



BMT Cordah Limited
ENVIRONMENTAL CONSULTANCY
AND INFORMATION SYSTEMS

Use of Oily Drilling Wastes in Civil Engineering Applications

A report for Cordah Research

BMT Cordah Limited,
Scotstown Road,
Bridge of Don, Aberdeen,
UK, AB23 8HG.
Tel: +44(0)1224 414200
Fax: +44(0)1224 414250
Email: main@bmtcordah.com
Website: www.bmtcordah.com

Report No:	BMT Cordah Ltd/COR.027/2002
Status:	Interim
Version:	Final
Date of Release:	September 2002
Terms:	The contents of this report are confidential. No part thereof is to be cited without the express permission of BMT Cordah Ltd or Cordah Research.

Approved and authorised for issue:

Thushari Hapuarachchi
Consultant

Marianne Lang
Principal Consultant

Contents

1	Use of oily drilling waste in civil engineering applications	5
1.1	Introduction	5
1.1.1	Project background	5
1.1.2	Structure of report	7
1.2	Background to the issue of oily drilling waste	7
1.3	Scope	8
1.4	Need for this study	9
1.5	Funding of work programme	9
2	Properties of oily drilling waste	11
2.1	Introduction	11
2.2	Origin of waste	11
2.3	Characterisation of waste	12
2.4	Discussion of results	14
3	Cement-based solidification/stabilisation of oily drilling waste	23
3.1	Introduction	23
3.2	Choice of application	23
3.3	Formulation of mix designs	26
3.4	Laboratory trials	28
3.5	Field scale trials	29
3.5.1	Physical properties	30
3.5.2	Chemical properties	31
3.6	Testing and monitoring of trial products	49
3.7	The way forward	49
4	Conclusions	53

Appendix

1	Legal and regulatory perspective on drilling waste and its reuse	54
1.1	Classification of waste	54
1.2	Legislation issues related to the reuse of drilling waste	59
1.2.1	Landfill Directive	59
1.2.1.1	Pre-treatment	60
1.2.1.2	Waste acceptance criteria	61
1.2.2	National Waste Strategy	62
1.2.3	Landfill tax	64
1.2.4	Aggregate tax	64
1.2.5	Criteria for evaluation recycling/reuse processes	64
1.2.6	Duty of Care	65
2	Cement solidification /stabilisation – Description of the technology	67
2.1	Introduction	67
2.2	Cement-based solidification/stabilisation	68
2.3	Hydration reactions of cement	69
2.4	The interaction of cement and waste components – immobilisation potential	71
2.4.1	Internal pH-induced precipitation	72
2.4.2	Redox-controlled precipitation	74
2.4.3	Adsorption/occlusion into and onto C-S-H gel	75
2.4.4	Crystallochemical incorporation within the cement matrix	76
2.5	General considerations of using cement-based solidification/stabilisation for treating oily drilling waste	77
3	Method statements	
3.1	Preparation of test cubes (BS 1881 Part 108)	78
3.2	Curing of test cubes (BS 1881 Part 111)	79

List of tables

Table 1 –	Summary of sources of offshore oily drilling waste	12
Table 2 –	Inorganic species analysed in the oily drilling waste and the test methods adopted	13
Table 3 –	XRF results of the mineral phases present in oily drilling waste (wt %)	22
Table 4 –	Laboratory scale mix designs which produced CBM with compressive strengths between 1-4 N/mm ² at 28 days of cure	29
Table 5 –	Compressive strength of CBM batches trialled at field scale	31
Tables 6 -21 –	Tank and NRA test results of CBM produced during field trials (trials 1-16)	33 - 48
Table 22 –	Compressive strength data for test pad	49
Table 23 –	EU law/imminent UK law – List of waste considered to posses hazardous properties	57
Table 24 –	Hazardous Directive Annex III – Properties of waste which render them hazardous	58
Table 25 –	Basic reactions of cement hydration	70
Table 26 –	Crystalline products of hydrated cements	76

List of figures

Figure 1 –	Total petroleum hydrocarbon (TPH) content in oily drilling waste (wt%)	15
Figure 2 -	Concentration of polycyclic aromatic hydrocarbons (PAH) in oily drilling waste (mg/kg)	16
Figure 3 –	Concentration of copper in oily drilling waste (mg/kg)	17
Figure 4 –	Concentration of lead in oily drilling waste (mg/kg)	18
Figure 5 –	Concentration of zinc in oily drilling waste (mg/kg)	19
Figure 6 –	A comparison of copper, lead and zinc concentrations in oily drilling waste samples (mg/kg)	20
Figure 7 –	Concentration of chloride in oily drilling waste (g/l)	21

Figure 8 –	Concentration of sulphate in oily drilling waste samples (g/l)	22
Figure 9 –	pH of oily drilling waste samples in distilled water	23
Figure 10 –	Concentration of phenol in oily drilling waste samples (mg/kg)	24
Figure 11 –	Stepped side liner construction	24
Figure 12 –	Phasing plan during landfill operation	25
Figure 13 –	Flow chart of work programme for the formulation of cement-based material incorporating oily drilling waste	27
Figure 14 –	The waste hierarchy as set out in the NWS (Scotland)	63
Figure 15 –	Schematic showing the overall course of cement hydration	69
Figure 16 –	Summary of hydration process occurring during the first few hours or days	70
Figure 17 –	Solubility of metal hydroxides as a function of pH	74
Figure 18 –	Solubility of metal sulphates as a function of pH	76

(1)

Use of oily drilling waste in civil engineering applications

1.1 Introduction

This report describes the results of a work programme undertaken to assess the potential reuse of oil drilling wastes in general civil engineering works by stabilising and encapsulating within cementitious binders.

1.1.1 Project background

In June 2002, Cordah Research Limited contracted BMT Cordah to undertake large scale field trials on the use of oily drilling waste, including drill cuttings, in general civil engineering applications. The aim of the project was to assess the commercial feasibility of producing cement-based products with unprocessed oily drilling waste through a comprehensive programme of structural integrity testing to evaluate the suitability for structural applications and contaminant leachability to evaluate the impact upon the environment. This report describes the results of the first phase of the work programme which was undertaken.

The participants of the research project included:

- ◆ Cordah Research Limited - funded the project through the Landfill Tax Credits scheme;
- ◆ BMT Cordah Limited - project manager for the research programme;
- ◆ On-site Land Solutions Limited - site managers for the large scale field trials;
- ◆ Leiths (Scotland) Limited - sub-contractors for product formulation at bench scale and compressive strength testing of products at both bench and field scale;
- ◆ Land-Drill Geotechnics (UK) Limited – sub-contractors for all analytical work including leach testing.

Stoneyhill Landfill Site (SLS) in Peterhead was chosen as the location for conducting the large-scale field trials. SLS located approximately 30 km north of Aberdeen is a former granite quarry, extending to an area of approximately 3.7 hectares. The site has a remaining capacity in the order of 700,000 tonnes of waste and is classified as non-hazardous under the new Landfill Directive requirements. SLS was chosen for the research programme because of:

Reuse of oily drilling waste in general civil engineering applications

- ♦ the availability on site of a commercial-scale cement batching plant with a capacity of 20 tonnes/hour which was leased for the duration of the research;
- ♦ the availability of a large licensed waste management area for conducting the trials; and
- ♦ proximity to a well engineered landfill site within which the identified applications could be evaluated and monitored over time.

The large scale field trials were conducted under an exempt waste management activity registered with the Scottish Environmental Protection Agency (SEPA) by On-Site Land Solutions under Section 38(f) of Schedule 3 to the Waste Management Regulations. It was agreed by BMT Cordah, to allow Stoneyhill Waste Management the use of the cement batching plant when not used for research purposes, to produce concrete products from virgin material on the Stoneyhill Quarry Construction Site.

Unprocessed oily drilling wastes for the field trials were provided by a leading local waste management company. Upon delivery, the waste was stored in large storage containers with a capacity of 20 tonnes. Cement-based products produced from oily drilling waste during the research programme was placed and monitored within the stoneyhill landfill to ensure that any potential leachate created was contained within an engineered landfill.



The cement batching plant at SLS



The large storage containers on site for the oily drilling waste

1.1.2 Structure of report

This report consists of an introductory chapter (Chapter 1) containing information about the background to the project and to the issue of drilling waste, scope of work and funding of the work.

The second chapter explains the research conducted to assess the variability of drilling waste and a detailed discussion of the various characteristics investigated is included. Bench and laboratory scale programmes undertaken to investigate the reuse of drilling waste in general civil engineering is dealt with in Chapter 3, while the in-depth chemistry behind cement solidification/stabilisation technology is discussed in Appendix 2. The legal and regulatory issues governing disposal and recycle/reuse of drilling waste is included in Appendix 1. Method statements for the experimental procedures used during bench and field trials, is contained in Appendix 3.

1.2 Background to the issue of oily drilling waste

A wide range of waste streams is generated through drilling activity in the UK Continental Shelf (UKCS). 'Cuttings' arising from drilling with OBM and WBM make up a significant fraction of this waste; under present UKCS operating conditions, oil contaminated drill cuttings are returned to shore for disposal or where practical are re-injected. Increasingly, water-based mud cuttings are also returned to shore for disposal. It is estimated that some 80,000-100,000 tonnes of cuttings will be generated annually in the UKCS.

Drill cuttings are a subset of a bigger offshore waste stream – bulk contaminated liquids – of which over 150,000 tonnes per annum are brought ashore at a time when the only cost effective disposal route – to landfill, has been banned by EU legislation through the implementation of the new Landfill Directive. This means that this waste also has to be treated (driven by legislation) prior to land disposal.

A variety of onshore treatment processes have been developed to process/treat cuttings contaminated with organic phase based muds (i.e. OBM & SBM). Of these, Indirect Thermal Desorption is the most widely used. High temperatures are used to remove low molecular weight hydrocarbons to produce a fine powder which is subsequently land-filled. At an estimated processing cost of £100-150 per tonne, thermal desorption represents an estimated £13 m burden on the industry and as the treated material requires ultimate landfill, the industry potentially incurs an additional £2.6m per annum for disposal.

When onshore processing commenced in the UK, the industry issue was perceived as that of recovering valuable hydrocarbons from the drilling wastes to be re-used. All recovered solids were expected to be inert for disposal, or use as sand or gravel replacement. This approach did not take the complex waste management and recycling legislation into account. It has transpired that the law as currently interpreted means that the recovered residuals –

predominantly oil and dried solids - remain in the waste stream and are not recycled as previously anticipated. It has also been established that contaminants other than hydrocarbons such as chlorides and sulphates, also present a constraint in the objective of the system.

Cuttings re-injection (CRI) offshore is being viewed by the industry as a possible solution to the issue of onshore disposal and as such is being vigorously researched. However, CRI is not always feasible; the technique requires particular geological and reservoir characteristics and on availability of re-injection sites, i.e. redundant or dedicated wells. Suitable pumps and associated equipment need to be installed offshore. Furthermore, there are growing concerns regarding the definition of CRI as "landfill" under the terms of the EU Landfill Directive. Despite the various constraints, CRI may be a disposal option in certain instances but not in all cases and consequently the demand for onshore processing/treatment of drilling waste will remain.

1.3 Scope

The research programme evaluated the potential use of oily drilling waste in general civil engineering applications. The study focused on producing cement bound material (CBM) and mass concrete for use in two areas:

- ◆ Civil engineering applications within contained areas of landfill sites, e.g. lay-down areas, partitioning walls etc.
- ◆ General civil engineering applications out with the contained area of landfill, e.g. pavement foundations, sub-bases etc.

The key objective of the research was to assess the feasibility of the process on a commercial scale. Therefore the main focus was large scale field trialling backed by a laboratory programme to fine tune the chemical and physical properties of the CBM and concrete. The large-scale trials were used to highlight technical and other issues which may be significant at commercial-scale production.

The Overall research programme which was identified above was divided into different 'Phases' of work; investigation of the first area of applications, i.e., lay-down areas and partition walls, were considered as Phase I of the programme and investigation of applications out with the contained area of a landfill as Phase II of the programme.

The research also assessed the variability of the oily waste brought ashore. This involved collection and detailed analysis of oily waste samples from the various batches provided to SLS for the large scale field trials.

1.4 Need for this study and application of its results

As OSPAR legislation stands at present, oil based mud cuttings (OBM) containing more than 1% oil-on-cuttings by weight cannot be discharged at sea. To date, technology has not provided cost-effective processing plant capable of treating large volumes of cuttings to the required standards offshore. OBM cuttings are thus brought back to shore for treatment and/or disposal, which is mainly to landfill. Increasingly, water based mud cuttings (WBM) are also returned ashore for disposal mostly due to challenges set out in corporate environmental policy; these cuttings are contaminated with organic and inorganic chemicals and consequently disposal at sea, particularly in more sensitive marine environments, is avoided. Thus, changing environmental legislation together with increased corporate social responsibility has placed a considerable waste handling and disposal issue upon the oil operator companies and the waste management companies.

In light of the large volumes of drilling waste anticipated to be brought ashore, it is imperative that suitable re-cycle and re-use options are identified to avoid the need for bulk disposal of the material in to landfill, and that these options are demonstrated, on a scientifically sound basis, to be technically and legally robust. The research programme described herein is an attempt to address the issue of identifying a potential large-scale outlet for unprocessed/processed drilling wastes – in this case the field of civil engineering.

The pathway for end-use of a treated waste material will depend upon the fulfilment of specific criteria endorsed by the Scottish Environment Protection Agency (SEPA). Thus any study conducted will need to adopt a systematic approach which includes an extensive physical and chemical testing regime to evaluate the success of the treatment process. It is anticipated that such an approach would also overcome ambiguities regarding the definition of the term 'waste' as applied to any recycled material, a fact which is widely regarded by the waste management industry to have hindered the development of bulk markets for recycled drilling waste thus far. Niche markets have been investigated at present such as the creation of cycle paths by a pro-active Oil Operator company. However, the scale of the problem warrants the development of a large-scale outlet for recycled material and the purpose of this study was to investigate the use of recycled drilling waste in general civil engineering works through a systematic approach which satisfy the criteria set by SEPA and also the standards set by the construction industry.

1.5 Funding of work programme

The research programme outlined in this report was intended to be funded through the landfill tax credit scheme. However, in December 2002, the Chancellor announced the effective closure of the scheme for research and development work. Consequently, only Phase I of the broader research programme which was outlined within the original scope of work (section 1.2) could be conducted and the forward plan for the programme has been severely limited due to the lack of further funds. Phase I constituted a preliminary study to

evaluate the commercial feasibility of producing cement-based products for use within the contained area of landfills. A continuation of the work programme, i.e., Phase II outlined in the original scope of work would have built on and added additional value to the results of Phase I.

(2)

Properties of oily drilling waste

2.1 Introduction

The broad variability in the waste returned ashore is a significant issue when developing recycle, reuse options for oily drilling waste. Offshore waste arises from a range of activities which include, drilling, pit cleaning, well intervention and well clean up operations with the composition of the waste varying from light to heavily contaminated material containing significant levels of oil. To date there has not been an assessment made of the degree of variability in the waste. Parameters such as pH, suspended solids, chemical oxygen demand (COD) and oil content are noted by some waste management companies, but the practice is not wide spread and a detailed analysis of the total chemical composition is not included.

A key objective of this research programme was to assess the degree of variability of oily waste through a comprehensive programme of characterisation of the material. It is anticipated that such a study will not only facilitate the development of new technologies for recycling and reuse of oily waste, but also contribute to the improvement in management of drilling waste off and on-shore.

2.2 Origin of waste

Offshore drilling waste brought ashore arises from a variety of activities and therefore vary broadly both in appearance and chemical composition. The waste researched during the present study is variously referred to by the industry as 'oily wastes', 'bulk fluids' or 'oily slops' and may include drill cuttings as well as aqueous and semi-solid wastes. The origin of offshore waste is summarised in Table 1.

In addition to the obvious variability of waste due to source, other contributing factors include:

- ◆ rock formation characteristics;
- ◆ type and formulation of drilling fluids used;
- ◆ technology used for the cleaning and separation of cuttings offshore.

Cuttings comprise a large proportion of the drilling waste that is brought ashore, although quantities of other oily wastes arriving at quaysides for treatment/disposal are rising sharply. These wastes may be lightly or heavily contaminated and contain varying amounts of oil; the

oil content can vary from less than 1% by weight of waste to approximately 30% by weight of waste.

Table 1 – Summary of sources of offshore oily drilling waste

Origin of waste	Description of waste produced
Exploratory and developmental drilling	Drilling muds (WBM, OBM, LTOMB), drill cuttings, well treatment fluids
Well clean-up operations	Brine, contaminated sea water, clean-up pills
Production Operations	Produced water, ballast water, displacement water, deck drainage, produced sand, cement residue, blow-out preventer fluid, gas & oil processing waste, slop oil, cooling water, desalination brine, fire control system test water
General activities	Sanitary and domestic waste
Infrequent operations	Construction waste, pigging waste, hydrostatic testing, pipeline maintenance
Tank washing	Waste from cleaning of supply vessel tanks
Accidents	Oil spills, gas blowouts, chemical spills

2.3 Characterisation of waste

Representative samples of oily waste as provided for the large-scale field trials were subject to a suit of tests to gather information on:

- ◆ metallic species present;
- ◆ water soluble chloride and sulphate content;
- ◆ the Poly-cyclic Aromatic Hydrocarbons (PAH) content;
- ◆ the Total Petroleum Hydrocarbons (TPH) content;

- ♦ mineral composition;
- ♦ density;
- ♦ pH in solution

The funding available for the research dictated the number of oily waste samples which was analysed; a total of 27 samples were obtained during the life span of the project and provided for analysis to a UKAS accredited testing laboratory, Land-Drill Geotechnics (UK) Limited, based in Aberdeen. In order to obtain a homogeneous specimen of the waste, samples were collected from the large storage tanks which store the waste on site, immediately after it was discharged from the bulk tankers which transported the material from the quay side to the landfill site. However, meaningful analytical data on the chemical composition could only be obtained from 23 samples due to difficulties in the preparation of specimens for analysis.

The complexity of the waste contributed to difficulties in determining the various inorganic species which may be contained within unprocessed oily drilling waste and the corresponding tests which should be conducted to determine their respective concentrations. However, it was agreed to test the material for the list of inorganic species presented in Table 2 upon the advice of the UKAS accredited testing laboratory who have previously conducted numerous analyses on various other drilling wastes.

Table 2 – The inorganic species analysed in the oily drilling waste and the test methods adopted.

Inorganic Species	Test Method	Inorganic Species	Test Method
Arsenic	DIHM*	Zinc	DIHM*
Boron	DIHM*	Chromium	DIHM*
Cadmium	DIHM*	Selenium	DIHM*
Lead	DIHM*	Sulphate (water soluble)	DIHM*
Mercury	DIHM*	Chloride (water soluble)	DIHM*
Copper	DIHM*	Sulphide	DIHM*
Nickel	DIHM*	Cyanide	DIHM*

DIHM* - developed in-house method

In term of the organic species to be tested, it was decided to determine the mineral oil content represented by the TPH values with the aid of Fourier Transform Infra-red spectrometry (FTIR). The PAH compounds which account for some of the TPHs were narrowed down to the 13 most commonly occurring species as dictated by the United States Environmental Protection Agency (USEPA). The various mineral phases present within the drilling waste was determined with X-ray Fluorescence (XRF) spectrometry.

The PAH compounds tested for within oily drilling waste

- Naphthalene
- Anthracene
- Benzo(b)/benzo(k) Fluoranthene
- Acenaphthylene
- Fluoranthene
- Benzo-a-pyrene
- Acenaphthene
- Pyrene
- IPDA
- Fluorine
- Benzantracene/chrysene
- Benzo(g,h,i) perylene
- Phenanthrene

The mineral phases tested for within the drilling waste

- Na_2O
- P_2O_5
- TiO_2
- MgO
- SO_3
- MnO
- Al_2O_3
- Cl
- Fe_2O_3
- SiO_2
- K_2O
- BaO
- CaO
- LOI^*

LOI^* - Loss on Ignition

2.4 Discussion of results

The wide variability amongst the waste is very apparent from the data in Figures 1 to 10. The detailed chemical breakdown obtained for the individual samples are included in Appendix 3.

The oil based nature of the waste is represented in the levels of TPH within the samples which range from less than 1% by weight of sample up to 35% by weight of sample. The

main sources of TPH are the base fluids used in oil-based drilling muds and to a lesser degree, the crude oil which the cuttings may come into contact with down the bore hole.

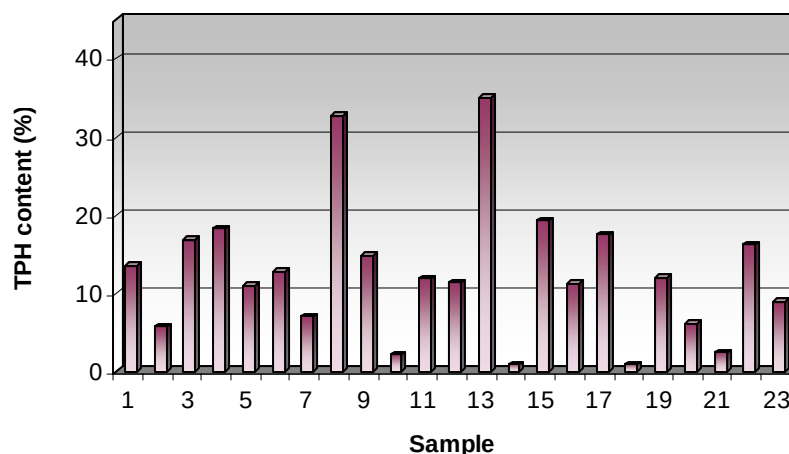


Figure 1 - The total petroleum hydrocarbon (TPH) content in oily drilling waste samples (% w/w)

The PAH levels within the samples remain low with only one sample showing levels in excess of 1000 mg/kg. In the majority of the specimens, PAHs were below the instrument detection limit and in the few specimens which contained detectable levels of PAH, Naphthalene, Acenaphthylene and Acenaphthene were the three most prevalent compounds; samples were tested for 13 different species as included in the USEPA list of priority pollutants (1988) which contains 16 PAH compounds based upon the impact on the environment and to general human health. Naphthalene is one of three low toxicity hydrocarbons ($C_{10} - C_{15}$) commonly used in base fluids contained in oil-based drilling muds. The other species may have come from a variety of sources due to the 'mixed' nature of the waste.

A comparison of the TPH and PAH data shows that polycyclic aromatic compounds account for only a small proportion of the TPHs present in the waste. In the case of some specimens, significant levels of TPH is observed while the PAH levels are below the detection limit of the instrument.

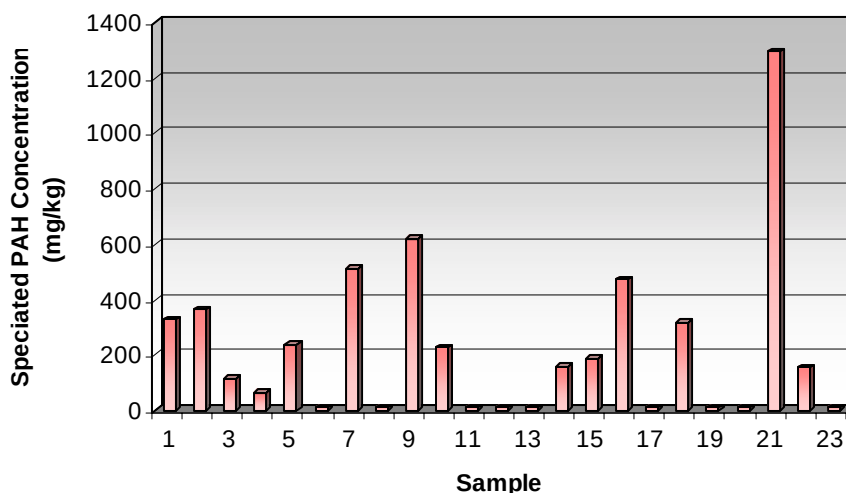


Figure 2 - The concentration of polycyclic aromatic hydrocarbons (PAH) in oily drilling waste (mg/kg)

Metallic species such as boron, cadmium and selenium were found to be below the detection limit of the instrument in all samples analysed. Concentrations of nickel, chromium and arsenic were also very low with the exception of a few specimens where nickel level up to 43 mg/kg and arsenic levels up to 24 mg/kg were detected. But on the whole, these levels can be considered to be fairly low.

Lead, copper, zinc and chromium however, were found in significant concentrations. Samples which showed detectable levels of one heavy metal generally also tended to contain other heavy metals as depicted by Figure 6.

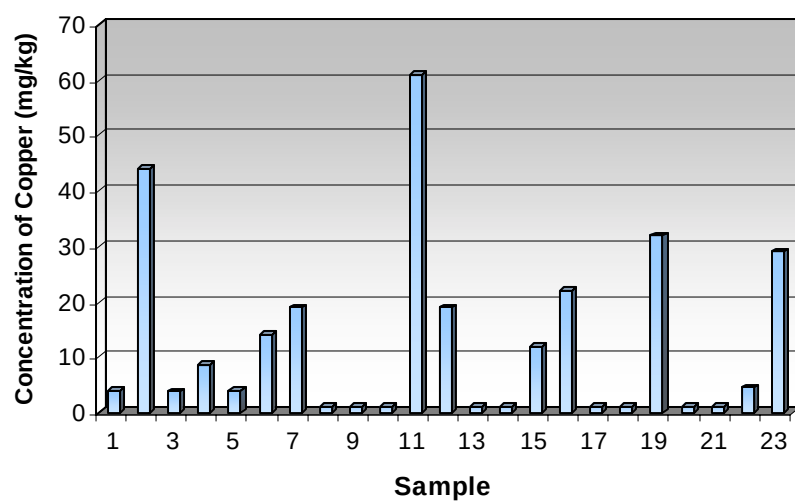


Figure 3 – The concentration of copper in oily drilling waste samples (mg/kg)

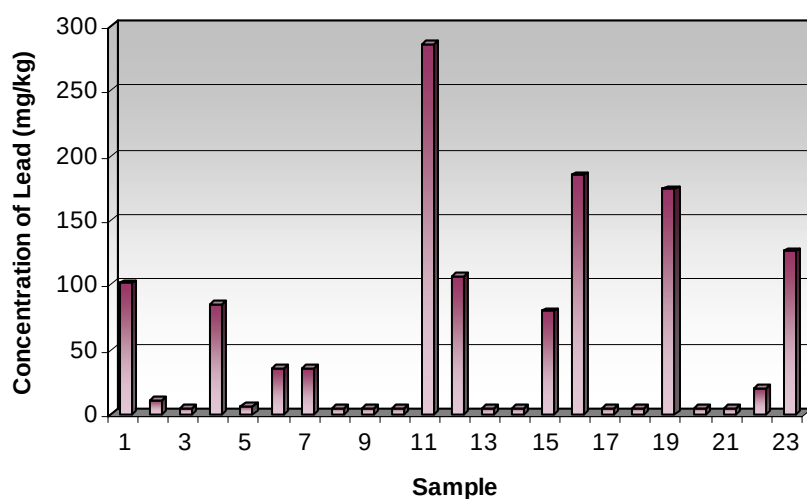


Figure 4 – The concentration of lead in oily drilling waste (mg/kg)

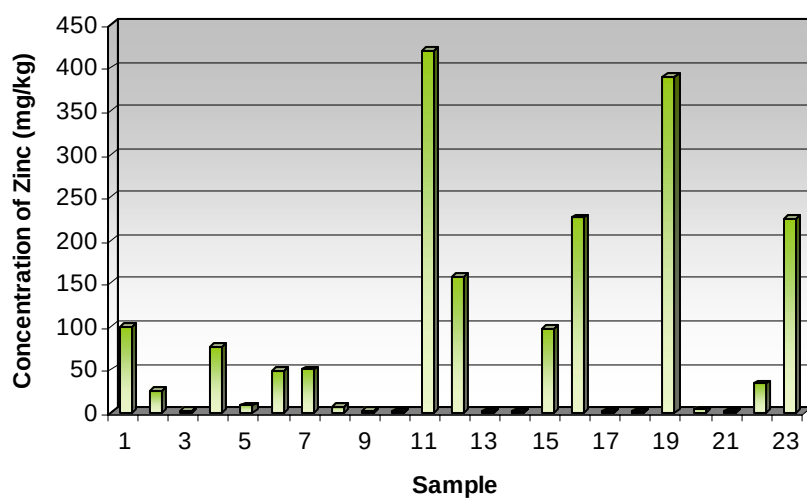


Figure 5 – The concentration of zinc in oily waste samples (mg/kg)

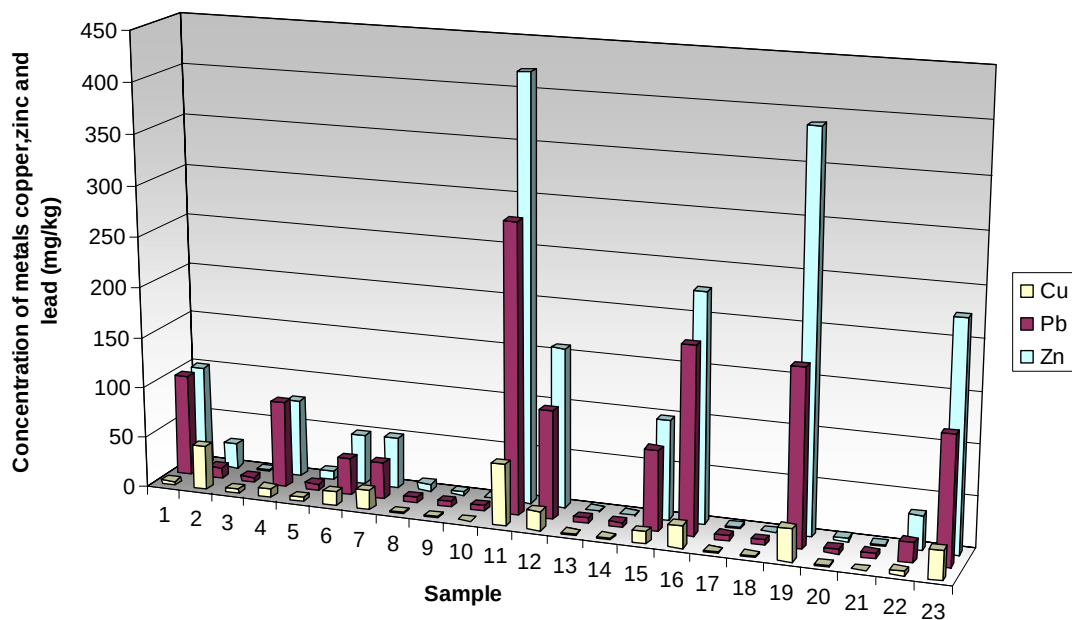


Figure 6 – A comparison of copper, lead and zinc concentrations in oily drilling waste samples.

The main source of metals in the waste is the barium sulphate (barite) which is used as a weighting agent in drill mud formulations. Depending upon the purity of the barite, the amount of metals could vary significantly and account for the variation seen amongst the oily waste samples analysed. Zinc and lead are found in comparatively larger concentrations than copper. The reasons for this are not known.

Sea water is commonly used at offshore installations for cleaning purposes as well as to 'slurrify' drill cuttings for ease of transportation to onshore processing plants. Thus it was decided to test the chloride (Cl^-) and sulphate (SO_4^{2-}) content of the waste; results are depicted in Figures 7 and 8. The leachability of these anions will also be of significance when developing recycle/reuse options for the waste. As with other species evaluated, Cl^- and SO_4^{2-} varies widely from one batch to the next reflecting the variability of the waste.

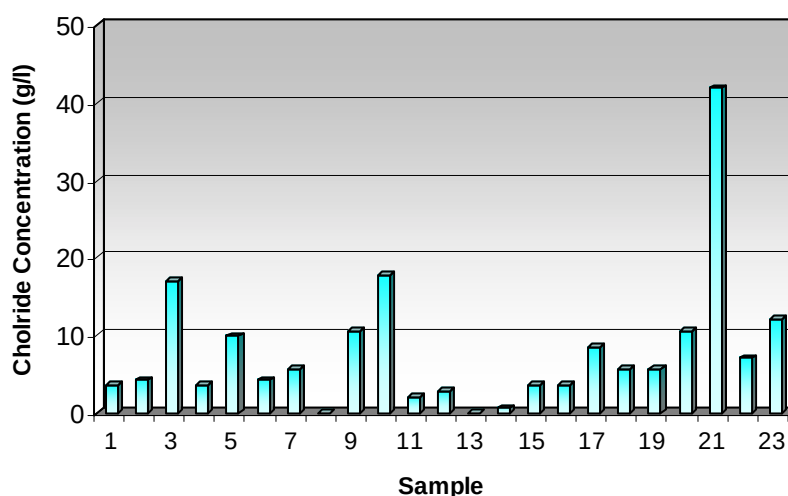


Figure 7 – The concentration of Chloride in the oily waste (g/l)

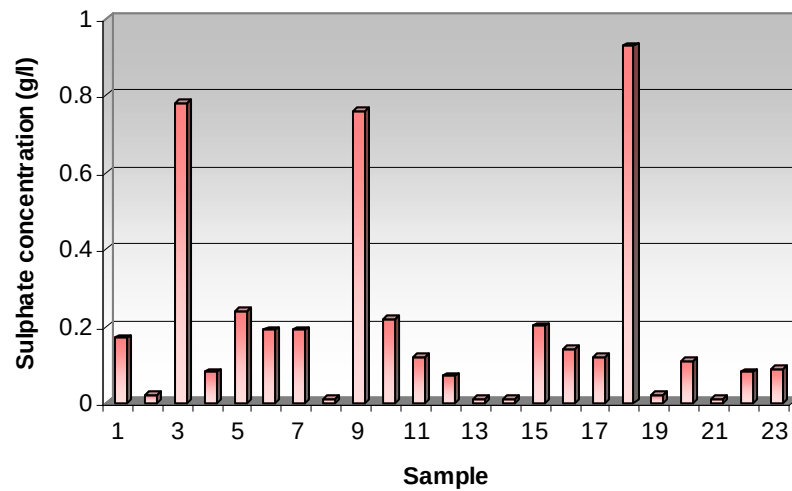


Figure 8 – The concentration of sulphate in oily drilling waste samples (g/l)

The pH of the waste in distilled water varied generally between 6 and 10, with the exception of a few samples which exhibit values in excess of 10. The higher pH values may result from the various additives which may be contained in the mixed waste.

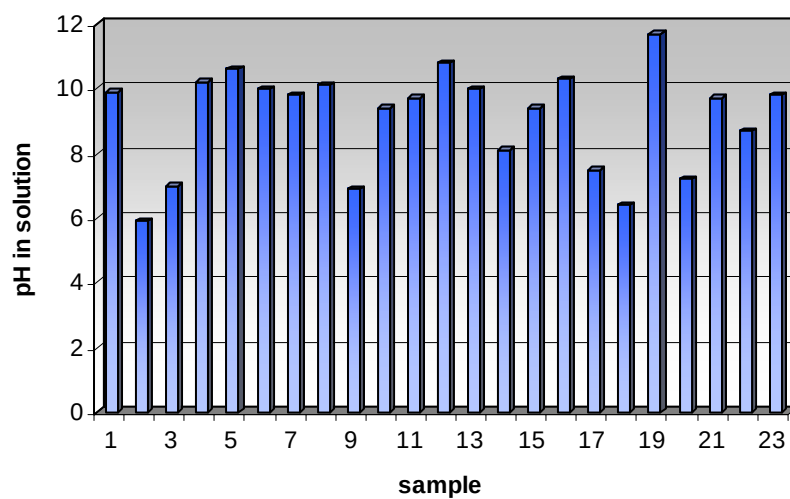


Figure 9 – pH of oily drilling waste samples in distilled water

The phenol content of oily waste was generally very low with the majority of samples showing concentrations of less than 5 mg/kg. A few samples did however, exhibit higher concentrations but this may have been due to errors during sample preparation and analysis. A main source of phenol is biocides used in drill mud formulations to prevent bacterial activity within drill muds during storage on offshore platforms.

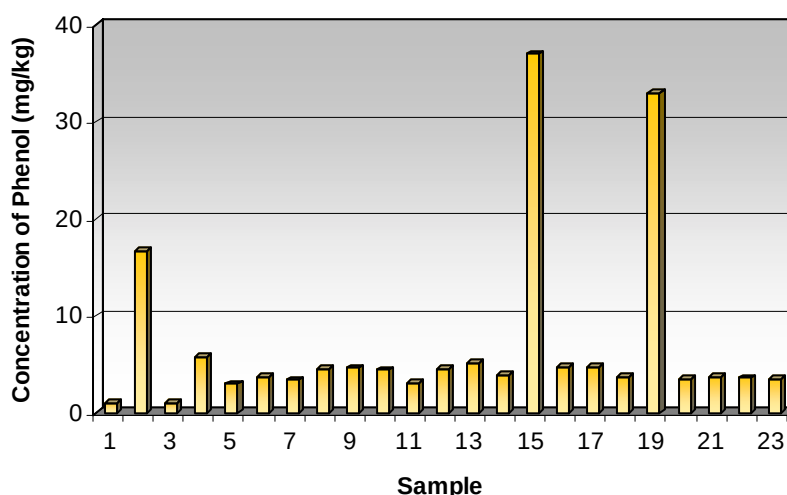


Figure 10 – The concentration of Phenol in oily drilling waste samples (mg/kg)

Useful results pertaining to the mineral composition of the various waste samples could only be obtained from a total of 13 specimens out of the original 27 samples collected for the study. This data depicted in Table 3 shows oxides of barium (BaO) and silica (SiO_2) to be the predominant mineral phases present within oily wastes. SiO_2 is the main constituent of sand which is present in varying quantities in the waste; this is reflected in the results which show SiO_2 to vary from 6 wt% to approximately 20 wt%. Similarly, oxides of barium can be attributed to the large quantities of the weighting agent barite present in used drill muds. The BaO_2 content varied from 20% w/w in the waste to approximately 50% w/w. As barite is contained primarily in drill cuttings, variation in the BaO_2 content is an indication of proportion of cuttings contained within the oily waste. Thus based on the BaO_2 results, the amount of cuttings in the waste appear to vary widely, providing a clear indication of the 'mixed' nature of the drilling waste arriving at quay sides.

Reuse of oily drilling waste in general civil engineering applications

Mineral	1	2	3	4	5	6	7	8	9	10	5	8	10	11	12	13	14	15	16	17	18	19	20	21	22	23
Cd	226	<0.5						2.52	1.25	1.88	<0.1	3.20	1.18	1.49	2.42	0.86	2.75	0.78	2.20							
Cr								0.91	3.36	18.1	0.02	7.22	10.2	0.75	6.47	0.03	0.07	2.15	14.9	33.1						
Cu								3.36	0.44	0.50	0.03	3.23	1.15	1.99	4.23	0.92	4.17	2.46	2.41	2.58						
Ni								18.3	8.66	6.51	6.71	18.6	11.2	13.0	20.9	11.8	20.2	19.1	14.4	14.8						
P ₂ O ₅								0.02	0.12	0.12	0.31	0.07	0.17	0.15	0.10	0.16	0.10	0.18	0.16	0.08						
SO ₃								7.22	17.4	11.9	19.9	8.65	14.4	12.7	8.08	18.1	9.47	16.1	13.7	8.56						
Cl								10.2	3.37	4.25	2.69	4.96	2.45	3.36	3.01	1.40	3.75	2.03	2.10	5.72						
K ₂ O								0.75	0.46	0.43	0.35	0.87	0.46	0.49	0.83	0.55	0.79	0.82	0.61	0.42						
CaO								6.47	4.13	3.54	2.69	6.24	3.60	5.44	3.24	4.08	2.77	6.19	4.91	8.28						

Reuse of oily drilling waste in general civil engineering applications

MnO	0.07	0.15	0.06	0.22	0.09	0.15	0.14	0.09	0.10	0.09	0.17	0.13	0.10
Fe₂O₃	2.15	2.30	0.98	3.12	2.62	2.58	2.77	3.01	2.51	3.05	2.74	3.72	2.49
BaO	14.9	45.4	30.1	49.5	21.9	41.6	36.5	20.0	46.7	22.4	30.1	35.7	20.5
LOI	33.1	15.4	39.2	13.5	28.2	20.1	20.6	32.5	11.9	28.6	18.3	19.6	28.9

Table 3 - XRF results of the mineral phases contained in oily drilling waste (% wt)

Mineral	Sample												
	2	3	4	5	8	10	11	14	15	16	18	21	22
Na₂O	2.52	1.25	1.88	0.76	3.20	1.18	1.49	2.42	0.86	2.75	0.78	2.20	
MgO	0.91	3.36	18.13	0.02	7.22	10.21	0.75	6.47	0.03	0.07	2.15	14.93	33.1
Al₂O₃	3.36	0.44	0.50	0.03	3.23	1.15	1.99	4.23	0.92	4.17	2.46	2.41	2.58
SiO₂	18.31	8.66	6.51	6.71	18.62	11.24	13.08	20.95	11.80	20.29	19.16	14.47	14.88
P₂O₅	0.02	0.12	0.12	0.31	0.07	0.17	0.15	0.10	0.16	0.10	0.18	0.16	0.08
SO₃	7.22	17.43	11.93	19.95	8.65	14.47	12.70	8.08	18.17	9.47	16.16	13.72	8.56
Cl	10.21	3.37	4.25	2.69	4.96	2.45	3.36	3.01	1.40	3.75	2.03	2.10	5.72
K₂O	0.75	0.46	0.43	0.35	0.87	0.46	0.49	0.83	0.55	0.79	0.82	0.61	0.42
CaO	6.47	4.13	3.54	2.69	6.24	3.60	5.44	3.24	4.08	2.77	6.19	4.91	8.28

Reuse of oily drilling waste in general civil engineering applications

TiO₂	0.03	0.02	0.03	0.01	0.02	0.02	0.03	0.04	0.02	0.05	0.01	0.02	0.02
MnO	0.07	0.15	0.06	0.22	0.09	0.15	0.14	0.09	0.10	0.09	0.17	0.13	0.10
Fe₂O₃	2.15	2.30	0.98	3.12	2.62	2.58	2.77	3.01	2.51	3.05	2.74	3.72	2.49
BaO	14.93	45.43	30.12	49.50	21.94	41.69	36.55	20.09	46.78	22.48	30.14	35.74	20.52
LOI	33.1	15.4	39.2	13.5	28.2	20.1	20.6	32.5	11.9	28.6	18.3	19.6	28.9

(3)

Cement-based solidification/stabilisation of oily drilling waste

3.1 Introduction

The main aim of this study was to conduct a broad ranging programme which looked at the suitability of cement-based products incorporating unprocessed oily drilling waste in various civil engineering applications. The overall approach was to initially focus on applications within contained areas of landfills, and pending the success of the research, to extend the scope of the work to include general civil engineering applications out-with landfills. To the researchers' knowledge a dedicated work programme which looked at reuse options of unprocessed drilling waste has not been conducted thus far. The above outlined approach was therefore adopted to conduct a systematic investigation of the integrity and quality of cement-based products which could be formulated with oily drilling waste, and to evaluate the applications for which they can be successfully used.

The work reported in this document constitutes the first phase of the programme, i.e., applications within the contained area of landfills. Landfills were an obvious choice for the initial phase of work due to the carefully engineered nature of the site which allows the performance of products to be monitored in a 'real' environment, while minimising any potential impact upon the environment. A key objective of the research was to assess the commercial feasibility of the production process. Thus the main focus was large-scale field trialling backed by a laboratory programme to 'fine-tune' the physical and chemical properties of the various products.

3.3 Choice of Applications

Two possible applications were identified within the contained area of a landfill where cement-based material containing oily drilling waste could potentially be used. These are:

- ◆ the construction of lay-down areas which provide a stable surface above the compacted waste for vehicle movements; and
- ◆ the construction of partition or bund walls which separate the individual 'cells' within the landfill.

These applications required the formulation of cement bound material or CBM which is pavement material that uses cement as a binder. CBM is currently categorised into CBM 1-5 with compressive strengths ranging from 4.5 N/mm² to 12.5 N/mm² at 7 days of curing,

depending upon the application. The varying grades of CBM are mechanically superior, and often a cheaper alternative, to crushed rock or bituminous bases. Thus correctly formulated CBM can provide many superior properties to unbound materials used in pavement foundation and base layers. Phase 1 focussed on producing CBM1 commonly referred to as 'soil cement' by combining oily drilling waste with Portland cement and sand.

SLS is operated as a fully engineered landfill employing the internationally accepted and recognised "composite" approach to lining non-hazardous landfill facilities; a flexible membrane liner made of high density polyethylene to a thickness of 2mm has been placed above a low permeability mineral layer. The mineral layer consists of a 300 mm thick layer of bentonite enhanced soil with a maximum permeability of $1 \times 10^{-9} \text{ ms}^{-1}$.

The side slopes of the containment bunds are lined with a 3 metre thick layer of natural clay sourced locally. A second geosynthetic clay liner is placed between the natural clay and the waste to ensure complete containment. Although the regulatory specification for the natural clay layer is a thickness of 1 metre⁰, a thicker layer of 3 metres is required to allow equipment movement during the placing and compaction of the clay.

Due to the steep side slopes of the modified quarry, the natural clay liner is constructed in steps as the level of waste builds up within the landfill as shown in Figure 11. Traditionally, the 'lay-down' areas adjacent to the side containment bunds, which provide a firm level slab over the compacted waste for vehicle movements during receiving of the natural clay, have also been constructed from clay. As each step or 'lift' of the side liner is completed, the contaminated clay (due to contact with the waste beneath) of the 'lay-down' areas is 'scrapped off' and compacted against the geosynthetic clay liner providing a barrier between the virgin clay on the outside and the waste on the inside. This design provides further protection against potential leaching of contaminants from the landfill.

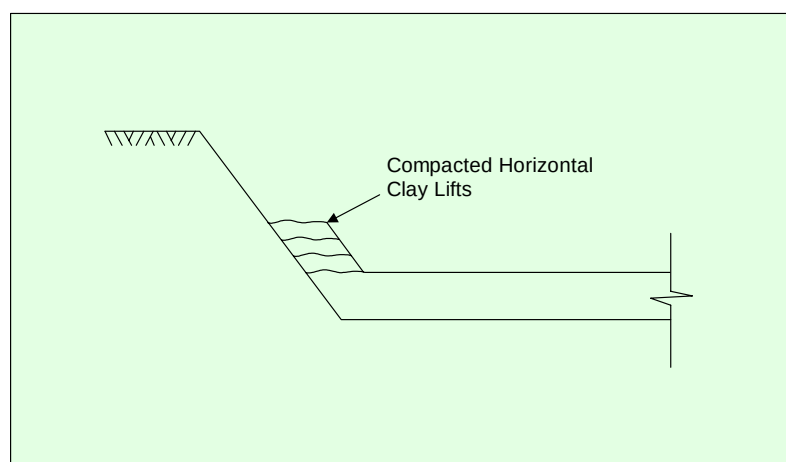


Figure 11 – Stepped side liner construction

The use of natural clay for the 'lay-down' areas within the landfill has several disadvantages:

- ◆ The maximum thickness of the clay layer should not be more than approximately half a metre – a thicker layer is prone to deep 'cracking' with continued vehicle movement over time, exposing the compacted waste beneath;
- ◆ Potential contamination of virgin clay used in the side liner – as virgin clay is piled on the lay-down areas prior to placing and compaction, there is a potential for contamination by coming into contact with the waste beneath;
- ◆ Unfavourable weather conditions hinder vehicle movements on the clay surface;
- ◆ Use of large quantities of a natural resource – as clay compacts with continued vehicle movement, it must be consistently replenished to maintain thickness of surface.

CBM was identified as being an alternative for natural clay when constructing the 'lay-down' areas as it provides a much more stable working surface with structural integrity.

A fully engineered landfill such as Stoneyhill is operated in accordance with a 'phasing plan' which means the disposing of waste in one phase until it reaches the final grade as shown in Figure 12. The partition walls or bund walls between the different phases are traditionally constructed by compacting a soil mix comprising mostly of sand and gravel. Although this technique has been used effectively thus far, it has the disadvantage of reducing the space available for the waste within each phase owing to the wide, 'pyramidal' shape in which the partition walls are constructed to impart stability to the structure. Replacing the sand and gravel with CBM would impart structural integrity to the partition walls, thereby enabling them to be constructed in a more linear manner, which in-turn increases the capacity within the individual phases.

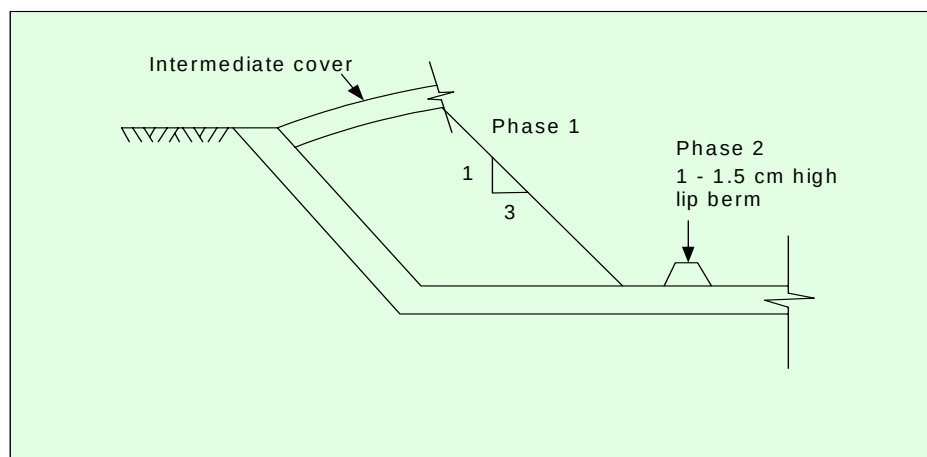


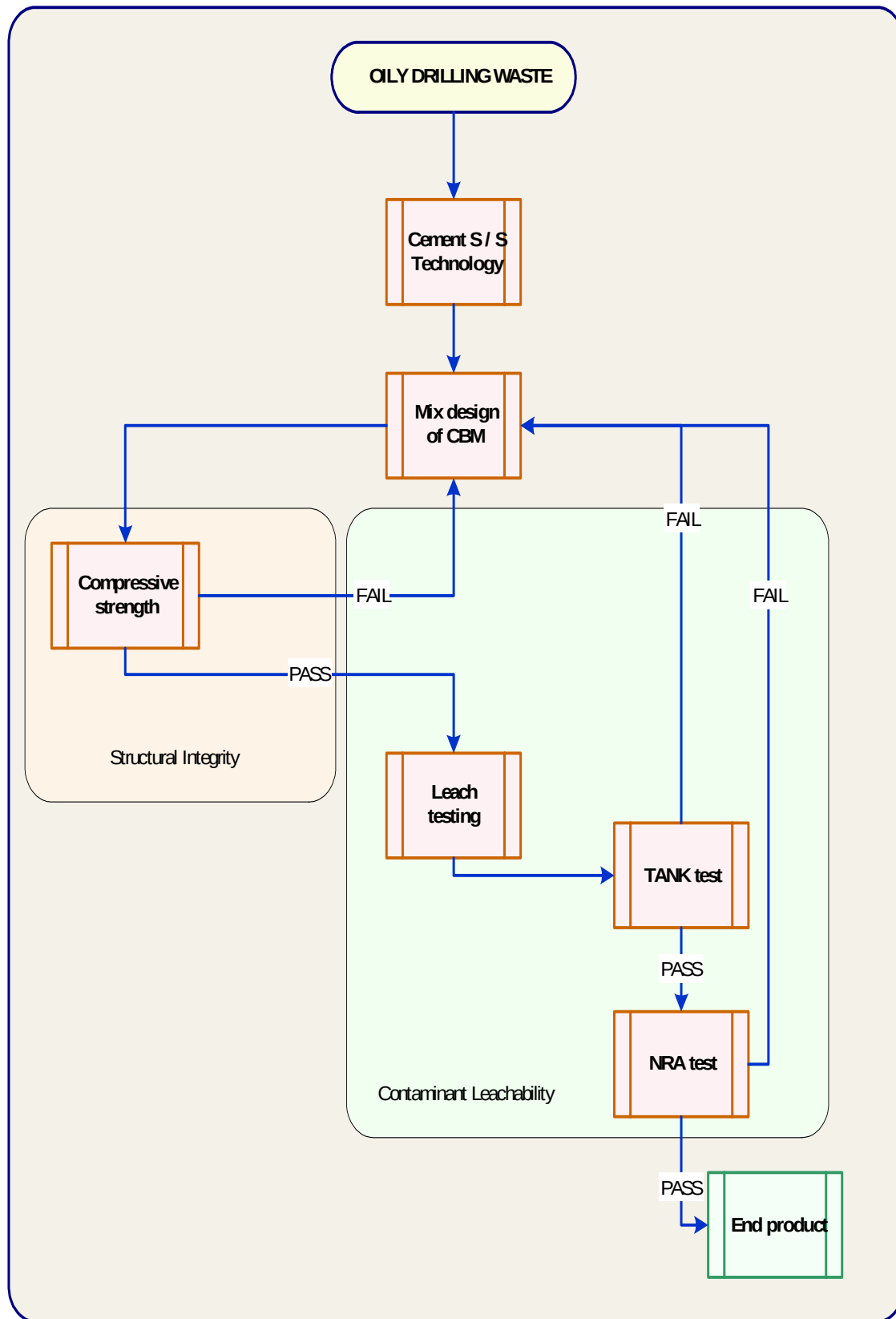
Figure 12 – Phasing plan during landfill operation

3.4 Formulation of Mix Designs

A key objective of the research was to assess the feasibility of the process on a commercial scale. Thus, the main focus was large scale field trialling backed by a laboratory programme to achieve the physical and chemical properties which were required from the CBM, i.e., the mix designs were fine-tuned in the laboratory prior to adopting it on site. It was however, imperative that the two processes were conducted concurrently, i.e., once a mix design had been formulated to the required physical properties (compressive strength) at bench scale it was tested at field scale by trialling large runs of CBM on site. This was necessary as various technical and other issues that may be significant at commercial scale production will only be highlighted when formulations are trialled at large-scale as opposed to bench-scale. Mix designs that proved to be successful after bench and field scale tests were finally tested for contaminant leachability with a view to commercial production for a specified civil engineering application.

The iterative procedure which was adopted when formulating CBM at bench-scale is depicted in Figure 13; the first stage of design satisfied the required physical properties in terms of compressive strength and the second stage satisfied the durability requirements in terms of contaminant leachability. Two separate leach tests were conducted in succession to evaluate leachability - the 'tank' test, recommended for monolithic material and the NRA (National Rivers Authority) test which is usually recommended for granular material. The NRA test represented a 'worse case' scenario in terms of durability. Method Statements for compressive strength testing is detailed in Appendix 3.

Figure 13 - Flow chart of work programme for the formulation of cement-based material incorporating oily drilling waste



3.5 Laboratory trials

Oily waste was mixed with binder which was a blend of Ordinary Portland Cement (OPC) and pulverised fly ash (pfa). Sand sourced from the Stoneyhill quarry was used to improve the compressive strength of the mix designs. Dosage levels of oily waste, binder and sand were varied to achieve CBM with compressive strength of 1-4 N/mm² at 28 days of curing. Due to the extended turn-around times involved in assessing contaminant leachability from the laboratory formulations, contrary to the original programme stated in section 3.4 it was decided to base the choice of formulations to be adopted on site on compressive strength development over time, i.e., only the section denoted 'structural integrity' in Figure 13 was tested.

A key strength parameter in cement products is the water to cement ratio. In general, although a high w/c ratio improves workability, it decreases strength. The inconsistency of the oily waste proved it was infeasible to vary the w/c ratio at bench-scale. This would also have introduced operational difficulties when trialling the formulations at field-scale due to greater scale of operation. The mix formulation was therefore modified by using the oily waste as the hydrating medium to render a mix of cement and sand workable.

The data in Table 4 contains formulations that produced CBM with the required compressive strength. Strength development over 28 days was similar in all formulations although mixes 1 and 4 which contained comparatively less oily waste in the blend, produced the higher strengths of 4.0 and 3.5 N/mm² at 28 days of cure. Both formulations contained the same level of binder which was a blend of ordinary Portland cement (opc) and pulverised fly ash (pfa) in proportions of 70 wt% and 30 wt% respectively.

It becomes apparent from data in table x that the oily waste content as well as the binder dosage in the blends, can have an effect upon strength development. This difference appears to be relatively minor at laboratory scale but may prove to be more significant when trialled at field scale, highlighting the importance of conducting a concurrent bench-scale and field-scale study to evaluate the commercial feasibility of the process.

Mix design 2 was chosen as the formulation to be adopted on site as it contained the highest dosage of oily waste in the formulation in comparison to mixes 1, 3 and 4. Despite the higher proportion of oily waste in the blend, compressive strength of mix 2 was still within the targeted values of 1-4 N/mm². Additionally, the cement dosage in mix 2 was lower than in the other mix designs, contributing to the cost effectiveness of the formulation. The economics of the process was an important factor when evaluating potential recycle-reuse options.

Table 4 – Laboratory scale mix designs which produced CBM with compressive strengths between 1-4 Nmm² at 28 days of cure

Mix	Constituents	Quantity (per m3)	Compressive Strength (N/mm ²)			
			3 days	7 days	14 days	28 days
1	Binder	200 kg	2.0	3.0	3.5	4.0
	Sand	1944 kg				
	Oily waste	210 litres				
2	Binder	200 kg	1.0	1.5	1.5	2.0
	Sand	1886 kg				
	Oily waste	333 litres				
3	Binder	175 kg	1.0	2.0	2.0	2.5
	Sand	1972 kg				
	Oily waste	229 litres				
4	Binder	200 kg	2.5	3.0	4.0	3.5
	Sand	1900 kg				
	Oily waste	220 litre				

3.6 Field-scale trials

Mix design 2 chosen from the bench-scale research was trialled using batches of oily waste as provided for the trials by a leading local waste management company. This was imperative for assessing the commercial feasibility of the process and the consistency in the quality of the product in terms of the compressive strength development and contaminant leachability.

Oily waste, binder and sand were blended in the mixing chamber of the batching plant for approximately 20 minutes to the proportions dictated by the chosen formulation. Samples were removed immediately after processing each batch by producing cement cubes (x 12) of dimension 100 mm × 100 mm × 100 mm in line with British Standard BS1881: Part 108 for compressive strength measurements and leach testing.

3.6.1 Physical properties

Compressive strength development was monitored over time by obtaining measurements after 7, 14 and 28 days of cure for the different batches of processed waste (see data in Table 5). The emphasis was on compressive strength at 28 days of cure and therefore it was decided to focus on 7 and 28 day measurements as there did not appear to be a significant difference in strength development between 7 and 14 days. Depending upon the number of CBM cube samples available at the time of testing, compressive strength data at 7, 14 and 28 days were obtained by testing multiple cube specimens; this is depicted by the multiple data points in Table 5.

The compressive strength of the different batches of oily waste varies generally between 1.0 N/mm² and 4.5 N/mm², as observed during the laboratory trials. The exception appears to be batch 12 which produced compressive strength of 6.5 N/mm² after just 6 days and values in excess of 14 N/mm² at 28 days. The reason for these high values in comparison to the rest of the data is not known but may be attributed to an error in the mixing chamber in terms of the quantities of raw materials processed.

The variation in strength development over time was attributed to the lack of homogeneity in the oily waste; the waste used for the trials varied from 'drill cuttings' containing very little hydrocarbons to oily liquids containing high levels of hydrocarbons. The presence of hydrocarbons impedes the hydration reactions of cement leading to poor strength development over time. Visual observations made on the appearance of the waste prior to treatment correlates with the measured compressive strengths - greater the solids content of the waste, greater the measured compressive strength at 28 days of curing.

Table 5 – Compressive strength of CBM batches trialled at field-scale

Trial no.	Compressive Strength (N/mm ²)		
	7 days	14 days	28 days
1	1.5, 1.5, 1.5	2.5	3.0, 3.0
2	1.0	-	1.5, 1.5, 1.5
3	2.5	-	4.5, 4.5, 4.0
4	3.0	-	3.5, 3.0, 3.0
5	-	-	5.0, 5.0, 5.5, 4.5
6	-	5.0	5.5, 5.5, 6.0
7	3.0	-	4.5, 4.5, 4.5
8	2.5	-	3.0, 3.0, 2.5
9	1.0	-	2.5, 2.5, 2.0
10	0.5	-	1.0, 1.0, 1.0
11	-	-	2.0, 1.5, 2.0, 2.0
12	6.5	-	15.0, 15.5, 14.5
13	1.0	-	2.5, 2.0, 1.5
14	1.5	-	3.0, 3.5, 3.5

3.6.2 Chemical properties

Despite the lack of homogeneity in the untreated waste, the leachability of contaminants from the solidified/stabilised products remains below the level of the instrument detection limit for all samples analysed – see Tables 6 to 21. Similar results are observed with both the Tank test which is appropriate for monolithic material and the NRA test where the cement samples are crushed to less than 10 mm to represent a worse case scenario for leachability.

With the exception of soluble chloride, leaching of all other contaminants listed in the Inter-departmental Committee for the Redevelopment of Contaminated Land 59/83 list (ICRCL 59/83 list) of contaminants remain below the stipulated trigger limits. The ICRCL 59/83 guidelines provide a list of trigger concentrations for potential contaminants in soil in relation to the end use of a site. It was decided to adhere to the ICRCL guidelines in the absence of specific regulatory guidelines for contaminant leachability from secondary products incorporating oily drilling waste. The New Dutch List assesses contamination of groundwater with the action value typically being used as unacceptable water quality for a minor aquifer. Contaminant

leachability from all samples analysed from the different large-scale trials fall below the action limits dictated by the New Dutch List.

Chlorides, however, pose a significant problem in terms of their presence in the product, and their propensity to leach out. The main sources of chloride in drilling waste are the weighting agents added to the drill mud formulations in the form of sodium and calcium chloride. Additionally, sea water is commonly used at offshore installations for cleaning purposes as well as to 'slurrify' drill cuttings for ease of transportation to onshore processing plants; typically sea water contains ~ 55 wt% of chloride (Cl)⁰. During cement solidification/stabilisation of the oily waste, a proportion of chlorides are incorporated within the solid phases of hydration and the remainder is mainly found dissolved in the pore fluid which constitutes the aqueous phase of a hydrating cement product. It is this dissolved chloride which is released from the s/s product during leach testing of the material. Thus, despite the high initial chloride leachability, a gradual decrease in the amount of leachable chloride is anticipated as the CBM weathers over time.

The overall low level of contaminant leachability, with the exception of chloride, from CBM was attributed to the small amount of oily waste incorporated in the product; hydrocarbons affect strength development by impeding the hydration reactions of cement. Consequently, to obtain CBM with appreciable compressive strengths, the proportion of waste that could be added to the cement/sand mix is limited. However, the variation in compressive strength measurements shows that however small the presence of hydrocarbons is, its impact upon strength development is significant.

Table 6 – Tank and NRA leach test results of CBM produced during field trials – trial no. 1. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.05	<0.05
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-		
Sulphate	0.1	0.2%	0.2%	2	6
Phenols	0.01	5	5	<0.1	0.15
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH	<5	<5		11.9	11.8
PAH:					
Napthalene	0.01				
Acenaphthylene	0.01				
Acenaphthene	0.01				
Fluorene	0.01				
Phenanthrene	0.01				
Anthracene	0.01				
Pyrene	0.01				
Benzathracene/Chrysene	0.01				
Benzo(b)/Benzo(k) fluroanthene	0.01				
Benzo-a-pyrene	0.01				
IPDA	0.01				
Benzo (g,h,l) perylene	0.01				
Total PAH	0.1	50	1000	<0.01	<0.01

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 7 – Tank and NRA leach test results of CBM produced during field trials – trial no. 2. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.05	<0.05
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	567	567
Sulphate	0.1	0.2%	0.2%	<5	<5
Phenols	0.01	5	5	0.12	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	12
PAH:					
Napthalene	0.01			<0.001	<0.001
Acenaphthylene	0.01			<0.001	<0.001
Acenaphthene	0.01			<0.001	<0.001
Fluorene	0.01			<0.001	<0.001
Phenanthrene	0.01			<0.001	<0.001
Anthracene	0.01			<0.001	<0.001
Pyrene	0.01			<0.001	<0.001
Benzathracene/Chrysene	0.01			<0.001	<0.001
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.001	<0.001
Benzo-a-pyrene	0.01			<0.001	<0.001
IPDA	0.01			<0.001	<0.001
Benzo (g,h,i) perylene	0.01			<0.001	<0.001
Total PAH	0.1	50	1000	<0.01	<0.01

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 8 – Tank and NRA leach test results of CBM produced during field trials – trial no. 3. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.05	<0.05
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	496	213
Sulphate	0.1	0.2%	0.2%	<5	<5
Phenols	0.01	5	5	0.13	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	11.8
PAH:					
Napthalene	0.01			<0.001	<0.001
Acenapthylene	0.01			<0.001	<0.001
Acenapthene	0.01			<0.001	<0.001
Fluorene	0.01			<0.001	<0.001
Phenanthrene	0.01			<0.001	<0.001
Anthracene	0.01			<0.001	<0.001
Pyrene	0.01			<0.001	<0.001
Benzathracene/Chrysene	0.01			<0.001	<0.001
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.001	<0.001
Benzo-a-pyrene	0.01			<0.001	<0.001
IPDA	0.01			<0.001	<0.001
Benzo (g,h,l) perylene	0.01			<0.001	<0.001
Total PAH	0.1	50	1000	<0.01	<0.01

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 9 – Tank and NRA leach test results of CBM produced during field trials – trial no. 4. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.05	<0.05
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	177	35
Sulphate	0.1	0.2%	0.2%	<5	<5
Phenols	0.01	5	5	0.12	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.2	11.8
PAH:					
Napthalene	0.01			<0.001	<0.001
Acenaphthylene	0.01			<0.001	<0.001
Acenaphthene	0.01			<0.001	<0.001
Fluorene	0.01			<0.001	<0.001
Phenanthrene	0.01			<0.001	<0.001
Anthracene	0.01			<0.001	<0.001
Pyrene	0.01			<0.001	<0.001
Benzathracene/Chrysene	0.01			<0.001	<0.001
Benzo(b)/Benzo(k) fluoroanthene	0.01			<0.001	<0.001
Benzo-a-pyrene	0.01			<0.001	<0.001
IPDA	0.01			<0.001	<0.001
Benzo (g,h,i) perylene	0.01			<0.001	<0.001
Total PAH	0.01	50	1000	<0.01	<0.01

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 10 – Tank and NRA leach test results of CBM produced during field trials – trial no. 5. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.05	<0.05
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	284	213
Sulphate	0.1	0.2%	0.2%	<5	<5
Phenols	0.01	5	5	0.12	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	11.8
PAH:					
Napthalene	0.01			<0.001	<0.001
Acenaphthylene	0.01			<0.001	<0.001
Acenaphthene	0.01			<0.001	<0.001
Fluorene	0.01			<0.001	<0.001
Phenanthrene	0.01			<0.001	<0.001
Anthracene	0.01			<0.001	<0.001
Pyrene	0.01			<0.001	<0.001
Benzathracene/Chrysene	0.01			<0.001	<0.001
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.001	<0.001
Benzo-a-pyrene	0.01			<0.001	<0.001
IPDA	0.01			<0.001	<0.001
Benzo (g,h,l) perylene	0.01			<0.001	<0.001
Total PAH	0.01	50	1000	<0.01	<0.01

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 11 – Tank and NRA leach test results of CBM produced during field trials – trial no. 6. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	0.01	0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.05	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	89	47
Sulphate	0.1	0.2%	0.2%	<5	19
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.3	11.8
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluoroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,i) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 12 – Tank and NRA leach test results of CBM produced during field trials – trial no. 7. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	0.01	0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	93	54
Sulphate	0.1	0.2%	0.2%	<5	<5
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<5	10
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.2	11.6
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	0.03
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 13 – Tank and NRA leach test results of CBM produced during field trials – trial no. 8. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	0.01	0.01
Cadmium	0.005	3	15	<0.0005	<0.0005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	127	87
Sulphate	0.1	0.2%	0.2%	<5	18
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.4	11.8
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 14 – Tank and NRA leach test results of CBM produced during field trials – trial no. 9. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	173	92
Sulphate	0.1	0.2%	0.2%	<5	6
Phenols	0.01	5	5	0.12	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	0.25	0.31
pH		<5	<5	12.3	12.1
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluoroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,i) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 15 – Tank and NRA leach test results of CBM produced during field trials – trial no. 10. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	77	82
Sulphate	0.1	0.2%	0.2%	<5	<5
Phenols	0.01	5	5	0.12	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	11.9
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 16 – Tank and NRA leach test results of CBM produced during field trials – trial no. 11. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	496	567
Sulphate	0.1	0.2%	0.2%	<5	5
Phenols	0.01	5	5	0.12	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	12
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenapthylene	0.01			<0.01	<0.01
Acenapthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 17 – Tank and NRA leach test results of CBM produced during field trials – trial no. 12. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	218	215
Sulphate	0.1	0.2%	0.2%	<5	16
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	11.9
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 18 – Tank and NRA leach test results of CBM produced during field trials – trial no. 13. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	496	922
Sulphate	0.1	0.2%	0.2%	5	35
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	12
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluoroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,i) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 19 – Tank and NRA leach test results of CBM produced during field trials – trial no. 14. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	142	91
Sulphate	0.1	0.2%	0.2%	<5	9
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	11.7
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 20 – Tank and NRA leach test results of CBM produced during field trials – trial no. 15. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	55	26
Sulphate	0.1	0.2%	0.2%	<5	9
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12.1	11.4
PAH:					
Napthalene	0.01			<0.01	<0.01
Acenaphthylene	0.01			<0.01	<0.01
Acenaphthene	0.01			<0.01	<0.01
Fluorene	0.01			<0.01	<0.01
Phenanthrene	0.01			<0.01	<0.01
Anthracene	0.01			<0.01	<0.01
Pyrene	0.01			<0.01	<0.01
Benzathracene/Chrysene	0.01			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.01			<0.01	<0.01
Benzo-a-pyrene	0.01			<0.01	<0.01
IPDA	0.01			<0.01	<0.01
Benzo (g,h,l) perylene	0.01			<0.01	<0.01
Total PAH	0.01	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

Table 21 – Tank and NRA leach test results of CBM produced during field trials – trial no. 16. Samples cured for 28 days at room temperature prior to leach testing.

Chemical species	Reporting Limit* (mg/kg)	ICRCL list Cat. D** (mg/kg)	ICRCL list Cat. P*** (mg/kg)	NRA test results (mg/l)	TANK test results (mg/l)
Arsenic	0.001	10	40	<0.001	<0.001
Boron		3	3	<0.01	<0.01
Cadmium	0.005	3	15	<0.005	<0.005
Lead	0.05	500	2000	<0.025	<0.025
Mercury	0.0001	1	20	<0.0001	<0.0001
Copper	0.01	130	130	<0.01	<0.01
Nickel	0.01	70	70	<0.01	<0.01
Zinc	0.01	300	300	<0.01	<0.01
Chromium (VI)	0.01	25	-	<0.01	<0.01
Selenium	0.0001	-	-	<0.0001	<0.0001
Chloride		-	-	104	60
Sulphate	0.1	0.2%	0.2%	<5	7
Phenols	0.01	5	5	<0.03	<0.03
Sulphide	0.1	-	-	<0.1	<0.1
Cyanide (total)	0.05	250	250	<0.05	<0.05
pH		<5	<5	12	11.7
PAH:					
Napthalene	0.001			<0.01	<0.01
Acenaphthylene	0.001			<0.01	<0.01
Acenaphthene	0.001			<0.01	<0.01
Fluorene	0.001			<0.01	<0.01
Phenanthrene	0.001			<0.01	<0.01
Anthracene	0.001			<0.01	<0.01
Pyrene	0.001			<0.01	<0.01
Benzathracene/Chrysene	0.001			<0.01	<0.01
Benzo(b)/Benzo(k) fluroanthene	0.001			<0.01	<0.01
Benzo-a-pyrene	0.001			<0.01	<0.01
IPDA	0.001			<0.01	<0.01
Benzo (g,h,l) perylene	0.001			<0.01	<0.01
Total PAH	0.001	50	1000	<0.1	<0.1

*Reporting limit - The detection limit of the analytical instrument

** ICRCL cat. D – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for gardens

** ICRCL cat. P – Inter-departmental Committee for the Redevelopment of Contaminated Land limits for parkland

3.7 Testing and monitoring of trial products

The effects of longer-term weathering of CBM produced during the field trials were assessed by laying down the CBM adjacent to the side wall liner within the landfill. The material was compacted to form a test pad of dimension 10 m × 6 m at a thickness of 1.5 m. The strength variation observed with the cube samples would also apply to the test pad but there were no localised areas of weaknesses observed across the compacted surface.

The performance of the test pad was monitored by removing CBM cores for compressive strength measurements after 7 and 28 days of curing; data shown in Table 22. This data supported the observations of a Consultant Structural and Civil Engineer (personal communications with Ramsey and Chalmers, Consultant Structural Engineering Consultants) whose opinion after a visual inspection of the test pad was that the material had sufficient engineering properties in terms of hardness and durability for its intended application. Variation in strength observed with the test pad, over time, are consistent with compressive strength data obtained for the cube samples made immediately after processing each batch of oily waste during the field trials. Thus, strength development in the CBM appears to be unaffected by natural weathering within the first 28 days of curing. Longer-term monitoring of compressive strength unfortunately could not be conducted due to lack of fund. Similarly, leachate and surface run-off from the test pad was also not monitored due to funding limitations.

Table 22 – Compressive strength data for test pad

7 days	28 days
1.2	3.5
1.5	4.5
1.5	4.5
2.0	2.5
2.5	4.0

5.8 The way forward

The results and observations made during the large-scale trials highlighted many issues which will have to be taken in to consideration when looking at commercial-scale production:

- ♦ The variability of the waste – the waste which arrives onshore varies from oil and water based drill cuttings to oily slops which include tank bottom sludges to wash-

down waters. The lack of homogeneity in terms of both the chemical composition and consistence makes it very difficult to formulate a single cement-based mix design to stabilise and solidify all offshore oily waste.

- ◆ The hydrocarbon content of the waste has a significant effect upon the hydration reactions of cement – hydrocarbons coat un-reacted clinker grains thereby preventing water from coming into contact with the grains to commence hydration. Consequently offshore wastes with high levels of hydrocarbons cannot be effectively stabilised and solidify with Portland cement-based binders to yield a product with appreciable structural integrity.
- ◆ The discrepancy in performance results between bench and field scale trials – the scale of processing differ significantly at bench and field scale; average weight of mix design at bench-scale is approximately 1 kg as opposed to batches of 20 tonne at field scale. Consequently most operational issues such as accuracy of delivery, liquid/solid ratio etc which can be controlled at bench-scale, can not be met when processing at field-scale.

The conclusions drawn from the first phase and the various operational constraints encountered during the field-scale trials, show that to successfully incorporate drilling waste in cement-based products, the waste will have to be pre-treated prior to solidification/stabilisation with cement. Pre-treatment will have to include several stages in order to:

- ◆ minimise variability in performance between different batches – a 'mixing' stage to blend the different batches will increase homogeneity;
- ◆ reduce the fraction of soluble chloride which leaches out of the final product – a mechanism whereby the soluble chlorides are 'washed out' of the bulk waste will significantly reduce chloride leachability. One such technology is 'Water Shearing' where the oily waste is passed through a high pressure water jet;
- ◆ remove the oily and aqueous fractions from the bulk waste – separation of the oily fraction will improve the setting of cement leading to improved compressive strength development over time.

With the conclusion of Phase I, additional work can be undertaken to improve the results of Phase I through a series of consecutive complimentary phases as summarised below.

1. Phase II – optimisation of present formulation at field-scale

Installation of a specialised flow meter to quantify delivery of feedstock to mixing chamber of the batching plant. Consecutive field trialling to:

- ◆ Assess the consistency in the quality of CBM produced in terms of compressive strength and contaminant leachability.
- ◆ Evaluate the optimal amount of oily waste which can be incorporated into the blend to produce CBM with the desired physical and chemical properties.

2. Phase III – the incorporation of treated drill cuttings residue into CBM

Formulation of CBM with improved physical and chemical properties through the addition of thermal de-sorption process residue to the present blend of unprocessed oily waste, binder, and aggregate. Treated drill cuttings residue which is currently classified as inert waste due to its low contaminant content, could be used as a filler material to improve the physical properties of the CBM. The pozzolanic nature of the thermal residue (inferred from previous work) is anticipated to enhanced contaminant immobilisation in the solidified/stabilised product.

The mix designs will be initially formulated in the laboratory and subsequently trialled at field-scale to assess consistency of quality and the commercial feasibility of the process. Longer term monitoring of CBM will be conducted to evaluate strength development and contaminant leachability under 'real' conditions.

3. Phase IV - the incorporation of drill cuttings sourced from historic drill cuttings piles found on the sea bed

The work in Phase III will be repeated during this phase with the incorporation of cuttings from historic cuttings piles to assess the impact on physical and chemical properties of the CBM.

In terms of the chemical composition and physical nature, cuttings in historic piles significantly differ from 'fresh' cuttings presently brought ashore. The successful incorporation of this material in CBM could provide a potentially viable commercial outlet when considering the various disposal options for the large amounts of cuttings lying in the bottom of the North Sea.

Formulation of CBM during Phase IV will be limited to laboratory scale trials because of the difficulties in obtaining large quantities of historic drill cuttings for field-scale trials.

4. Phase V – the incorporation of shredded tyres in CBM and mass concrete

Formulation of CBM and mass concrete with the incorporation of shredded tyres to add a degree of internal flexibility which makes the products suitable as pads over soft ground - for example, on compacted waste in landfills. The flexibility of the material would prevent cracking due to vehicle movement, thereby avoiding the need for replacement on a regular basis. The work would involve both laboratory and field trials to assess quality and commercial feasibility. Monitoring of products with time will be conducted to assess durability and performance under weathering.

(6)

Conclusions

- ◆ Compressive strength varies significantly for the different batches produced during the large-scale field trials. This could be attributed to the lack of homogeneity in the waste; the solid/liquid ratio varied significantly from one batch to another and consequently it was very difficult to maintain delivery of accurate quantities of waste during processing.
- ◆ Strength development in the treated products can be improved by removing the aqueous and oily components from the untreated bulk waste prior to treatment. Hydrocarbons impede the hydration reactions of Portland cement-based binders leading to poor strength development.
- ◆ Leachability from the solidified/stabilised products remains below the regulatory guidelines for all metals and polycyclic aromatic hydrocarbons (PAHs).
- ◆ The leach test data indicates that the CBM produced could be used for low grade civil engineering applications as contamination was low with respect to heavy metals and polycyclic aromatic hydrocarbons (PAH).
- ◆ The concentration of leachable chloride in CBM was above acceptable regulatory guidelines. It is anticipated that pre-treatment of the oily waste, for example by 'washing' the waste using a 'water shearing' technique, will remove soluble chloride.
- ◆ The main objectives of phase 1 were to produce CBM with compressive strengths between 1-5 N/mm², with minimal operational control, and contaminant leachability below the ICRL trigger concentrations, were achieved.
- ◆ Additional objectives of Phase 1 which were to establish the consistency of compressive strength and contaminant leachability between different batches were achieved. This information has highlighted operational constraints which will be encountered at commercial-scale production.

Appendix 1

Legal and regulatory perspective on drilling waste and its reuse

1.1 Classification of waste

The management of drilling waste on offshore platforms and supply vessels which transport the waste ashore and constraints related to the effectiveness of such management, means that the different types of wastes are often 'mixed' by the time they arrive at the quayside for treatment and/or disposal. From the time that this material leaves the offshore rigs and is transported by vessels to the onshore processors it is accepted as legal 'waste' and therefore subject to EU waste regulation.

The definition of 'waste' arises as a combination of the following legislation:

- ◆ the EC Waste Framework Directive (Council Directive 75/442/EEC, as amended);
- ◆ the Environmental Protection Act 1990 section 75 (as amended by the Waste Management Licensing Regulations 1994 and the Environmental Act 1995 section 120); and
- ◆ recent case decisions of the Court of Appeal and the European Court of Justice.

Although the legal sources are in transition, effectively any material, substance, product or article is waste if the producer of it, or the person in possession of it, discards it or intends to discard it, or is required to discard it. When assessing if something is waste, consideration therefore has to be given to actions, intentions, and/or requirements.

The various EU Waste Framework Directives have not yet been fully transposed into UK law, especially with regards to 'hazardous' or 'special' waste; at present UK law is in a state of flux and transition with regards to these issues but it is anticipated to change in the very near future.

Under the EU Waste Framework Directive waste is categorised into:

- ◆ hazardous;
- ◆ non-hazardous; and
- ◆ inert.

Hazardous waste is defined as any waste which is covered by Article 1(4) of Council Directive 91/689/EEC of 12 December 1991 on hazardous waste and comprises waste which is featured in the European Waste Catalogue (EWC), provided, in each case that the relevant waste has one or more

of the properties listed in Annex III to the Directive. The EWC is a composite list integrating the general list of wastes with that of hazardous waste. Excerpts from this list as far as relevant to drilling waste are shown in Table 23 and the Annex III hazard properties, any one or more of which the wastes may display, are listed in Table 24.

Any material which is defined as 'waste', and does not possess any properties which render it 'hazardous' or 'special', is technically classed as 'non-hazardous' waste. Thus all waste included in the EWC with the exception of those considered 'hazardous', comprise the non-hazardous category.

'Inert' is a sub-category within the 'non-hazardous' group and is defined as waste which does not undergo any significant physical, chemical or biological transformations. Inert waste is required not to dissolve, burn or otherwise physically or chemically react, biodegrade or adversely affect other matter with which it comes into contact in a way likely to give rise to environmental pollution, or cause harm to human health. Additionally, the total leachability and pollutant content of the waste and the ecotoxicity of the leachate must be insignificant, and in particular not endanger the quality of surface water and/or groundwater.

As UK law stands at present, 'hazardous' waste is referred to as 'special' waste. Current review of UK law is however, most likely to result in the term 'special' being dropped in favour of 'hazardous', in keeping with the EU terminology. This will also be consistent with the language used in the new Landfill Directive.

The nature and inconsistency of offshore drilling waste makes the classification of this material, once transported to shore, complex and ambiguous. This task is not assisted by the transitional state of UK Waste Law. Presently, unprocessed oily drilling waste is categorised as 'hazardous' or 'special' and therefore subject to Special Waste Regulations and to the hazardous waste provisions of the Landfill Regulations. However, depending upon the water content of the waste, it may also be categorised as liquid waste and therefore prohibited from land disposal in line with the requirements of the Landfill Directive; liquid waste is defined as:

- ◆ any waste that near instantaneously flows into an indentation void made in the surface of the waste;
- ◆ any waste (load) containing free draining liquid substances in excess of 250 litres or 10%, whichever represents the lesser amount.

Despite these clear definitions, the task of categorising unprocessed drilling waste as 'hazardous' or 'liquid' is not always clear-cut and is often left to the discretion of the Regulator. The matter is even more complex when dealing with processed drill cuttings; until recently the oil industry and certain onshore processors and landfill operators have considered the 'waste' element to be removed from unprocessed drill cuttings at the point of thermal treatment at onshore processing facilities. Thus the powdery residue from thermal treatment plants have been dispatched in bags to landfill, where they are used by landfill operators to assist in lining landfills and treated as non-waste 'product' in this

context. The liquid fraction of the process – the distilled recovered base oil has also been transported for use as a fuel in power stations or recycled in the offshore industry for further use (or re-use) in the drilling process, again as a non-waste 'product'.

In line with the recently introduced Special Waste Regulations within the UK and the strict judgements on the legal definition of waste by the European Court of Justice, treated drill cuttings are now considered 'special' waste by SEPA. This definition has caused widespread debate as to its validity, especially as this comes under the realms of 'border-line' cases, i.e., instances where the relevant articles are capable of economic re-utilisation, which remain a highly contentious area of EU and UK law. The position adopted by SEPA with regards to thermally treated cuttings appears to be based on not meeting the EU pollution avoidance objectives and the relevance of one of the overriding principles identified by the European Court in defining waste. These observations appear to be founded on evidence of residual hazardous contaminants and the potential for contaminant leachability.

The implications of being classed as 'special' or 'hazardous' is that most landfills which are currently used by the North Sea oil industry for disposing of processed and unprocessed cuttings may not be licensed to accept these materials under the New Landfill Directive. This will be reflected in the cost of disposal which is anticipated to increase significantly. The cost of disposing 'liquid' waste which includes certain unprocessed cuttings, waste oils and various fluids is also anticipated to rise sharply with changes to the Landfill Directive, encouraging the development of novel recycle/reuse options.

Table 23 – EU law/imminent UK law – list of wastes considered to possess hazardous properties (reproduced from the EU Hazardous Waste Directive)

European Waste Catalogue – Oil related wastes

- ◆ Oil-containing drilling muds and wastes resulting from exploration of minerals;
- ◆ Drilling muds and other drilling wastes, containing “dangerous substances” [see below], which are wastes resulting from exploration of minerals;
- ◆ Certain wastes from waste management facilities which carry out physical/chemical treatment of waste, including oil and concentrates from separation, and liquid and/or solid combustible wastes containing “dangerous substances”; and
- ◆ In so far only as not covered by any of the above three categories, there is a general sweep up provision listing “oil wastes not otherwise specified”

Dangerous substances

- ◆ Explosives: substances and preparations which may explode under the effect of flame or which are more sensitive to shocks or friction than dinitrobenzene;
- ◆ Oxidising: substances and preparations which give rise to highly exothermic reactions when in contact with other substances, particularly flammable substances;
- ◆ Easily flammable:
 - o substances and preparations which may become hot and finally catch fire in contact with air at ambient temperature without any application of energy;
 - o substances and preparations which may become hot and finally catch fire after brief contact with a source of ignition and which continues to burn or to be consumed after removal of the source of ignition; or
 - o liquid substances and preparations having a flash point below 21°C, or
 - o gaseous substances and preparations which are flammable in air at normal preparation; or
 - o substances and preparations which, in contact with water or damp air, evolve highly flammable gases in dangerous quantities;

- ◆ Toxic: substances and preparations which, if they are inhaled or taken internally or if they penetrate the skin, may involve serious, acute or chronic health risks and even death;
- ◆ Harmful: substances and preparations which, if they are inhaled or taken internally or if they penetrate the skin, may involve limited health risks;
- ◆ Corrosive: substances and preparations which may, on contact with living tissues, destroy them; and
- ◆ Irritant: non-corrosive subjects and preparations which, through immediate, prolonged or repeated contact with the skin or mucous membrane, can cause inflammation.

Table 24 – Hazardous Directive Annex III – Properties of waste which render them hazardous

H1 'Explosive': substances and preparations which may explode under the effect of flame or which are more sensitive to shock or friction than dinitrobenzene.

H2 'Oxidising': substances and preparations which exhibit highly exothermic reactions when contact with other substances.

H3-A 'Highly Flammable': liquid substances and preparations which may become hot and finally catch fire in contact with air at ambient temperature without any application of energy, or – solid substances and preparations which may readily catch fire after brief contact with a source of ignition and which continue to burn or to be consumed after removal of the source of ignition, or – gaseous substances and preparations which are flammable in air at normal pressure, or – substances and preparations which, in contact with water or damp air, evolve highly flammable gases in dangerous quantities.

H3-B 'Flammable': liquid substances and preparations having a flash point equal or greater than 21°C and less than or equal to 55°C.

H4 'Irritant': Non-corrosive substances and preparations which, through immediate, prolonged or repeated contact with the skin or mucous membrane, can cause inflammation.

H5 'Harmful': Substances and preparations which, if they are inhaled or ingested or if they penetrate the skin, may involve limited health risks.

H6 'Toxic': Substances and preparations (including very toxic substances and preparations) which, if they are inhaled or ingested or if they penetrate the skin, may involve serious, acute or chronic health risks and even death.

H7 'Carcinogenic': Substances and preparations (including very toxic substances and preparations) which, if they are inhaled or ingested or if they penetrate the skin, may induce cancer or increase its incidence.

H8 'Corrosive': Substances and preparations which may destroy living tissue on contact.

H9 'Infectious': substances containing viable micro-organisms or their toxins which are known or reliably believed to cause disease in man or other living organisms.

H10 'Teratogenic': Substances and preparations which, if they are inhaled or ingested or if they penetrate the skin, may induce non-hereditary congenital malformations or increase their incidence.

H11 'Mutagenic': Substances and preparations which, if they are inhaled or ingested or if they penetrate the skin, may induce hereditary genetic defects or increase their incidence.

H12 Substances and preparations which release toxic or very toxic gases in contact with water, air, or an acid.

H13 Substances and preparations capable by any means, after disposal, of yielding another substance, e.g., a leachate, which possesses any of the characteristics listed above.

H14 'Ecotoxic': Substances and preparations which present immediate or delayed risks for one or more sectors of the environment.

1.2 Legislation issues related to the re-use of drilling waste

Changes to legislation have acted as the main driver for the increasing research into exploring potential recycle/reuse options for offshore drilling waste. In light of this, waste legislation and guidance relevant to the UKCS has been briefly reviewed below.

1.2.1 Landfill Directive

The EU Council Directive pertaining to the land-filling of waste, the Landfill Directive (1999/31/EEC) implemented in 1999, has been transposed into law in England and Wales via the Landfill Regulations 2001. Scottish regulations have not emerged as yet but the Scottish Executive has introduced a transitional and provisional implementation Direction. This does not differ materially from that introduced in England and Wales as harmonisation of the law is one of the key objectives of the new Directive.

Landfills are currently regulated in England and Wales by the Environment Agency (EA) and in Scotland by SEPA through either waste management licensing under the Environmental Protection

Act 1990 (EPA 1990) or the Pollution Prevention and Control (PPC) Regulations 2000. The Landfill Regulations 2001 incorporate the additional requirements of the Landfill Directive and is intended to supplement and amend the PPC Regulations 2000 to meet the wider objectives of the Directive.

The Landfill Directive is primarily aimed at reducing the amount of biodegradable waste being disposed of in landfill. However, it will have a significant impact upon non-municipal waste designated as 'special' waste and 'liquid' waste due to:

- ◆ co-disposal of waste being prohibited – all landfill sites will have to be categorised as hazardous, non-hazardous or inert by 2004;
- ◆ landfill disposal of liquid, clinical and certain other hazardous wastes including tyres, being banned; and
- ◆ the requirement to pre-treat 'special' waste prior to landfill.

The impact of the new Directive upon drilling waste, whether processed or otherwise is significant; both processed and unprocessed drilling waste may be classed as 'special' or hazardous' depending on the level of leachable contaminants, pH, hydrocarbon content etc. Consequently, the waste requires disposal in hazardous landfills with pre-treatment prior to disposal. In the event of being classed as liquid, which is the case for certain offshore wastes with high water contents, land disposal has not been an available option since the regulations were implemented on the 16th of July 2001. Furthermore, the stringent waste acceptance criteria introduced by the new Directive has significant practical implications for the oil industry 'waste chain', a topic discussed in detail in section 2.2.6. Indeed, the Landfill Directive is viewed by many in the offshore industry as playing a crucial role in shifting the trend in onshore management of drilling waste from land disposal to recycle and reuse.

1.2.1.1 Pre-Treatment

A requirement of the Landfill Directive with significant economic consequences across the waste management industry is the requirement to pre-treat hazardous waste prior to land disposal. The main focus of pre-treatment is to reduce the negative impact of the land filling of waste on the environment and human health. To meet the overall objectives of the new Directive, the Scottish Executive views pre-treatment as fulfilling three criteria⁰:

- ◆ It must be a physical/thermal/chemical or biological process;
- ◆ It must change the characteristics of the waste; and
- ◆ It must do so in order to:
 - Reduce its volume; or

- Reduce its hazard nature; or
- Facilitate its handling; or
- Enhance its recovery.

Thus the change in characteristics of the waste must come about as a result of the 'process' discussed in the first bullet point, and be for the purpose of achieving one of the four objectives set out in the third bullet point. The requirement for pre-treatment does not allow the mixing and dilution of waste solely to meet the waste acceptance criteria during land disposal. However, without this provision, if the waste acceptance criteria being developed were expressed in terms of concentration of contaminants, it is possible that a waste could be diluted so as to eventually render it 'acceptable' for landfill. An example of such a treatment would be the mixing of an acid waste with alkali waste to produce a pH-balanced waste such that the 'offending' acidic or alkaline properties have been reduced. The critical word is 'solely' – so the treatment above would not be the only pre-treatment necessary.

1.2.1.2 Waste acceptance criteria

The Landfill Directive stipulates stringent monitoring of waste acceptance criteria for all landfills as set out in Article II of the Directive. It is intended that an amended Duty of Care regime which will include reference to the European Waste Catalogue (EWC) when classifying the waste, will assist the overall process.

Monitoring of waste acceptance criteria are implemented through a three level hierarchy which would be applied to all classes of landfill:

- ◆ Level 1 - Basic characterisation (a thorough determination of the leaching and/or characteristic properties of the waste);
- ◆ Level 2 - Compliance testing (this is periodic checking to ensure that the waste has not changed since characterisation at Level 1);
- ◆ Level 3 - On-site verification (this is rapid checking at the landfill site).

Basic characterisation of the waste must be carried out by or on behalf of the waste holder before land disposal to provide information on:

- ◆ Source and origin of waste;
- ◆ Information on the process producing the waste (description and characteristics of raw materials and products;

- ◆ Data on the composition of the waste and the leaching behaviour, where relevant;
- ◆ Appearance of the waste (smell, colour, physical form etc);
- ◆ Information to provide the EWC code;
- ◆ For hazardous waste: hazardous properties according to Annex III Directive 91/689.

A waste which has been deemed acceptable for disposal in a specific class of landfill following basic characterisation is subsequently subject to compliance testing, prior to the delivery of the waste to the landfill site. Compliance testing is recommended to be conducted at least once a year for each individual waste stream to determine if the waste complies with the results of the basic characterisation, the acceptance criteria for the landfill class and the permit requirements for the specific landfill. The relevant parameters to be checked should generally be determined by the landfill operator on the basis of the results of the basic characterisation. The selection of parameters, including the reason for the selection, should be documented and duly justified. On-site verification which is the final step of monitoring, involves the visual inspection of the waste at the landfill site before and after unloading for disposal.

The stringent requirements of the new Directive would undoubtedly raise the overall cost of land disposal for oily drilling waste while continued land-filling will contradict the ultimate objective of the National Waste Strategy - to reduce waste going into landfill. The stringent waste acceptance criteria would pose a significant burden across the whole of the offshore industry; a pertinent issue facing the industry is the inconsistency of the waste brought ashore which makes the task of identifying the nature and source of the individual waste streams at the quayside practically impossible. In addition to these obvious impacts, concerns also exist over available landfill capacity for hazardous waste due to classification of sites. The categorisation of unprocessed drilling waste as 'liquid' wastes in certain instances, have further exacerbated the issue of onshore waste management.

1.2.2 National Waste Strategies

The National Waste Strategy for Scotland (NWS) was launched in February 2003 and provides a framework for reducing waste sent to landfill and, for increasing recovery and recycling. The waste strategies for England and Wales have similar objectives and are aimed to move the UK towards more sustainable waste management practices.

The NWS (Scotland) is implemented through eleven Area Waste Plans which have been set up across Scotland to produce a sustainable and integrated waste management approach for Scotland as a whole by taking into account local geographical, economic and social variations. It is anticipated that the Scottish Executive and SEPA will work with the producers of waste to develop and implement waste management arrangements in accordance with the Best Practicable Environmental Option (BPEO) for the individual wastes.

Central to the NWS (Scotland) is the concept of the waste hierarchy that sets out the preferences for different treatment options for wastes – Figure 14. The hierarchy provides a theoretical framework which should be used as a guide for ranking the waste management options being considered as part of the BPEO assessment. It offers an order which can be used when considering various waste management options, starting with a review of how less waste may be produced. Once this has been conducted, all options in the hierarchy should be considered for each component material within the waste stream. As different options are likely to be favourable for different waste materials both environmentally and economically, the BPEO for a particular waste stream will generally consist of a mix of various waste management methods.

Oily drilling waste which falls under the category of non-municipal waste has been identified as a priority waste stream within the NWS (Scotland). Recycling and reuse of the material is anticipated to be promoted by examining current waste management practices and barriers to recycling/reuse. The need to develop codes and standards for recycling/reuse has also been highlighted to clarify ambiguities and to avoid disputes between the waste management industry and SEPA regarding the acceptability of a recycle/reuse process or a product. Additionally, oily waste has also been identified by SEPA as one of the most common categories of 'special' waste disposed to landfill within Scotland. Thus the NWS (Scotland) has effectively raised the profile of oily waste and the need for sustainable solutions for its disposal, with emphasis on recycle and reuse. This will undoubtedly encourage current industry wide efforts made to promote recycling and reuse through increased investment into research and development for alternatives to land disposal for both processed and unprocessed oily drilling waste.

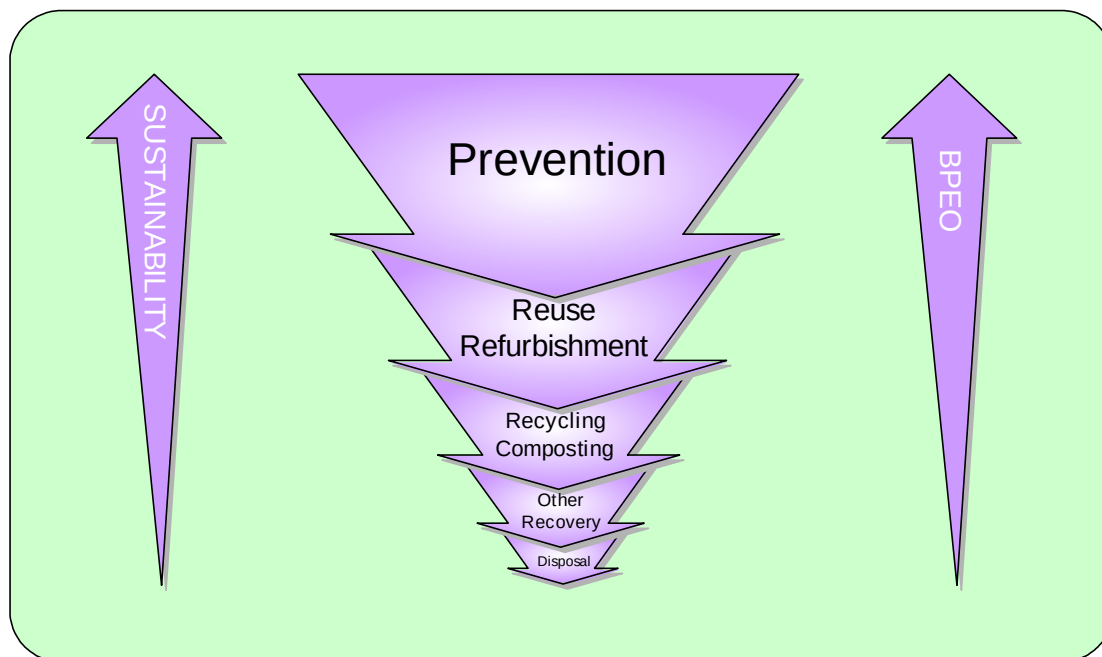


Figure 14 – The waste hierarchy as set out in the NWS (Scotland)

1.2.3 Landfill tax

The Landfill Tax Regulations were introduced in the UK in 1996 in an attempt to reduce the amount of waste disposed to landfill through increased cost of disposal. The regulations allow HM Customs & Excise to levy a tax (landfill tax) on the disposal of waste to licensed landfill sites (unless exempt). The tax is applied to the holder of the landfill site, who in turn applies the rate of tax as part of the landfill charge to those depositing the waste.

Landfill tax is charged on a tonnage basis and is currently set at two levels:

- ◆ inactive or inert waste - £2 per tonne (includes rocks, soil, ceramics, concrete, minerals, furnace slag and ash);
- ◆ standard rate - £13 per tonne (all other wastes unless exempt – includes dredgings from inland waterways and harbours, mining and quarry waste, pet cemeteries, contaminated land remediation waste, landfill site restoration materials and quarry fillings).

The standard rate is set to increase by £1/tonne each year until 2004, subject to Parliamentary approval. Untreated drill cuttings classified as special waste attracts a higher disposal fee to reflect the higher level of landfill engineering required for hazardous wastes as well as the higher rate of tax. Disposal costs for offshore oily waste are anticipated to continue to rise, due in part to increases in landfill tax, and in part to the increased costs associated with the requirements of the Landfill Directive. Consequently, the waste producers, in this case the oil operator companies, are under pressure to identify alternative means of disposal with strong emphasis on recycling/reuse.

1.2.4 Aggregate Tax

The 'Aggregate Tax' was introduced in the UK on the 1st of April 2002. The tax applies to primary aggregate such as sand, gravel and rock which are subject to commercial exploitation in the UK. It also includes aggregate dredged from the seabed within UK territorial waters (12-mile limit). The tax is levied at £1.60 per tonne and would have a significant impact upon the use of natural aggregate due to the rise in associated costs. Offshore cuttings are exempt from the tax. The implication of this is that construction products successfully made from oily waste, particularly drill cuttings, could potentially offer a competitive alternative to natural aggregate, driving the case for recycling and reuse further.

1.2.5 Criteria used for evaluating the success of recycling/reuse processes

Criteria used by the regulator for evaluating the authenticity of a recycling/reuse process or product have significant impacts upon the drive towards removing oily drilling waste from the waste stream. In the case of the North Sea oil industry such criteria are exercised by SEPA within the framework of the NWS to distinguish between genuine recovery and 'sham' recovery. Sham recovery refers to a

waste management practice in which a process operator seeks to avoid disposal costs by 'recycling' waste inappropriately.

The European Court of Justice states that a by-product or residual product from a process does not constitute 'waste' if it is destined for direct reuse in a further process in its present form, and if the use of a residue as substitute or ingredient is as environmentally sound as the material that it is replacing. This provision thus applies in circumstances where the by-product or residue has an economic value as product and can be subject to other legislation applicable to those products. However, if these secondary products cannot be directly used as an integral part of a production process, i.e., requires to be processed further, it is considered waste and the process a 'sham' recovery.

As with the principles of EU case law, UK Waste Law does not consider production residues, secondary raw materials and by-products used in industrial processes to constitute waste, provided they are not subjected to further operations associated with the recovery of the waste. The UK Department of the Environment for instance, does not consider recyclable material as waste if it is:

- ◆ material which is within the 'commercial cycle or chain of utility';
- ◆ material which can be put into immediate use without going through a specialized waste recovery operation; or
- ◆ waste which has been processed to such an extent that it can be used as a raw material.

Re-utilization does however, remain a contentious area of EU and UK law not least due to the complexities of existing waste legislation but also due to difficulties in applying the precise definition of waste in such 'border-line' cases.

1.2.6 Duty of Care

The Duty of Care applies to anyone who imports, produces, carries, keeps, treats or disposes of waste or, as a broker, has control of it. Every individual or organization subject to Duty of Care must take all such measures as are reasonable in the circumstances to:

- ◆ Prevent contravention by any other person of the waste management provisions of the 1990 Act;
- ◆ Prevent the escape of the waste from his control or any other person;
- ◆ Ensure that the waste is transferred to an authorized person, such as the holder of the waste management license, a person operating under the terms of a licensing exemption registered with the Environment Agency or a registered waste carrier;

- ◆ Ensure that a written description is transferred with the waste.

Each individual or organization to whom the Duty of Care applies has a legal obligation to comply with it and failing to do so can result in severe penalties such as unlimited fines on conviction in Crown Court. Practical guidance on complying with the Duty of Care is provided in Waste Management, the Duty of Care, A Code of Practice.

Duty of Care liability plays a significant role when attempting to promote effective onshore waste management of offshore oily waste, especially with regards to the issue of land disposal. The main reason for this is that the wider issue of liability is rather ill-defined at present due to the lack of relevant Case Law. Consequently, there appears to be a discrepancy between the views of the onshore waste management industry, and the large oil operators as producers of the waste, who appear to be over-cautious due to sensitive corporate policy.

Appendix 2

Cement Solidification and Stabilisation – Description of the Technology

2.1 Introduction

Solidification/stabilisation (s/s) is rapidly becoming an acceptable environmental technology and its application for the treatment/conditioning of many waste streams is already well established around the world. A wide range of reagents are being used and trialled in s/s systems mostly ameliorate the physical characteristics of semi-solid and liquid wastes to enable them to be transported and disposed of to landfill. Emphasis is also increasingly being placed on using s/s technology to reduce the leachability of potentially hazardous constituents from waste/reagent composites through the use of pozzolanic materials such as cement, pulverised fuel ash (pfa), lime etc.

s/s is a combination of two processes, each defined as follows ⁽³⁾:

Solidification is the process of producing from liquid, sludge, or loose solids a more or less monolithic structure with structural integrity; it may also refer to the process that results in a soil-like material as opposed to a monolithic structure. Solidification does not necessarily reduce leaching of hazardous waste components. However, when a waste is solidified its mass and structure are altered, decreasing the migration of solutions within the mass.

Stabilisation refers to a purposeful chemical reaction that is carried out to render the waste constituents less leachable. This is accomplished by chemically immobilising hazardous components or reducing their solubility through chemical reactions.

To date the most common use of s/s globally is as a final treatment step for treating hazardous waste prior to land disposal. However, in the field of civil engineering cement-based s/s is used to yield a 'value added' product that can be used in various applications such as bulk fills in earthworks or as a foundation material for redevelopment (personal communications with TRL and BRE). Although s/s technology is routinely used in waste treatment outside of the UK, wide application within the UK has been handicapped due in part to the low cost of conventional landfill of hazardous waste, and in part to scepticism arising from a lack of knowledge about its many benefits. However, with increasingly stringent environmental legislation and greater environmental awareness, the use of the technology is becoming more widespread.

2.2 Cement-based solidification/stabilisation

'Traditional' cement-based s/s technology has utilised the hydration of Portland cement to chemically stabilise and physically encapsulate waste components. Portland cement (or simply "cement") which we know today is made by heating together limestone and clay (or some other source of silica) at about 1450°C to form 'clinker'. A small amount of gypsum (anhydrous CaSO_4) is added to the clinker and subsequently ground to a fine powder having a specific surface area of 3000 to 400 cm^2g^{-1} . The purpose of adding gypsum is to delay the setting of cement by 1 to 2 hours allowing time for the cement to be handled or worked.

The use of cement-based systems in the environmental field has a history which dates back about 30 years with roots of present-day commercial applications going back to four primary areas of technology:

- ◆ radioactive waste solidification and disposal;
- ◆ mine backfilling;
- ◆ soil stabilisation and grouting;
- ◆ production of stabilised base courses for road construction.

To date radioactive waste treatment remains a s/s process based on cement alone. The other three applications commonly use admixtures which include cement; cement or coal combustion fly ash or both are used in mine backfilling, sodium silicate plus setting agents, cement and organic polymerising systems for grouting and soil stabilisation, and lime/fly ash for road-base construction. Additionally, waste residue generators, especially waste disposal site operators have also used cement, fly ash, lime, soil and various combinations of these materials to solidify semi-solid material and liquids for disposal in landfills where some stabilisation was required in the fill material to improve 'handleability'.

Since its inception cement-based s/s has been dominated by engineering disciplines and as a consequence the technology has been viewed primarily as a physical containment process with emphasis on properties such compressive strength, permeability, and resistance to weathering. However, cements exhibit a number of chemical features which are responsible for its increasingly widespread use in waste treatment processes; hydration of cement requires water and thus cement-based systems readily incorporate wet wastes. The alkalinity of cement greatly reduces the solubility of many toxic inorganic constituents and inhibits microbiological processors. Moreover, cement can be used as an activator for other potentially cementitious materials, e.g., glassy slag or fly ash which are frequently used to modify the chemical composition and microstructure of cement matrices; slag and fly ash are characterised by having a mean particle size finer than cement thus leading to a more coherent and denser matrix in well cured pastes.

2.3 Hydration reactions of cement

The chemical reactions of cement hydration which are primarily responsible for the significant immobilisation potential shown by cements are complex. This is partly because cement clinker is poly-mineralic in nature and partly because the reaction kinetics are complex and the nature of the resulting products are ill defined ⁰. Figure 15 shows the generally accepted overall course of hydration kinetics. When mixing Portland cement with water, heat is evolved and the mix water becomes strongly alkaline. The reaction slows after a few minutes and the following period known as the induction period, or dormant period, normally lasts several hours. The macroscopic behaviour of the system can be correlated with the microstructural changes which occur within the matrix.

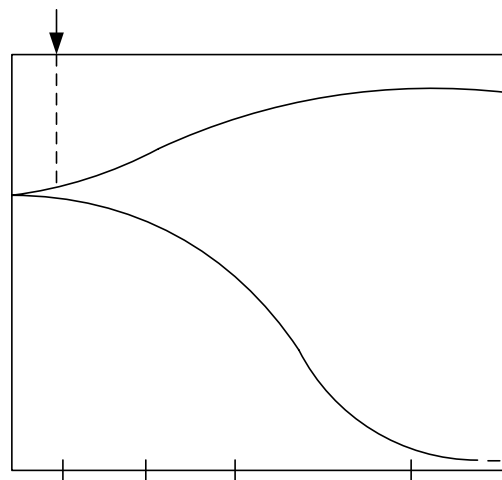


Figure 15 – Schematic showing the overall course of cement hydration. As the mix hardens, mix water is gradually transformed to pore water: this is indicated by the vertical dashed line.

During the first few minutes of reaction, the anhydrous clinker grains become coated with a nearly-amorphous precipitate which acts as a semi-protective film and slows reaction during the induction period. During this stage the mix remains relatively fluid. Towards the end of the induction period the breakdown of the film marks the onset of rapid hydration. This onset also initiates the development of a continuous but initially tenuous gel network linking particles, with the result that physical stiffening occurs. Thereafter, as the gel continues to stiffen and densify, strength gain commences. Figure 16 illustrates some of the processes involved in early hydration. Modern Portland cements typically achieve two-thirds of hydration in 28 days, although some anhydrous clinker remains even after a year.

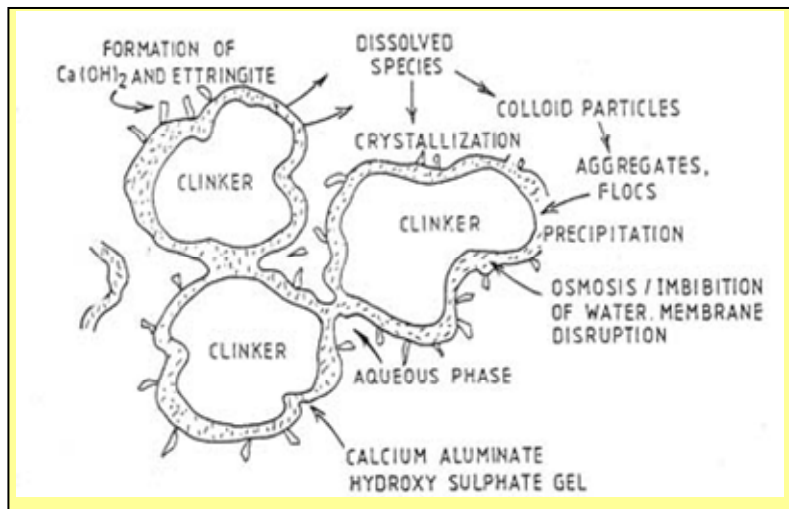


Figure 16 – Summary of hydration process occurring during the first few hours or days (taken from Glasser⁰)

The strength development in cements after setting is primarily due to tricalcium silicate (C_3S) and beta-dicalcium silicate ($\beta\text{-C}_2\text{S}$) both of which give the same reaction products calcium hydroxide (Ca(OH)_2) and calcium silicate hydrate gel (C-S-H). All of the reaction products with the exception of Ca(OH)_2 , have a low solubility in the lime-saturated medium of the cement paste compared to the anhydrous minerals in the original cement. The basic hydration reactions are listed in table 25. Tricalcium aluminate (C_3A) hydrates first, causing the rapid setting which produces a rigid structure. Setting rate is controlled by the amount of gypsum added in the cement manufacture. The ettringite which forms does not contribute to setting, but coats the clinker particles and retards the setting reactions. Hydration of C_2S and C_3S , which accounts for approximately 75% of the cement by weight, is responsible for strength development after the initial set.

Table 25 - Basic reactions of cement hydration

Reactants	Products	Heat Evolved (cal/g)
$\text{C}_3\text{A} + 6\text{H}$	C_3AH_6	207
$\text{C}_3\text{A} + 3\text{CS} + 32\text{H}$	$\text{C}_6\text{AS}_3\text{H}_{32}^*$	347
$2\text{C}_3\text{S} + 6\text{H}$	$\text{C}_3\text{S}_2\text{H}_3^{**} + 3\text{CH}$	120
$2\text{C}_2\text{S} + 4\text{H}$	$\text{C}_3\text{S}_2\text{H}_3^{**} + \text{CH}$	62
$\text{C} + \text{H}$	CH	279

*Ettringite **tobermorite gel

The products of hydration of Portland cement include both crystalline and amorphous phases. Calcium hydroxide, $\text{Ca}(\text{OH})_2$, one of the main crystalline phases (typically 20-25% by weight of cement) buffers the pH of a hydrating cement system at about 12.5. This relatively high pH plays a significant role in the chemical reduction in solubility of toxic metals. The gel phase of hydration (typically 60-70% by weight of cement), designated C-S-H (cement shorthand: $\text{C}=\text{CaO}$, $\text{S}=\text{SiO}_2$, $\text{H}=\text{H}_2\text{O}$), with a C/S ratio between 1.8-2.0 is the principal constituent of Portland cement and on account of its nano-porosity, it controls many of the sorptive properties associated with hydrated cements. The successful use of cements in s/s of waste components arises as a result of these chemical and physical mechanisms operating within cement matrices.

2.4 The interaction of cement and waste components – immobilisation potential

The successful use of cement to chemically stabilise a waste form while imparting structural integrity to the treated or conditioned end-product arises due to a combination of chemical and physical processes which take place within the cement matrix. These include:

- ◆ internal pH-induced precipitation;
- ◆ redox controlled precipitation;
- ◆ adsorption/occlusion into and onto C-S-H gel;
- ◆ crystallochemical incorporation within the cement matrix.

Several major factors affect the morphology and performance of cement-based waste forms. Fineness affects the rate of hydration, with finer grinds showing accelerated strength development during the first 7 days. Considerable heat is liberated by the exothermic hydration reactions which can help to speed-up the curing process. However, when dealing with large quantities of cement or cement/waste composites the limitations on heat dissemination may cause the heat developed to affect the final physical properties of the solid and may also drive off low molecular volatile organic compounds (VOCs). The rate at which heat is generated will be dictated by the cement composition, and also by certain waste constituents.

The water to cement ratio (w/c) plays an important role in cement hydration. The volume of the cement itself doubles upon hydration, creating a network of very small gel pores. The volume originally occupied by the added water forms a system of much larger capillary pores. As the w/c ratio increases the percentage of larger pores increase, significantly increasing the permeability of the solidified waste form. In a 'pure' cement system the permeability is essentially zero at a w/c ratio of 0.32, but increases exponentially as the w/c ratio reaches 0.6 to 0.7. Very high w/c ratio will leave "bleed water", or water that appears as standing water on the surface of the solid mass. This occurs at w/c ratio of 0.5 or greater. The addition of gelling agents such as sodium silicate and/or bulking agents such as flyash allow much higher w/c to be achieved without generation of "bleed water". They may however, affect the final physical and chemical properties of the waste form and they do

affect set time. The cementitious or pozzolanic reactions require sufficient “free” water if they are to go to completion and the water content of a waste as measured by total solids determination is not necessarily all available for reaction.

During cement-based s/s, the amount of lime produced in cement hydration is of importance. A typical cement composition will produce about 30% Ca(OH)_2 which corresponds to an acid neutralisation capacity of 8 meq/g of dry cement. This results in a pore solution of pH 12 to 13. The cementitious reactions which occur in these processors require the pH to be above 10 and this is initiated by the dissolution of free lime from the solid which continues throughout the setting and curing stages of the mixture. However, a “false set” can happen when enough free lime is present initially to start the reaction, but availability decreases as the process proceeds and the reactions stop or slow down. Thus any reaction that competes successfully for the calcium ion may inhibit setting.

The long-term durability of concrete and cement mortars is well documented and proven. Nevertheless, chemical and physical attacks due to environmental conditions do occur. The chemical attacks are defined as interfacial phenomena which take place through complex mechanisms which include ion exchange, dissolution of hydrated solids or formation of new insoluble compounds. Sulphates are the most destructive compounds normally present in the environment as they react with aluminates in the cement structure to form the expansive sulphotoaluminate, ettringite, as well as acids which would lower the pH within the matrix.

2.4.1 Internal pH-induced precipitation

It is widely accepted that cement-based waste forms rely heavily on pH control for metal containment. It is also thought to affect the leachability of some other inorganic and organic species. A large number of metal hydroxides have a minimum solubility in the range of pH 7.5 – 11 which makes a high pH within solidified and stabilised matrix desirable. However, all metal hydroxides do not reach minimum solubility at the same pH and thus the optimum pH of the system must be a compromise. Furthermore, hydroxides of some metals such as lead exhibit solubilities above the regulatory limit or target concentration. This is depicted by the solubility curves for various metal hydroxides in water as shown in Figure 17.

In contrast to the solubility of metal hydroxides, the solubilities of sulphides are much lower under alkaline conditions. This can be seen from Figure 18 which depicts the solubility of metal sulphides as a function of pH. Sulphides also do not exhibit increased solubilisation at high pH, at least not at the pH levels encountered in most cement-based s/s systems. The exception to the trend however, is chromium which does not form sulphides under alkaline conditions.

Figure 17 – Solubility of metal hydroxides as a function of pH (taken from Roger D. Spence, Chemistry and microstructure of solidified waste forms, Lewis publishers)

Although solubility can be used as a measure to predict leachability in s/s waste forms, it must be used with caution as many of these constants have been derived for individual species or for relatively simple combinations, in a specific solvent, which is often pure water. Moreover, waste forms contain other soluble species which may change the actual solubility of any metal due to effects such as common ion, complexation, total ionic strength and redox potential. Some metals such as Chromium exhibit different solubilities in its various valence states, and may exist in cationic or anionic form, or both in the same waste.

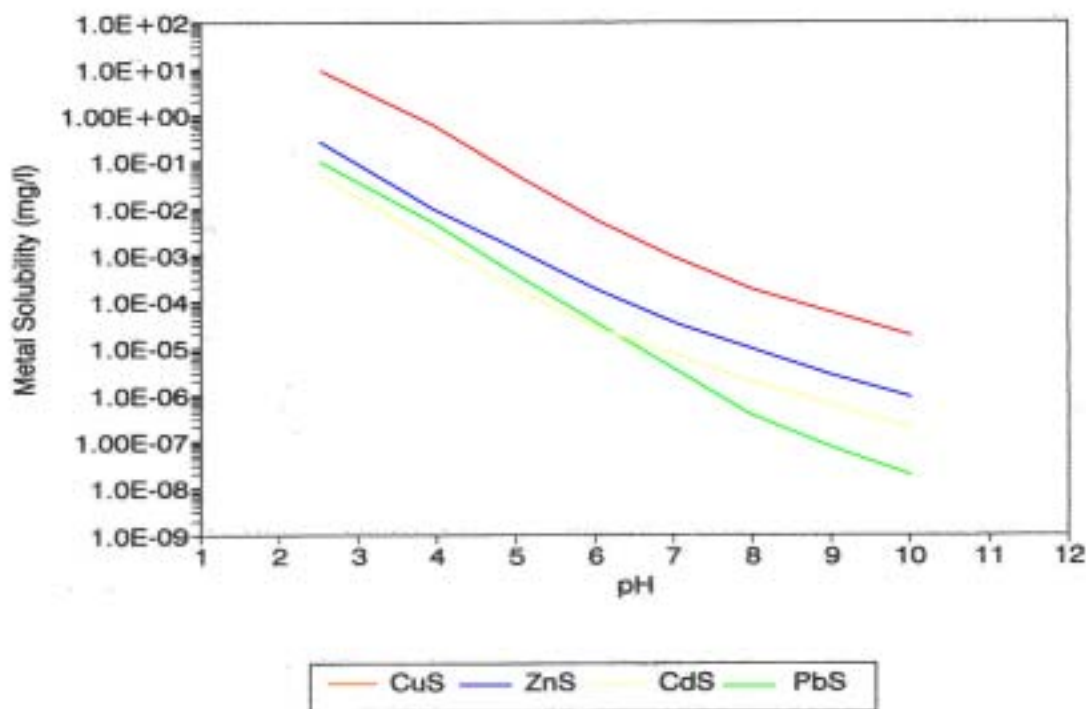


Figure 18 – Solubility of metal sulphides as a function of pH (taken from Roger D. Spence, Chemistry and microstructure of solidified waste forms, Lewis publishers)

2.4.2 Redox-controlled precipitation

The redox potential, E_h , is the oxidation-reduction potential (ORP) referred to the hydrogen scale, expressed in millivolts. It establishes the ratio of oxidants and reductants existing within a s/s system. The presence of strong oxidants and reductants can change the valence state of many metals, affecting their chemical speciation and, therefore, their mobility by orders of magnitude. For some metals such as arsenic, both valence and speciation as either cations or anions can change easily with redox potential. Other metals such as iron, manganese, chromium, mercury, nickel and selenium can also have more than one valence state in aqueous systems. Furthermore, nitrogen and sulphur have multiple valence states which affect the speciation of metals in a given system; copper, cadmium and zinc can be strongly influenced by redox processors despite having only one valence state in aqueous media.

Cements are normally produced under oxidising conditions and, the E_h of cement pore fluids typically give values in the range +100 to +200 mV. The main constituents which contribute to the electro-

activity within the pore fluid are believed to be oxygen dissolved in mix water and sulphite, SO_3^{2-} , formed by condensation of kiln vapour during the production of clinker. The cement solids may additionally contain small amounts of Fe^{2+} and Mn^{2+} but these remain insoluble at high pH. Thus cement on its own has an inherently weak electro-activity but the addition of small amounts of an electro-active material which can control the E_h , allows significant scope for tailoring the chemical properties of the matrix.

In practice, real waste can strongly condition the redox potential; the presence of organics and of reactive metals, including steel, is expected to lower the E_h . Widely used cement supplementary materials such as flyash and slag have different impacts on E_h . The most favourable conditions for immobilising toxic waste is a low E_h , so that multivalent metals, typically present as anionic species when in high state of oxidation, can be reduced in high pH environments to lower-valent, less-soluble cationic species. Although the persistence of reducing conditions within a s/s matrix can not be guaranteed, many saturated disposal environments will have a characteristically low E_h .

2.4.3 Adsorption/occlusion into and onto C-S-H gel

The sorption properties within a hydrating cement matrix can be predominantly attributed to the gel phase of hydration, C-S-H, which has a high specific surface area measured by N_2 adsorption at 10 to $50 \text{ m}^2/\text{g}^{-1}$. The “structure” of C-S-H is thought to contain blocks of layer-structured calcium silicate material with silicon in a low state of polymerisation. This facilitates a strong unsatisfied surface charge that results in strong bonding to water molecules and to other C-S-H nano-particles. The arrangement of irregularly stacked blocks creates large volumes of micropores of varying spherical diameter. The large specific surface thus created, together with its high density of irregular hydrogen bonding, provides a strong potential for sorption.

The surface charge of C-S-H varies with its composition; Ca rich C-S-H has a positive surface charge, and as a result sorb anions such as sulphate. However, as the C:S ratio decreases, the surface charge diminishes, reaching zero at a C:S ratio of about 1.2 and eventually becoming negative at low C:S ratios. Thus SiO_2 rich C-S-Hs with low C:S ratios are better at sorbing cationic species than the CaO rich gels.

In cement-based s/s systems, C-S-H generated during the first few days of hydration is known to have an especially high chemical reactivity, occluding many toxic species which would otherwise be soluble. This is believed to be due to the importance of colloid generation and coalescence resulting in sorption of toxic species during early hydration. The amount of C-S-H generated during early hydration can be significantly increased by the addition of additives such as sodium silicate which contribute soluble silica. Promoting the generation of through-solution C-S-H in this manner can have a significant impact upon the ability of the system to remove dissolved species by sorption into or onto colloids.

2.4.4 Crystallochemical incorporation within the cement matrix

In addition to the poorly crystalline C-S-H, cement hydration also produces well crystalline phases which contribute to the immobilisation potential of cement-based systems. Some of these phases are listed in Table 26; the phases present can vary depending on the formulation of the cement, cure duration, temperature etc.

Table 26 – Crystalline products of hydrated cements

Crystalline Phase	Oxide Formulation
AF _t	Ca ₆ Al ₂ O ₆ (SO ₄) ₃ ·32H ₂ O
AF _m	Ca ₄ Al ₂ O ₆ (SO ₄)·12H ₂ O
C ₄ AH ₁₃	Ca ₆ Al ₂ O ₇ ·13H ₂ O
Hydrogrossularite or Hydrogarnet	Ca ₃ Al _{1.2} Fe _{0.8} SiO ₁₂ H ₈
Stratlingite or Gehlenite Hydrate	Ca ₂ Al ₂ SiO ₉ ·8H ₂ O
Portlandite	Ca(OH) ₂
Hydrotalcite	Mg ₄ Al ₂ (OH) ₁₄ ·2H ₂ O

AF_t - AF_t or Ettringite consists of an open structure with a framework of calcium, aluminium and sulphate. It has a high water content with water molecules randomly distributed within the structural tunnels. The AF_t framework is chemically very stable and has a low solubility despite the high water content. The sulphate (SO₄²⁻) in the AF_t framework can be substituted for by oxy-anions such as CrO₄²⁻, AsO₄³⁻, CO₃²⁻, NO₃⁻ etc. Additionally, the open channel structures of the framework provide a significant potential for the inclusion of small organic molecules in cement-waste composites.

AF_m - As with AF_t, AF_m or monosulphate is a sulphate containing crystalline phase of hydration. However, unlike AF_t, AF_m forms in the presence of limited sulphate availability and is meta-stable at room temperature. The capacity of AF_m to fix small mono-valent anions such as iodine in its I⁻ species is more superior to that of AF_t.

Other phases – Portlandite or Ca(OH)₂ does not offer any significant potential for crystallochemically incorporating waste constituents. The role played by other phases such as grossularite, stratlingite, gehlenite hydrate, hydrotalcite etc is not well understood at present.

1.5 General considerations of using cement-based s/s for treating oily drilling waste

Oily drilling waste as used for the study described in this report contains a cocktail of contaminants; hydrocarbons form a large part of it and also soluble chlorides. Studies have shown that heavy metal compounds – oxides and hydroxides, chlorides, sulphates, nitrates – interact in the hydration reactions of cement both during setting and latter during the hardening process⁰. In addition to affecting the setting and hardening rate, these interactions may also function to fix the metals chemically or physically in the microstructure. Some soluble chlorides such as the alkali chlorides may also be found dissolved within the pore fluid of the cement matrix and consequently may readily leach out upon contact with water. However, as the amount of readily available chloride is exhausted over time, a gradual decrease in leachability should be observed. Thus, secondary products produced from drilling waste would exhibit significant chloride leachability

The fixation of hydrocarbons

It is a fairly well researched fact that hydrocarbons, particularly high molecular weight compounds impede cement hydration by coating clinker grains, thereby preventing water molecules from reaching the clinker grains to initiate hydration. Soluble chlorides are mostly contained within the pore structure of the cement matrix and consequently removed as it comes into contact with

Appendix 3

Method statements for compressive strength testing

Cubes of 10cm x 10cm x 10cm will be cast and cured at room temperature as described below in accordance with BS 1881 – Part 108 & 111.

The compressive strength of the cubes will be determined after 3, 7 and 28 days of curing in accordance with BS 1881 - Part 116.

3.1 Preparation of test cubes (BS1881 Part 108)

Equipment:

- ◆ 100mm x 100mm moulds in accordance with BS 1881 Part 108.
- ◆ Oil or grease to thinly coat internal sides and base plate of the mould.
- ◆ Scoop, approx. 100mm wide.
- ◆ Plasterer's steel float.
- ◆ Compacting bar, at least 380mm long and having a ramming face 25mm square.

Procedure:

- ◆ Place the lightly oiled/greased mould on a rigid horizontal surface.
- ◆ Use the scoop to place concrete in the mould in layers approx. 50mm deep and compact each layer by using the compacting bar.
- ◆ The strokes should be uniformly distributed over the cross-section of the mould and ensure the bar does not penetrate significantly any previous layer nor forcibly strike the bottom of the mould when compacting the first layer.
- ◆ After the top layer has been compacted, smooth it level with the top of the mould, using the plasterer's float, and wipe clean the outside of the mould.
- ◆ The moulds should be left on a flat surface and covered with wet hessian and polythene sheeting and left for 24 hours in their mould.
- ◆ The temperature should be $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

3.3 Curing of test cubes at 20°C (BS 1881 Part 111)

Equipment:

- ◆ Curing tank constructed of corrosion-resistant material.
- ◆ Domestic water should be used to fill tank.
- ◆ Temperature should be $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

Procedure:

- ◆ Demould the specimens and use a wax crayon to mark each cube with the following information, date of casting, mix reference number and specimen reference number.
- ◆ Place specimens in water curing tanks.
- ◆ Each cube should be fully submerged.
- ◆ There should be at least a 15mm gap horizontally between specimens and to the sides of the tank to ensure adequate water circulation.
- ◆ Specimens should be removed from curing tanks immediately prior to compressive strength testing conforming to BS 1881 Part 116.