are also found.

Sources of these discharges include traffic, accidental spillages, road and verge maintenance and agricultural activities. Comparison of S/S material leachate data, with concentration data of contaminants in highway surface run off, showed the leachate concentrations to be within the range of surface run off concentrations (Luker and Montague, 1994).

10.3.1 Highway drainage systems

Highway drainage systems have two functions. The principal function is to remove surface water. The secondary function is to provide adequate drainage for small amounts of ground water or percolated water passing through the highway pavement and sub-base. Disposal of liquid and solid discharges occurs through out falls to a receiving water course or soakage system (Luker and Montague, 1994). Research has shown that the use of filter drains within a highway drainage system can limit the discharge of contaminants to below EQS levels. However accidental spillages of fuel oil were found to have a greater environmental impact than highway discharges (McNeill and Olley, 1998). It may be concluded that the use of pollution prevention measures for discharges from modern highways should be capable of attenuating any leachate produced by a S/S material used in the highway construction.

11 Conclusions

This study investigated the effects of ageing on the physical and chemical properties of six stabilised / solidified materials. The report included data from two independent phases of testing. Phase 1 demonstrated the application of S/S technology to treat three contaminated soils, two metal smelting slag residues, and a PFA. Phase 1 testing was conducted jointly by CIRIA and VHE Construction plc. The results are given in Jardine and Johnson (2000). The University of Portsmouth conducted Phase 2 testing with funding from the Highways Agency, administered by TRL. The S/S materials examined by Phase 2 were made available by VHE Construction plc. A secondary objective of the Phase 2 study was to assess the suitability of S/S materials for reuse in highway construction.

Phase 1 of the trial was conducted at short notice without the opportunity to carry out a study to determine the optimum mix design for each material. The same mix design was used for each material. No physical or chemical specifications were set for the S/S materials. These factors limited the interpretation of the data from the trial.

The 28 day unconfined compressive strengths achieved by all six S/S materials exceeded the former WRA requirements for S/S materials and were greater than documented site examples. PFA based material C was predicted to achieve the highest compressive strength. Materials B and D, containing significant concentrations of inorganic contamination, developed slightly greater mean long term compressive strength than material C. Contamination appeared to control the long term strength of materials A and E. The strength of material F was controlled by the particle size distribution of the untreated material.

This trial has shown that contamination may cause time dependent effects on the early and long term compressive strength of S/S materials. However the effects vary greatly between S/S materials produced from similar contaminated materials. Treatment process factors such as particle size distribution of the untreated material may have a greater effect on compressive strength than low levels of contamination.

Crushed B material taken from the trial site was subjected to physical tests detailed in the MCHW 1 and designed to determine the suitability of a material for use in road construction. The crushed material B has been shown to be suitable for:

- Earthwork Class 1 and 6 applications.
- Sub-base Type1 material.
- As an aggregate in a cement bound material.

The six materials were analysed to determine the total chemical content of components within the materials. Samples of the six materials were also subjected to the NRA leaching test method. Analysis of the leachate showed metals Cd, Hg, Ni, and Zn were effectively stabilised by the S/S treatment, as were low concentrations of hydrocarbons and PCB organic contaminants.

Metal contaminants Al, Cr, Pb and organic contaminants total phenols and TPAHs were shown to have leached from one or more of the treated materials, at concentrations greater than environmental quality standard values. However comparison of the leachate data with dilution factors determined by CIRIA Report 167, showed materials B, C, E and F to pose no threat to a water resource. Potentially no environmental restrictions to the reuse of the materials B, C, E, and F in road construction would be required. Materials A and D were found to pose a potential threat to a water resource that may require some

restrictions on the reuse of the material in road construction. These two materials had the same pollution category as unbound PFA, which is already used in road construction applications.

The leachate concentrations of contaminants from the S/S materials were found to be similar to concentrations found in normal discharges from highway runoff. Modern highway drainage systems with pollution control are considered to be more than adequate to deal with the leachate produced by a S/S material in a sub-base application.

The lack of testing of aged stabilised / solidified materials produced by commercial processes, has been highlighted (Hills and Pollard, 1997). The deficiency of research in the application of commercial S/S technology has resulted in inertia and the slow acceptance of S/S technology as a remediation option in the UK. This trial has shown that contaminated materials can be effectively treated using cement stabilisation / solidification technology.

12 Further work

A programme of further research and dissemination is required to show industry and regulators that the use of cement stabilisation / solidification is an effective remediation technology, and to demonstrate the potential for reuse of S/S materials.

A standard leachate test method for cement stabilised / solidified materials incorporating the effect of solidification on contaminant immobilisation is required. Environmental guidelines for the reuse of cement stabilised / solidified materials such as the dilution factors proposed in Baldwin *et al.* (1997) will aid the reuse of treated materials in construction applications.

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15 Plates

Plate 1 Belmix batching plant used for S/S treatment

Plate 2 Construction of main slab December 1994

Plate 3 Drilling of Phase 2 core samples

Plate 4 Packaging of Phase 2 core samples

Plate 5 Calcareous precipitate from material D slab

Plate 6 Carbonation of material C slab surface

Abstract

This study investigated the effects of ageing on the physical and chemical properties of six stabilised / solidified materials. The materials were analysed to determine their total chemical content and subjected to the NRA leaching test method. Analysis of the leachate showed metals Cd, Hg, Ni, and Zn were effectively immobilised, as were low concentrations of hydrocarbons. Metal contaminants Al, Cr, Pb and organic contaminants phenols and TPAHs leached from one or more of the treated materials. Comparison of the leachate data with environmental quality standards and published dilution factors, showed four stabilised / solidified materials posed no threat to a water resource. Other findings from this study showed that contamination controlled the early strengths of all six materials and the long term strength of two materials. A crushed stabilised / solidified material met the physical and environmental specifications required for earthwork Class 1 and 6 materials, aggregate in a cement bound material and Type 1 sub-base road construction materials. The lack of testing of aged stabilised / solidified materials produced by commercial processes, has been highlighted by previous research. This study has shown that the physical and environmental properties of several stabilised / solidified materials improved with age.

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