



TECHNOLOGY DEMONSTRATION PROJECT REPORT: TDP6



BIOPILE FIELD DEMONSTRATION AT
THE AVENUE COKING WORKS

CONTAMINATED LAND: **APPLICATIONS IN REAL ENVIRONMENTS**

CL: AIRE

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CL:AIRE was established as a public/private partnership in March 1999, to facilitate the field demonstration of remediation research and technology, including innovative methods for site characterisation and monitoring, on contaminated sites throughout the UK. The results of project demonstrations are published as research or technology demonstration reports and disseminated throughout the contaminated land community.

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**Contaminated Land: Applications in Real Environments
(CL:AIRE)**

October 2004

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This is a CL:AIRE Technology Demonstration Project Report. Publication of this report fulfils CL:AIRE's objective of disseminating and reporting on remediation technology demonstrations. This report is a detailed case study of the application of biopile technology based on specific site conditions at the Avenue Coking Works near Chesterfield. It is not a definitive guide to the application of biopile technology. CL:AIRE strongly recommends that individuals/organisations interested in using this technology retain the services of experienced environmental professionals.

EXECUTIVE SUMMARY

Avenue Coking Works in Wingerworth, Chesterfield was in operation for over 40 years producing smokeless fuels and associated coal carbonisation by-products. The site closed in 1992, and became the responsibility of East Midlands Development Agency (EMDA) as part of English Partnerships' National Coalfields Programme.

Jacobs Babtie (formerly Babtie Group) was commissioned by EMDA as principal consultants in the remediation and redevelopment of the 98 hectare site. In order to identify the nature and extent of soil and groundwater contamination at the site, an extensive site investigation involving approximately 135 boreholes and 320 trial pits was undertaken at the site. They were positioned using a 25 m herringbone grid pattern in areas which were likely to be heavily contaminated, and a 50 m herringbone grid was used where contamination was considered less likely. The contamination was most prevalent in three main areas, the solid waste tip containing builders' rubble, metal, and gas works derivatives; lagoons of grossly contaminated silts; and the main plant area. Contaminants of major concern for all three locations include polycyclic aromatic hydrocarbons (PAH); phenols; mineral oil; benzene, toluene, ethylbenzene and xylene (BTEX); and cyanides.

The geology at the site consists of unconsolidated superficial deposits of made ground, soil and alluvium underlain by the Lower and Middle Coal Measures of Carboniferous Age.

A programme of field demonstrations was carried out to assess a number of remedial technologies including bioremediation.

Following a tendering process, a contract was awarded to DEC NV to undertake a solid phase bioremediation trial. DEC NV chose to demonstrate the potential for bioremediation using a three phased approach. This consisted of slurry biodegradation tests, followed by a bench-scale bioreactor test and then a field-scale active biopile.

DEC NV undertook laboratory-scale slurry biodegradation tests on different samples from the site provided by Jacobs Babtie. The samples included two samples from the waste tip and two samples from the plant area.

The slurry and bioreactor laboratory tests demonstrated that both the waste tip and the plant area materials were suitable for bioremediation and therefore those materials were used in the field-scale biopile.

The field-scale biopile was undertaken in a purpose-built weatherproof shed which was constructed on a concrete base. This design allowed greater control over moisture levels, the rate of injection of air into the biopile and allowed the air emissions to also be monitored. The shed was equipped with an air extraction system. The biopile consisted of 200 m³ of screened contaminated material, half from waste tip material and half from plant area material. After placement, the material was pre-treated with a commercial nutrient formula and then mixed and sampled. The biopile was mixed and sampled every two weeks with water being added when necessary to maintain the moisture content at approximately 70 %.

The field-scale biopile confirmed the biodegradation patterns found in the laboratory tests. Plant area material with high initial naphthalene concentrations only showed naphthalene and 3-ring PAH degradation during the 13 week treatment period. Waste tip material with low initial naphthalene concentrations showed 2, 3 and 4-ring PAH removal. Removal of BTEX and phenols was observed for both samples.

In conclusion, the trial of solid phase bioremediation using biopile technology demonstrated that limited biodegradation took place. Field-scale treatment only lasted 80 days and therefore was not running to its end due to time pressures. The respiration data and microbial activity remained high in most of the samples when the trial was stopped. For BTEX, naphthalene, phenol, mineral oil and some 3 and 4-ring PAH, significant reductions in material concentrations were observed. However, the usefulness of bioremediation of these materials will ultimately depend on the cleanup criteria that are set, because significant residual concentrations of these compounds remain present in the material after treatment. Moreover, a number of contaminants such as higher ring PAHs, showed little sign of degradation.

The cost to undertake the biopile field trial was £91,269 which equates to approximately £183 per tonne based on 500 tonnes. This cost did not include any laboratory analysis undertaken by EMDA. The cost per tonne is very high as it was a trial and the volume of material being treated was small and was treated in a purpose-built shed. In fixed location treatment centres, contaminated material similar in composition can be treated using biopile technology for about £15 - £20 per tonne. On site treatment costs could be slightly less than this. However, if material had to be treated on site in a shed to prevent odour then the cost would rise to about £25 - £35 per tonne.

The cost of disposing of this type of material to landfill was running at about £30 - £40 tonne including landfill tax before the implementation of the Landfill Directive (July 2004). From July 2004, these costs are likely to rise considerably as the landfill tax rate increases, and the options for disposing of hazardous and non-hazardous material from contaminated sites to landfill significantly reduces due to the limited volume of hazardous landfill space available.

ACKNOWLEDGEMENTS

This report was prepared from information provided from technical reports and discussions with individuals who were involved with the remediation trials at Avenue Coking Works. In particular CL:AIRE would like to acknowledge the contribution from the East Midlands Development Agency, DEC NV and Jacobs Babbie.

CL:AIRE also wishes to acknowledge Dr Gordon Lethbridge of Shell Global Solutions and Dr Russell Thomas of Parsons Brinkerhoff who reviewed and commented on the draft report.

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ABBREVIATIONS

AEG	Allied Exploration & Geotechnics Limited
AvR	Dutch Council for Accreditation
B(a)P	Benzo (a) Pyrene
bgf	Below ground level
BTEX	Benzene, Toluene, Ethylbenzene and Xylene
CDM	Construction Design and Management
CLEA	Contaminated Land Exposure Assessment
CMS	Chip Measurement System
C:N:P	Carbon to nitrogen to phosphorus ratio
DEFRA	Department of the Environment, Food and Rural Affairs
DOE	Department of Environment
DRO	Diesel Range Organics
EA	Environment Agency
EC	European Commission
EMDA	East Midlands Development Agency
GC-FID	Gas Chromatography-Flame Ionisation Detector
GCMS	Gas Chromatography Mass Spectroscopy
H _c	Henry's Law constant
HLSA	High Level Stocking Area
HSP	Health and Safety Plan
HPLC	High Performance Liquid Chromatography
ICRCL	Interdepartmental Committee for the Reclamation of Contaminated Land
km	Kilometre
K _{ow}	Octanol-water partition coefficient
kVA	Kilo Volt Amp
LDPE	Low Density Polyethylene
LLSA	Low Level Stocking Area
LOI	Loss on Ignition
mg/kg	Milligram per kilogram
mm	Millimetre
NA	Not Available
NRA	National Rivers Authority
OJEC	Official Journal of the European Community
OS	Ordnance Survey
PAH	Polycyclic Aromatic Hydrocarbons
PID	Photoionisation Detector
PRO	Petrol Range Organics
PVC	Polyvinyl Chloride
QA/QC	Quality Assurance/Quality Control
SCN ⁻	Thiocyanate
SEM	Solvent Extractable Matter

SNIFFER	Scotland and Northern Ireland Forum for Environmental Research
TNVA	Total Non Volatile Aromatic
TOC	Total Organic Carbon
UKAS	United Kingdom Accreditation Scheme
USEPA	United States Environmental Protection Agency
UV	Ultra Violet
VOC	Volatile Organic Compound
µg/l	Microgram per litre
µS	Microsiemens

1. INTRODUCTION

1.1 BACKGROUND

Avenue Coking Works in Wingerworth, Chesterfield was in operation for over 40 years producing smokeless fuels and associated coal carbonisation by-products. These operations have caused extensive land contamination which will require remediation if the site is to be redeveloped. The three main primary sources of contamination are: a waste tip containing builders rubble, metal and coal gasification derivatives; a lagoon; and a contaminated plant area. Contaminants of major concern for all three locations include polycyclic aromatic hydrocarbons (PAH); phenols; mineral oil; benzene, toluene, ethylbenzene and xylene (BTEX); and cyanides.

Jacobs Babtie (formerly Babtie Group) was commissioned by EMDA as principal consultants in the remediation and redevelopment of the 98 hectare site. A detailed site investigation involving approximately 135 boreholes and 320 trial pits has been undertaken at the site. A programme of field demonstrations was carried out to assess a number of remedial technologies, including bioremediation. Following a tendering procedure, a contract to undertake a field trial of solid phase bioremediation using biopile technology was awarded to DEC NV, a company headquartered in Belgium with UK operations located in East Grinstead, West Sussex. The aim of the trial was to assess the bioremediation potential of the waste tip and plant areas of the site.

DEC NV chose to demonstrate the potential for bioremediation using biopile technology and a three phased approach was designed for the trial. The first two phases were laboratory-based and consisted of a slurry biodegradation test followed by a bench-scale bioreactor test. A third phase field-scale trial was designed using results from the laboratory tests and this is the focus of this report.

Method statements and health and safety procedures were prepared and approved and regulatory approval was received from the Environment Agency (EA).

During 2001, slurry biodegradation tests were conducted between April and August, bioreactor tests between July and September and the field trial from July to October. The field trial was performed within a purpose-built enclosure on the concrete hardstanding of the former coke stocking area at the Avenue Coking Works. Chemical analyses were performed by ALcontrol Laboratories, located in the UK and Holland.

1.2 PURPOSE AND OBJECTIVES

The purpose of the field trial was to help assess the technical and economic performance of an *ex situ* biopile technology that might be applied as part of the full-scale remediation of the site.

The purpose of this report is to describe the design and construction of the active biopile and provide an objective assessment of the performance of the technology. Specific objectives include:

- A description of site characteristics including the nature and distribution of contaminants
- A description of the design and operation of an *ex situ* biopile
- An assessment of the technical performance of *ex situ* active biopile technology
- An assessment of system costs

1.3

REPORT ORGANISATION

The report is divided into the following sections:

1. Introduction;
2. Background to Solid Phase Soil Bioremediation;
3. Site Description;
4. Laboratory Treatability Studies;
5. Technology Demonstration Support Issues;
6. Biopile Construction and Demonstration;
7. Performance Evaluation;
8. Economic Considerations, and;
9. Conclusions.

2. BACKGROUND TO SOLID PHASE SOIL BIOREMEDIATION

2.1 INTRODUCTION

This chapter provides a summary of bioremediation and biopile technology. Additional discussion can be found in King *et al.*, (1998) and in appropriate Environment Agency remedial data sheets (2001).

2.2 WHAT IS BIOREMEDIATION?

Bioremediation involves the use of microorganisms, commonly bacteria or fungi, to transform or degrade contaminants ultimately to non-toxic by-products. It includes biodegradation (breakdown of compounds) and bioaccumulation (accumulation of compounds, in particular metals, by the microorganisms) and it is the most common *ex situ* remediation technique used in the UK. *Ex situ* bioremediation ranges from simple landfarming where contaminated soil is aerated by mixing, through to engineered passive and active biopiles and windrows to more complex bioreactors, which involve the treatment of contaminated soil in enclosed reactor vessels under controlled conditions. Bioremediation can also be carried out *in situ*. The heterogeneity of the subsoil and the type of soil on many sites generally renders *in situ* techniques very difficult to complete successfully.

Biodegradation processes harness the ability of microorganisms to use compounds present in the environment as nutrient sources. This includes compounds rich in carbon (e.g. other organic contaminants and hydrocarbons) or containing nitrogen, phosphorus and certain trace elements. Some of these compounds can be harmful to human health or represent a risk to the environment and are regarded as contaminants.

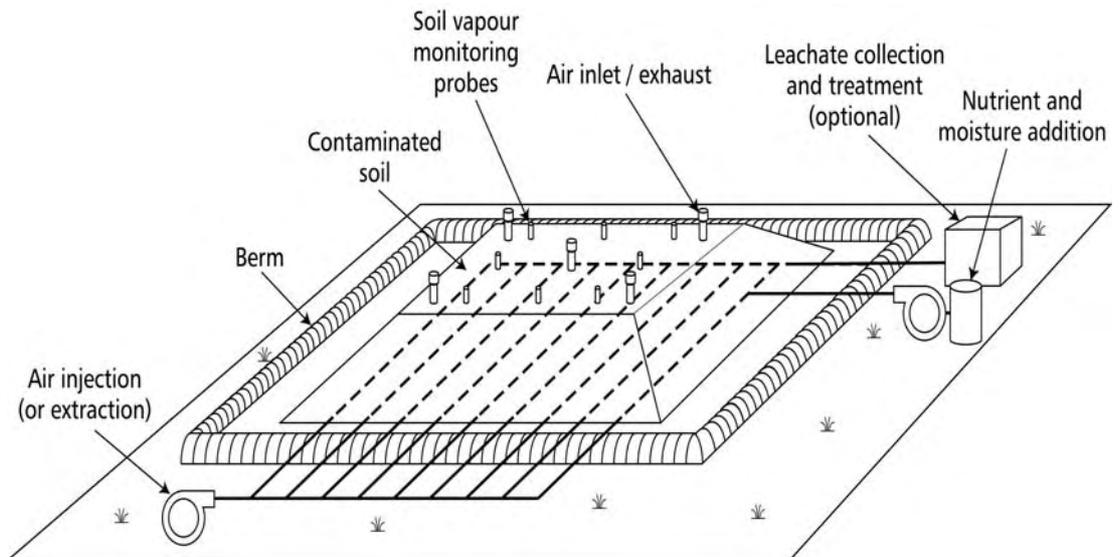
The principal advantages of bioremediation are that contaminants are destroyed without destroying the biotic content of the soil; soil structure is retained; the soil can be reused; the process uses harmless reagents; and the process can be carried out on site which reduces transportation costs and further environmental impact. A limitation of the process is that it can require a relatively long remediation time, depending on the ground conditions and the contaminant.

Contaminants that can be treated by bioremediation include petroleum products such as gasoline, diesel and crude oil which contain complex mixtures of aliphatic and aromatic hydrocarbons. Biodegradation of aliphatic and aromatic hydrocarbons becomes more difficult with increasing carbon chain length and with the increased number of aromatic rings. For example, biodegradation of 2, 3 and 4-ring PAHs is possible, but soil contaminated with 5 or 6-ring PAHs is more difficult to treat.

For all organic compounds the age and degree of weathering is a key factor. During weathering, contaminants are subjected to leaching, volatilisation and biodegradation of the more soluble components. Contaminants which remain in the ground for a long time are typically less bioavailable and, consequently, biological breakdown will proceed at a much slower rate. However, some natural *in situ* degradation will already have occurred over the period prior to the start of the remediation.

2.3 WHAT IS A BIOPILE?

A biopile is an *ex situ* engineered treatment system which involves mounding the contaminated soil or material in a contained area in such a way as to optimise biodegradation conditions (see Figure 2.1).



Adapted from: New Mexico Environment Department

Figure 2.1: Schematic of a biopile

Biopiles can be either an on-site or off-site technique. To optimise the biological breakdown, a number of parameters must be controlled and/or adjusted. Under aerobic conditions, the most important parameter is the oxygen content. In actively managed biopiles an air injection or air-extraction system is used to optimise oxygen levels within the pile. Furthermore, the following parameters are usually also optimised:

- Structure of the soil (frequent homogenisation, addition of bulking agents e.g. wood chippings, sand)
- Nutrient content (addition of nitrogen and phosphorus)
- Moisture content (maintaining soil moisture at between 40 % – 88 % of soil field capacity)
- pH (adding of alkaline or acid reagents)

Typically, biopiles are constructed to a height of between 0.5 m and 4 m. The average lifespan of a biopile can be from a few months to several years, depending on the intensity of the operations, the nature of the contaminants and the desired final contaminant concentration.

Points of special significance include the design of the aeration system, the clay and organic content of the soil, the potential presence of inhibitors of microbial growth and the permeability of the soil:

- High clay content can result in poor aeration and can result in poor bioaccessibility as contaminants diffuse into micropores whose throats are too narrow to let microbes in
- Organic matter is important with respect to adsorption of organic contaminants
- Contaminants such as cyanides may be present in sufficient concentrations as to be toxic to the microorganisms
- Soils must be sufficiently permeable to allow the transport of oxygen (or other electron acceptors), moisture and nutrients

The treatment of low permeability, clay-rich soils is a particular challenge since the migration of solutions, gases and the microorganisms themselves is more limited. Pre-treatment of such soils, which might include disaggregation, and the use of bulking materials as straw, wood chips, chopped tyres, is often carried out to increase the permeability.

Section 2.4 discusses the science of bioremediation starting with the classification of living organisms followed by physical and chemical requirements necessary for biodegradation.

2.4 SCIENCE OF BIOREMEDIATION

2.4.1 WHAT ARE MICROORGANISMS?

The term 'microorganisms' includes bacteria, archaea, protozoa, algae and fungi. All living organisms can be divided into prokaryotes and eukaryotes (see Figure 2.2). Prokaryotes consist of bacteria and archaea whilst eukaryotes consist of unicellular organisms (protozoa, fungi and algae) and multicellular organisms (animals and plants).

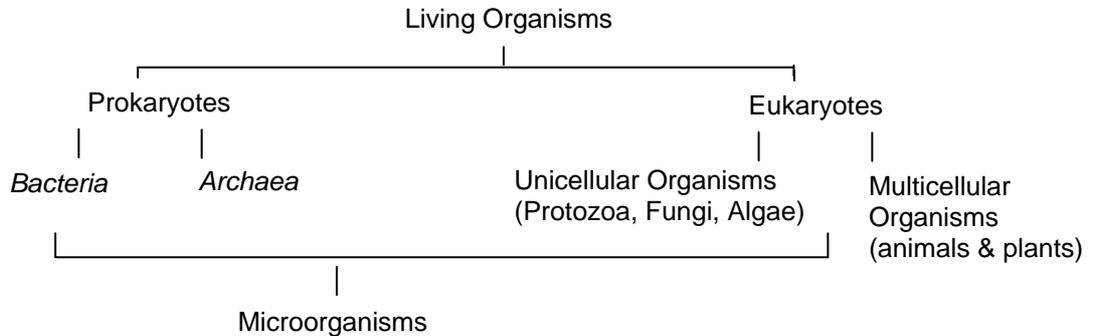


Figure 2.2: Classification of living organisms

In engineered bioremediation systems, bacteria and fungi are the important microorganisms. In certain situations fungi can be equally if not more important than bacteria. At the wet end of the optimal range, bacteria predominate, but at the drier end fungi predominate.

2.4.2 BIODEGRADATION REQUIREMENTS

The physical and chemical requirements to sustain bacterial life are illustrated in Figure 2.3.

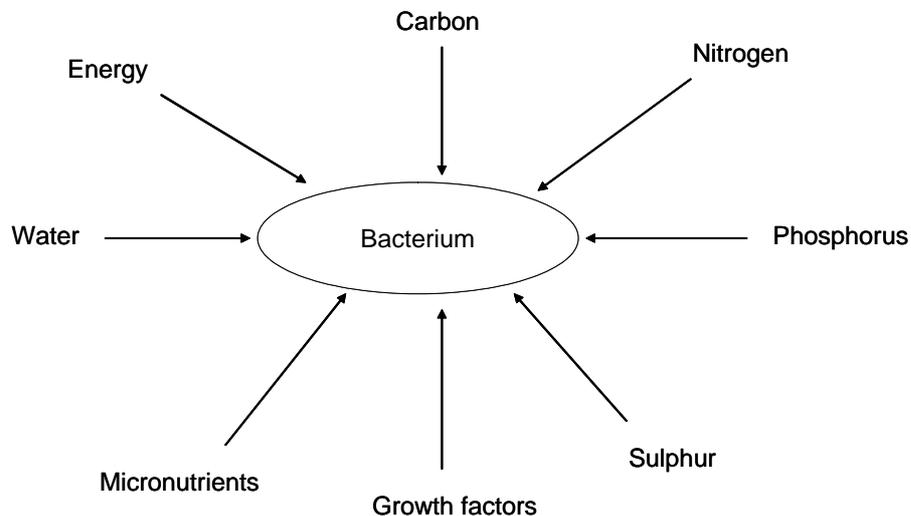


Figure 2.3: Physical and chemical requirements for bacteria

2.4.2.1 Nutrients and Water

Carbon, nitrogen, phosphorus and sulphur are essential requirements because they form the basis of all living organisms whilst phosphorus is also essential to make nucleic acids. Micronutrients include elements like zinc, copper and molybdenum. Other essential growth factors (substrates) are pre-formed organic compounds, required as nutrients by the microorganisms. In general, materials can only be transported across cell membranes in soluble form, so water is a requirement for all biochemical processes.

Carbon is required by most organisms as a nutritional substrate (or food source) for energy and growth. Those organisms that use organic carbon (e.g. hydrocarbons) are called heterotrophs and most microorganisms, including bacteria, belong to this group. Heterotrophs are the key organisms for bioremediation of organic compounds.

2.4.2.2 Energy

Heterotrophs can be subdivided into photoheterotrophs, such as green and purple sulphur bacteria which exploit light as a source of energy, and chemoheterotrophs, which exploit chemical forms of energy. Most microorganisms used in bioremediation are chemoheterotrophs. Table 2.1 summarises the energy and carbon sources for heterotrophs and gives some examples of each.

Table 2.1: Energy and carbon sources for selected heterotrophs

Heterotroph type	Energy source	Carbon source	Examples
Photoheterotrophs	Light	Organic	Green and purple sulphur bacteria
Chemoheterotrophs	Chemical	Organic	Most microorganisms e.g. <i>Pseudomonas sp.</i>

The biodegradation of organic carbon compounds (by chemoheterotrophs) is the result of microorganisms obtaining the energy that they require to survive and reproduce from the breakdown of chemical bonds in the carbon substrate. Enzymes are used to catalyse the bond-breaking process. The progressive breaking apart of the substrate eventually results in the conversion of harmful contaminant into either harmless or less-harmful substances.

The two main ways that heterotrophic microorganisms obtain the energy they require are via:

- Respiration (aerobic and anaerobic)
- Fermentation (anaerobic only)

During respiration an energy transfer process occurs which is mediated by a linked series of oxidation-reduction reactions that transfer electrons from a donor compound to another compound called the electron acceptor. When oxygen acts as the terminal electron acceptor the process is called aerobic respiration and carbon dioxide and water are produced as by-products (see Figure 2.4). In fermentation an organic compound acts as the electron acceptor.

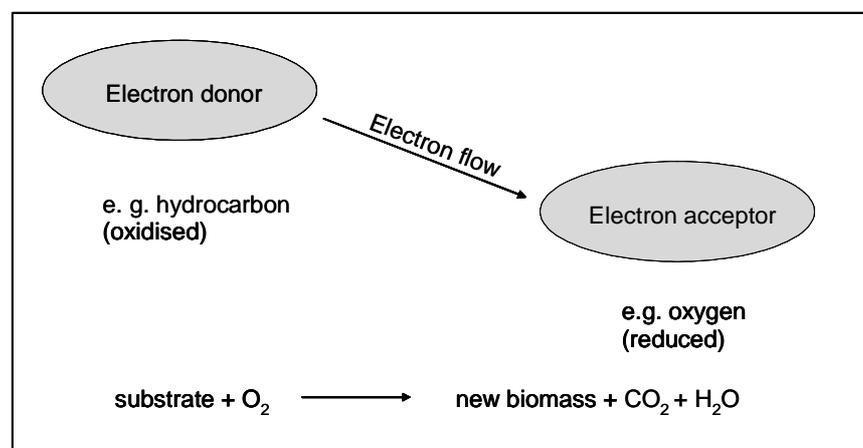


Figure 2.4: Aerobic respiration

However, other compounds such as sulphate, carbon dioxide and nitrate can also act as electron acceptors and when this occurs the process is called anaerobic respiration.

Generally, bioremediation uses aerobic microorganisms (which use aerobic respiration) although anaerobic bacteria (which use anaerobic respiration) are increasingly being used in some field situations and bioreactors.

Microorganisms which obtain their energy through fermentation are not generally used in bioremediation.

All reduced organic compounds can potentially act as sources of energy and hence are potential electron donors. Some are broken down less easily than others and these are described as 'recalcitrant' compounds (e.g. polychlorinated biphenyls).

2.4.2.3 Metabolic Pathways

For the biodegradation of complex hydrocarbons, several different enzymes are usually required to complete full degradation of the compounds which constitute the contaminant(s). The series of reactions by which the compounds are metabolised are called biodegradation pathways. These complex pathways are often interlinked with other metabolic pathways which allow the organism to convert these compounds into a wide range of other compounds.

Any one compound can follow many alternative degradation pathways depending on the specific organisms involved and whether the degradation is aerobic or anaerobic. Many of the common contaminants, such as naphthalene, phenol, benzene, phenanthrene and nitrobenzene, have interrelated degradation pathways. For example, naphthalene and nitrobenzene pathways both go via catechol and merge at catechol.

There are thousands of different microorganisms involved in biodegradation. Those most commonly found are bacteria of the genus *Pseudomonas*, including species such as *Pseudomonas fluorescens* and *Pseudomonas putida*. Bacteria of the genera *Bacillus*, *Acidovorax*, *Alcaligenes*, *Arthrobacter* and *Rhodococcus* have also been identified in material that has been bioremediated.

2.4.3 BIODEGRADATION IN BIOPILES

Biodegradation in aerated biopiles is predominantly the result of the action of chemoheterotrophic bacteria and fungi, using aerobic respiration as their energy source and a readily available carbon source. As discussed in Section 2.4.2.2, oxygen acts as the terminal electron acceptor during aerobic respiration and hence aerobic conditions need to be maintained in this form of biopile for conditions to remain optimal for biodegradation. The pile only needs to be aerated if the contaminant in question degrades better under aerobic conditions. Aerobic piles are best suited for hydrocarbon contaminants. However, an anaerobic pile may be suitable for highly chlorinated contamination which degrades better under these conditions.

Microorganisms are mostly found adsorbed onto soil surfaces which are in contact with any water present. Here they can form complex structures called biofilms. Biofilms provide an advantage to microorganisms, as they allow them to exist in a microenvironment that can chemically buffer against the less favourable conditions which may prevail in the surrounding water.

2.5 DEVELOPMENT OF BIOPILE TECHNOLOGY

Ex situ bioremediation was practised in the early years of the 20th century for the treatment of petroleum sludge from the oil industry (King *et al.*, 1998). Over subsequent decades a greater understanding of the processes taking place was obtained with process optimisation involving the addition of fertilisers, water management and pH control.

Ex situ bioremediation of a range of organic compounds has now been successfully applied at many sites in the UK. Materials contaminated with PAHs, often from former gasworks and colliery sites, have been bioremediated using a variety of *ex situ* techniques including biopiles.

2.6 PERFORMANCE OF BIOPILES

2.6.1 WHAT CAN BIOPILES TREAT?

Biopiles have been used to treat soils and similar material contaminated with BTEX, phenol, PAHs, nitroaromatics and herbicides/pesticides (EA, 2001). PAHs with two or three aromatic rings are degraded quite easily. PAHs with four aromatic rings are also degradable, but the breakdown occurs at a much slower rate. Biodegradation of PAHs with four or more aromatic rings, such as benzo(a)pyrene, occurs only very slowly in biopiles due to poor bioavailability, but is possible in bioreactors where environmental conditions can be more closely controlled.

2.6.2 VERIFICATION OF REMEDIAL TREATMENT USING BIOPILES

Increasing rates of landfill tax and the introduction of the Landfill Directive (Council of the European Commission, 1999) into UK law in July 2004 has made it more difficult to dispose of hazardous and non-hazardous material from contaminated sites to landfill, so more and more land will be remediated by on-site treatment of material. At the same time the new contaminated land regime introduced under Part IIA of the Environmental Protection Act 1990 requires that contaminated land is remediated to acceptable levels rendering the site suitable for use. Therefore, it is important to verify the performance of a biopile to establish:

- That any remedial targets for the contaminated material have been achieved
- That any reductions in contaminant concentration are due to bioremediation and not processes such as dilution and mixing
- Confidence in the use of the technology

Verification can be undertaken by:

- Sampling and analysis of material in the biopile during the treatment process
- Understanding and assessing the contribution that other processes, aside from bioremediation, may be making towards reducing contaminant levels

Additional discussion can be found in Environment Agency publications (2000a, 2000b and 2002 in draft).

2.6.2.1 Sampling and Analysis

Characterising heterogeneous contaminated material, whether *in situ* on a contaminated site or after being moved to a heap or stockpile prior to treatment, is extremely difficult. The sampling regimes used to characterise such situations are often not statistically robust. The number of samples that are necessary to characterise the concentration of a particular contaminant is related to the variability in contamination and the degree of confidence required in any sample mean that is to be calculated.

Sampling is time consuming and usually costly. Therefore, when using a biopile to remediate contaminated material, sampling should be focussed at the time when treatment is about to commence ($t=0$) and when treatment has ended ($t=end$). Spot sampling, measuring respiration levels and other techniques can be used during the operation to determine when $t=end$ is near.

Significant variations in the reported analyses of contaminated material are often found when identical samples are sent to different analytical laboratories, even when identical analytical protocols are used. Therefore, to reduce the likelihood of errors, samples should be

collected using agreed sampling protocols and then analysed at laboratories with recognised quality assurance and quality control systems in place.

2.6.2.2 Awareness of Other Processes

Reductions in contaminant concentrations during treatment are often attributed to bioremediation when other processes are wholly or partly responsible. These processes include:

- Loss of volatile material e.g. naphthalene, BTEX compounds
- Dilution/homogenisation of material
- Sorption of organic compounds to bulking agents
- Leaching following natural rainfall infiltration or artificial irrigation

Volatile losses can be measured and quantified during initial treatability studies carried out in a laboratory. However, these losses are difficult to quantify during an actual biopile operation which is usually open to the atmosphere. Even biopiles that are constructed within sheds are not completely enclosed: they have to be opened occasionally to allow the entry and exit of earth moving equipment.

The dilution of contaminated material on site can be monitored by retrieving good t=0 data along with the addition of bulking agents.

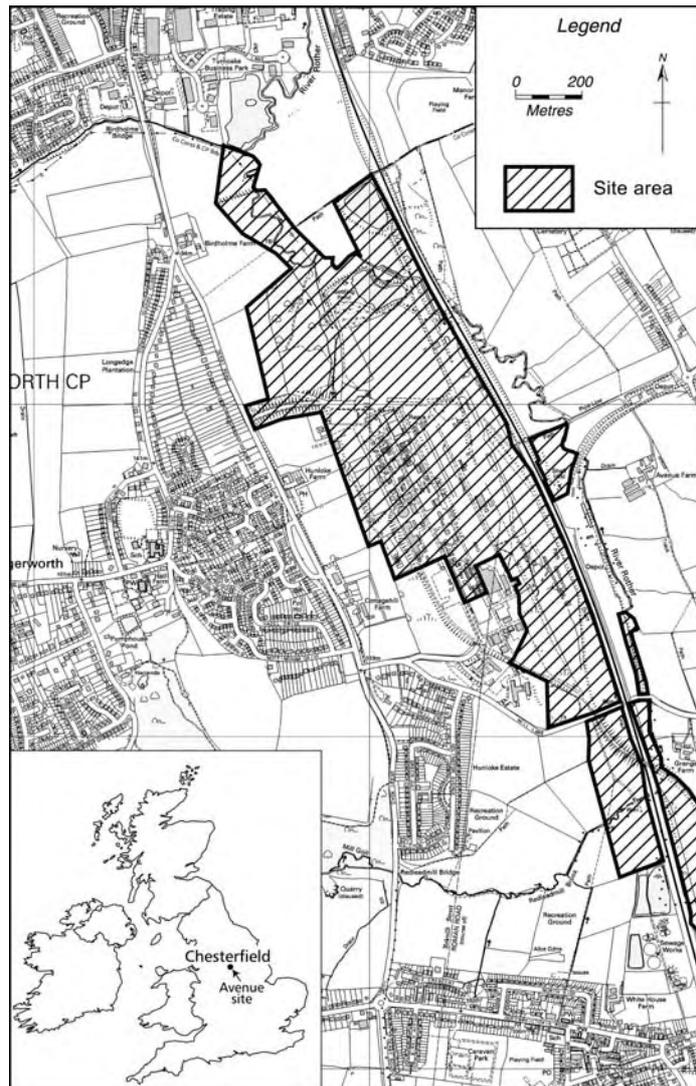
Leachate that is generated following the infiltration of rainfall or water through artificial irrigation can be collected and analysed to identify any contaminant losses that are not attributable to leaching.

Other indirect evidence of bioremediation using conservative biomarkers can be gathered. The production of carbon dioxide and consumption of oxygen during microbial respiration are indicators that biodegradation is occurring. These can be measured during initial laboratory-based treatability studies and during the field-scale biopile operation on contaminated material.

3. SITE DESCRIPTION

3.1 SITE LOCATION

The Avenue Coking Works is approximately 3 km southeast of Chesterfield and 30 km northeast of Nottingham in the county of Derbyshire (OS SK 438367). It covers 98 ha between the River Rother to the east and the A61 road to the west. The site is approximately 3.2 km long, north to south, and 0.7 km at its widest extent (Figure 3.1). The main London to Sheffield railway runs along the eastern boundary and junction 29 of the M1 motorway is 8 km to the east.



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Figure 3.1: Site location plan

3.2 SITE USAGE

The site was originally used for agriculture. From approximately 1883 until 1940, the eastern central region of the site was occupied by the Avenue Colliery Lime and Ironworks. The Avenue Coking Works and Chemical Plant was constructed in the mid-1950s as a fully integrated facility producing smokeless fuels and associated coal carbonisation by-products, such as sulphuric acid, ammonium sulphate, creosote and blended fuels. Town gas was also produced on site and supplied to the town of Chesterfield. Those works all closed in

1992. The majority of the site is now owned by the East Midlands Development Agency (EMDA). A small portion of the site, west of the main plant area, is currently leased to a manufacturer of bitumen-based products.

All above ground buildings, tanks and structures associated with these earlier operations (mainly located in the middle and west of the site) along with any residual surface materials (excluding those stockpiled for reuse during redevelopment), were cleared in a programme of demolition completed in March 2003.

Current surrounding land use is predominantly agricultural although the residential area of the Hunloke Estate lies approximately 300 m to the southeast of the site. A small industrial estate adjoins the site to the southwest. Immediately to the west of the site are open fields which are bounded by the A61, Sheffield to Derby road. To the west of the road lies the predominantly residential district of Wingerworth.

The site has been divided into six zones to help in the organisation and interpretation of recent site investigations. A description of each zone, as they were during the remediation trials, is given in Table 3.1.

Table 3.1: Surface conditions in each zone during remediation trials

Area		General Description
Zone 1	South of Mill Lane	This area is divided on a N-S axis by the main Sheffield to London railway line. To the west lies open farmland and to the east former rail sidings.
Zone 2	Eastern Site (coal handling/processing area)	The eastern site contains the former coal handling system and coal processing and storage facilities. 2.5 ha of hardstanding was used for bulk coal storage. A small area of land east of the railway was occupied by a colliery. Coke oven batteries and water cooling towers are situated in the north west corner.
Zone 3	Main Plant Area	The main plant area contains the former by-products processing facility and associated reservoirs, and includes a benzole rectification plant, tar plant, acid and base plant, creosote plant, pitch plant, gas purification plant and acid manufacturing plant. This area of the site is covered by large areas of hardstanding and an intricate and extensive network of pipes, gantries, distillation towers, tanks and associated containment systems. Two reservoirs are located to the north of this zone and are used to manage and treat contaminated run-off from the former waste tip and containment water from the main plant area.
Zone 4	Former Waste Tip, Lagoon 2 and High Level Stocking Area (HLSA)	The former waste tip, now overgrown by small trees and scrub, covers approximately 3 ha. Leachate from the tip discharges into Lagoon 2 and Pond 1. The HLSA was used to store quenched coke and covers approximately 5 ha immediately east of the landfill. The western boundary of the HLSA may extend over the waste tip.
Zone 5	Low Level Stocking Area (LLSA)	The LLSA is located immediately west of the former waste tip (Zone 4). Like the HLSA this area was used to store quenched coke. Scrubland makes up the remainder of the zone.
Zone 6	River Rother flood plain and Lagoon 4	The flood plain area is covered by open scrubland and marshland between the meanders of the River Rother. Lagoon 4 is in the most north easterly area of this zone and received wastewater and lime sludges from the main plant area.

Adapted from: Babtie Group (2001)

3.3 SUMMARY OF EARLIER ENVIRONMENTAL INVESTIGATIONS AND REPORTS

Between 1991 (the year preceding the closure of Avenue Coking Works) and 1996, several site investigations were undertaken to develop an understanding of site geology and hydrogeology and to assess the nature and extent of soil, surface water and groundwater contamination. In addition, the extent of any remaining coal reserves beneath the site was investigated.

3.4 RECENT INVESTIGATIONS

In 1999 Allott and Lomax, consulting engineers (now part of Jacobs Babbie), was appointed by EMDA as lead consultants to supervise and undertake further site investigations and risk assessments prior to redevelopment. This new work was carried out to address any deficiencies arising from the earlier site investigations and aid in the preparation of a remediation strategy to assess unacceptable risks to human health and the environment before any new land use commenced.

Investigation works were carried out in two stages. Stage 1, undertaken by Allied Exploration & Geotechnics Limited (AEG) during September and October 1999 and Stage 2, carried out by Wimtec Environmental Limited between August 2000 and April 2001.

3.4.1 STAGE 1 – SITE WORKS

Stage 1 works were limited to investigating the nature and extent of soil and groundwater contamination in and around the River Rother flood plain and Lagoons 2 and 4 (Zones 4 and 6).

Twenty-one trial pits were excavated to depths of between 2.3 m - 4.0 m below ground level (mbgl) and 24 boreholes were drilled. Twenty of the boreholes were drilled using cable percussive techniques to depths of between 3.5 mbgl and 16.8 mbgl and six of these were extended using rotary drilling to a maximum depth of 30 m. The remaining four boreholes were drilled using rotary open hole techniques only. All boreholes were completed as combined gas and groundwater monitoring wells. A further 15 window sample holes were drilled to a maximum depth of 7.0 mbgl on the Lagoon 2 retaining bund. Representative soil and groundwater samples were collected for chemical testing.

3.4.2 STAGE 2 – SITE WORKS

Stage 2 consisted of a comprehensive investigation across and outside the boundaries of the site. The main objectives were to:

- Determine if historic site activities had adversely impacted the environmental condition of the site
- Assess the degree and nature of any risk posed by contaminants to the site owner and adjacent land users or receptors
- Identify soils that would be suitable for treatment during the on site remediation trials
- Assess the suitability of the made ground and superficial deposits for future earthworks on site

The design of the site investigation took into account historic site information. Trial pits and boreholes were positioned using a 25 m herringbone grid pattern in areas which were likely to be heavily contaminated, and a 50 m herringbone grid was used where contamination was considered less likely. Additional test locations were selected in those areas with potential contamination hot spots.

Across the site 273 trial pits were dug and 109 boreholes drilled to various depths. In addition 16 boreholes were drilled outside the site to establish upgradient and downgradient groundwater quality. Trial pits were typically excavated to an average depth of 4.0 m, the maximum depth being 5.5 m. Boreholes were drilled in the made ground, alluvium and Coal Measures. Mean thicknesses of 15 m, 1 m and 27 m respectively were identified within the

boreholes. The maximum borehole depth was 100 m. Of these boreholes, 106 were completed as gas and groundwater monitoring wells. Figure 3.2 shows selected trial pit and borehole locations within Zones 3 and 4. All the trial pit and borehole locations within these zones are not shown, only those in areas from where the materials for the remediation trials were later excavated.

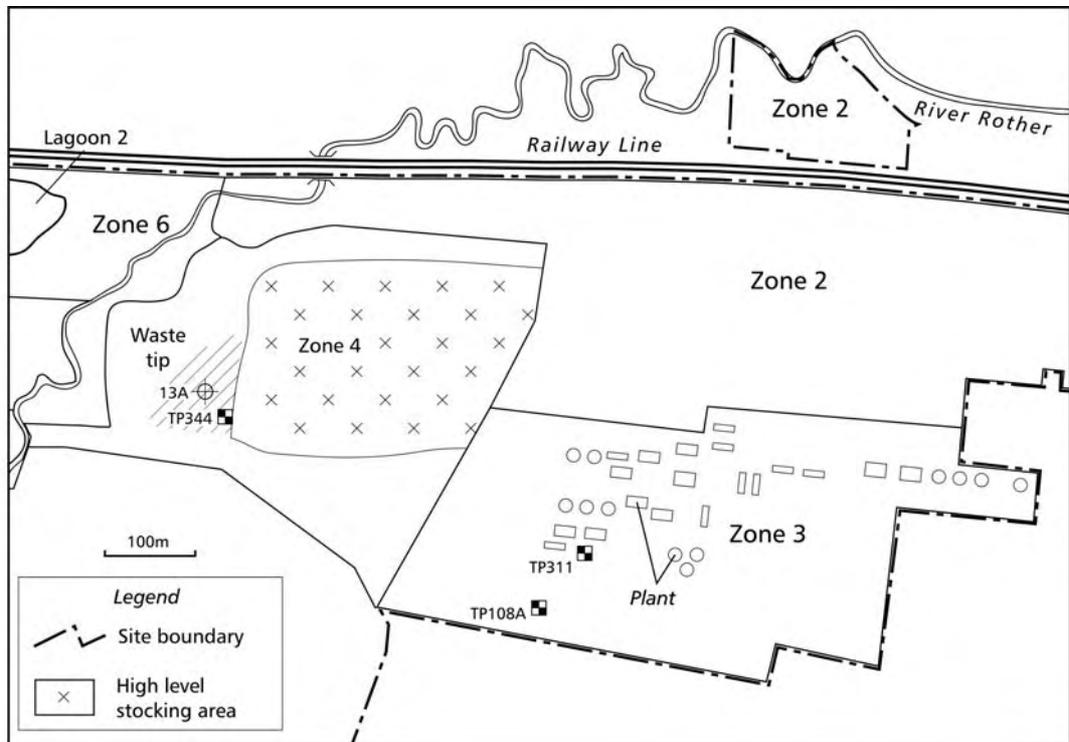


Figure 3.2: Location of selected trial pits and boreholes in Zones 3 and 4

The conceptual model has now been developed and the risk assessments are currently being validated. Work on the remediation strategy is ongoing.

During this period, remediation trials to determine which technologies would be suitable to treat contaminated soil at the site were also carried out.

3.5 GEOLOGY AND HYDROGEOLOGY

3.5.1 GEOLOGY

Site geology is described in terms of unconsolidated superficial deposits and bedrock geology.

3.5.1.1 Superficial Deposits

Superficial deposits at the site consist of made ground, top soil and alluvium. Thicknesses of made ground generally range between 0.5 m and 4.0 m but much thicker deposits up to 18 m and 10 m exist in Zone 4-HLSA and Zone 2 respectively. Made ground was absent from most areas of Zone 6 except in the vicinity of Lagoon 4 where depths up to 10 m were found. Made ground was absent from the fields covering the northwest and central parts of Zone 5 although thin layers varying between 0.2 m and 0.5 m persisted elsewhere. Generally the made ground consisted of silty sand, gravel and clay with soil, burnt shale, ash, slag, coal fines, wood, paper and miscellaneous waste.

Top soil in Zones 1 - 4 was between 0.3 m and 0.9 m in thickness. In Zones 5 and 6 top soil thickness ranged between 0.1 m and 1.6 m. Top soil was often absent from those parts of Zone 6 in the vicinity of the river.

Alluvium associated with the River Rother and its tributaries underlies the soil cover in Zones 1, 2, 4, 5 and 6 but was absent from Zone 3. The alluvium consists of between 2 m – 3 m of silty clay with intermittent lenses of sand and gravel. Greatest thicknesses of alluvium are found in Zone 2 and Zone 6 in the flood plain area. Due to river diversions deposits are often found some distance from current water channels.

3.5.1.2 Bedrock Geology

The site is underlain by the Lower and Middle Coal Measures of Carboniferous Age. Depths to weathered bedrock range from less than 1 m to between 4.5 m and 9 m in Zone 3 and between 10 m and 15 m in Zone 4. Characteristic rock types include mudstones, siltstones, shales, sandstones, coals and seat earths. The “Clay Cross Soft” and “2nd Ell” coal seams of local significance subcrop in the west and east of the site respectively with thick sandstone units along the western boundary. These rocks form the western limb of a large syncline which plunges gently towards the east northeast at 6° - 8°.

Significant geological deformation has led to the development of two major faults sets trending northeast-southwest and north northwest-south southeast. The area surrounding the site is on the western edge of the Derbyshire Coalfield and has been subject to underground and opencast mining, some of which occurred within the site boundary. The Coal Authority records show a number of shafts and adits across the site which have been filled and capped over the years to varying engineering standards.

3.5.2 HYDROGEOLOGY

During the 2000/2001 site investigations, groundwater monitoring wells were installed at varying depths to monitor variations in hydraulic head between groundwater within the unconsolidated superficial deposits and the underlying consolidated bedrock. Nineteen monitoring wells were completed in the made ground, nine in the alluvium/weathered coal measures and 77 in the Lower and Middle Coal Measures.

3.6 NATURE AND EXTENT OF CONTAMINATION

Soil and groundwater from the site were analysed for a range of organic and inorganic species typically associated with a former coke works (Department of the Environment, 1999). A complete list of analytes and assessment levels used is provided in Table 3.2 and includes total organic carbon (TOC) in soil and heavy metals, cyanides, PAHs, phenols, diesel range organics (DROs), petrol range organics (PROs) and volatile organic compounds (VOCs) in soil and groundwater.

The contamination is heterogeneously distributed throughout the soil, and originated from product spills, leaking tanks and pipes, demolition activity and poor waste management over many decades. In the absence of any regulatory remediation criteria at the time of the site investigations, a tier 2 generic quantitative risk assessment was undertaken, in accordance with DETR guidelines for environmental risk assessment and management (2000). This was used as an initial screen to identify contaminants of concern for a detailed quantitative risk assessment. For the purpose of the assessment, the end use of the site was assumed as public open space and the assessment was carried out using generic criteria.

Table 3.2: Analytical determinands for soil and groundwater analyses

Determinand	Soil	Assessment level	Groundwater	Assessment level
		(mg/kg)		(µg/l)
pH	✓	NA	✓	5.5-9.5
Electrical conductivity	X	NA	✓	1,500 ^a
Arsenic	✓	40	✓	50
Cadmium	✓	15	✓	5
Chromium (total)	✓	1,000	✓	50
Chromium (hexavalent)	✓	None ^b	✓	None ^b
Copper	✓	130	✓	3,000
Lead	✓	2,000	✓	50
Mercury	✓	20	✓	1
Nickel	✓	70	✓	50
Selenium	✓	6	✓	10
Zinc	✓	300	✓	5,000
Iron	✓	None ^b	✓	200
Asbestos ^c	✓	-	X	NA
Ammoniacal nitrogen	✓	None ^b	✓	500
Total cyanide	✓	250	✓	50
Free cyanide	✓	100	✓	None ^b
Thiocyanate	✓	50	✓	20
Sulphate	✓	0.2	✓	250 ^d
Sulphide	X	250	✓	50
SEM	✓	5,000	X	NA
Total PAHs ^e	✓	1,000	✓	0.2
Phenols (speciated)	✓	5	✓	10
TOC	✓	None ^b	X	None ^b
PRO	✓	1,000	✓	10
DRO	✓	1,000	✓	10
VOC	✓	None ^b	✓	None ^b

Adapted from: Babbie (2001)

Notes:

a. Conductivity in microsiemens (µS)

b. No assessment level

c. Selected samples

d. mg/L

e. Total PAHs is equal to the sum of the following individual PAHs: acenaphthene, anthracene, acenaphthylene, benz(a)anthracene, chrysene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-c,d)pyrene, naphthalene, phenanthrene, pyrene.

NA = Not available

3.6.1

SOIL CONTAMINATION

Table 3.3 provides the parameters which exceeded the generic soil assessment levels across the six zones. The table shows that contamination was most prevalent in Zone 3 the main plant area and Zone 4 containing the High Level Stocking Area (HLSA) and waste tip. Least contamination was found in Zone 1, the area south of Mill Lane, and Zone 5, the Low Level Stocking Area (LLSA)/scrubland.

Table 3.3: Parameters exceeding the generic soil assessment levels

Determinand	Zone					
	1	2	3	4	5	6
Arsenic	✓	✓	✓	✓	✓	✓
Cadmium			✓			
Copper	✓	✓	✓	✓		
Lead			✓			
Mercury			✓			
Nickel		✓	✓	✓	✓	✓
Selenium				✓		
Zinc		✓	✓	✓		✓
TNVA		✓	✓	✓		✓
Total cyanide			✓	✓		
DRO	✓	✓	✓	✓	✓	✓
PRO			✓	✓		
Total PAH		✓	✓	✓		✓
Total phenol			✓	✓		✓
Thiocyanate			✓	✓		
SEM		✓	✓	✓	✓	✓

Note: ✓ indicates exceedance of generic soil assessment level

Source: Babbie (2001)

In Zone 3, total PAH levels were above the assessment level of 1,000 mg/kg in approximately 9 % of samples with a maximum concentration of 7,018 mg/kg. Generally, the remaining samples were at concentrations below 200 mg/kg. Approximately 6 % of total phenol levels were above the assessment level of 5 mg/kg with a maximum concentration observed of 201 mg/kg. The remaining samples were generally below 0.2 mg/kg. DROs were above the assessment level of 1,000 mg/kg in approximately 13 % of samples with a maximum concentration of 17,556 mg/kg. PROs were above the assessment level of 1,000 mg/kg in only one sample where the concentration was 2,306 mg/kg.

As previously mentioned contamination levels were much higher in Zone 4 which contained the former waste tip. Total PAH levels were above the assessment level of 1,000 mg/kg in approximately 50 % of samples with a maximum concentration of 74,611 mg/kg. The remaining samples were at concentrations below 70 mg/kg. Approximately 65 % of total phenol levels were above the assessment level of 5 mg/kg with a maximum concentration of 15,413 mg/kg. The remaining samples were generally below 1 mg/kg. DROs were above the assessment level of 1,000 mg/kg in approximately 60 % of samples with a maximum concentration of 12.2 %. PROs were above the assessment level of 1,000 mg/kg in approximately 17 % of samples with a maximum concentration of 9,617 mg/kg.

3.6.2

GROUNDWATER CONTAMINATION

Table 3.4 gives a qualitative illustration of groundwater quality upgradient, below and downgradient of the site. Background water quality is poor, like other groundwaters from the Coal Measures, having elevated levels of Fe, ammonia, thiocyanate, PAHs, DROs and PROs with respect to assessment levels. Quality deteriorates further as it passes beneath the site and there is evidence of a contaminant plume migrating downgradient of the site towards the River Rother.

Table 3.4: Parameters exceeding the generic groundwater assessment levels

Determinand	Groundwater location		
	Background	Beneath site	Downgradient
pH		✓	✓
Cadmium		✓	
Chromium		✓	
Iron	✓	✓	✓
Lead		✓	✓
Mercury		✓	
Nickel		✓	✓
Selenium		✓	✓
Thiocyanate	✓	✓	✓
Total cyanide		✓	✓
Ammoniacal nitrogen	✓	✓	✓
Sulphate		✓	✓
Sulphide	✓	✓	✓
Phenol		✓	✓
Total phenol		✓	✓
Total PAH	✓	✓	✓
DRO	✓	✓	✓
PRO	✓	✓	✓

Note:

Concentrations exceeding assessment levels indicated by ✓

Source: Babbie (2001)

4. LABORATORY TREATABILITY STUDIES

4.1 INTRODUCTION

A first step in assessing whether a technology is a viable remediation option is to carry out laboratory-based treatability studies on representative contaminated material. Bioremediation is now such a frequently used remediation technology that many experienced practitioners are able to assess the viability of a site for full-scale treatment often on the basis of ground characterisation data and only simple slurry biodegradation tests. However, on this occasion it was felt that the following laboratory-based activities were required due to the variability of the material to be used in the trial:

- Characterisation of test materials
- Slurry biodegradation tests
- Solid phase bioreactor tests
- Bacterial counting

The first three of these are described in more detail in the following sections.

4.2 CHARACTERISATION OF TEST MATERIALS

Representative material samples from the waste tip and plant area were assessed for texture, contaminant type and concentration and potential for bioremediation including some tests for improving material structure.

The particle size distribution and physical characteristics of the contaminated materials are provided in Tables 4.1 and 4.2.

Table 4.1: Particle size distribution for material samples from the waste tip and plant areas

Sample	Gravel % (> 2 mm)	Sand % (63 µm - 2 mm)	Silt % (< 63 µm)
Waste tip TP344	52	34	14
Waste tip 13A	43	27	30
Plant area 311	62	18	20
Plant area 108A	59	35	6
Overall	40 – 60	20 – 35	5 – 30

Adapted from DEC NV (2001)

Table 4.2: Physical characteristics of the material samples

Sample	> 2 mm	63 µm - 2 mm	< 63 µm
Waste tip TP344	gravel, slags, coal	coal content high (LOI ^a = 13.5 %)	black (coal)
Waste tip 13A	mix of ash, gravel, coal	brownish sand with low mechanical strength (clay granules)	red-brownish, low settling velocity, high clay content
Plant area 311	mudstone mixed with coal and tar particles	fine mudstone particles with low mechanical strength	black (coal)
Plant area 108A	High slag/ash content	black (ashes+coal) LOI = 5 % - 7 %	black (coal)

Note:
a. loss on ignition

Adapted from DEC NV (2001)

Two samples were taken from both the waste tip and the plant area. The initial chemical composition for selected analytes of the four material samples is shown in Table 4.3.

Table 4.3: Selected chemical analyses of the material samples used in the slurry biodegradation tests (mg/kg)

Contaminant	Waste tip TP344	Waste tip 13A	Plant area 108A	Plant area 311
Naphthalene	8,100	5,400	4,200	1,600
Benzo(a)pyrene	350	160	370	840
Total PAH	23,860	11,936	9,000	14,231
BTEX	130	38	4.7	95
Phenol index ^a	7.3	550	0.57	6.7
Mineral oil	40,000	3,100	4,800	28,000

Note:

Source: DEC NV (2001)

a. phenol index = sum of water vapour soluble phenols. Generally gives phenol concentration.

4.3

LABORATORY-BASED TESTS

Laboratory tests included a slurry phase biodegradation test, a solid phase bioreactor test and bacterial counting. Summaries of the slurry biodegradation test and solid phase bioreactor test methods are summarised in Table 4.4 and are described in more detail in sections 4.3.1 and 4.3.2.

Table 4.4: Test methods for assessing the bioremediation potential of contaminated material

Test Method	Scale	Justification
Slurry phase biodegradation test	50 g	Select optimal treatment conditions Determine indicative biodegradation rates and achievable endpoints for slurry phase and solid phase treatment Measure respiration and contaminants in material
Bioreactor test	50 kg	Evaluate technical feasibility of solid phase bioreactor treatment Determine biodegradation kinetics on medium scale Measure contaminants in material and off-gas

Source: DEC NV (2001)

4.3.1

SLURRY BIODEGRADATION TESTS

4.3.1.1

Introduction

The slurry biodegradation test is a simple screening test used to measure bacterial respiration and to determine whether the contaminants within existing site materials can be biodegraded. Aerobic biodegradation occurs quickly when material is treated as an aerated and agitated aqueous slurry due to the mobilisation and mass transfer of contaminants into the aqueous phase. The test involved taking a 50 g sample and subjecting it to agitation and aeration to form a watery suspension.

The uptake of oxygen (O₂) and the production of carbon dioxide (CO₂), which occur during microbial activity, provide evidence that changes in contaminant levels can be attributed to biodegradation. The test can be carried out under different conditions with the addition of various nutrients, chemical additives or co-substrates, or inoculation with bacteria, as well as on controls using uncontaminated and sterile materials.

4.3.1.2

Objectives

The objectives of the slurry biodegradation tests were to:

- Measure the amount and rate of respiration by bacteria in the material

- Select optimal treatment conditions
- Determine maximum biodegradation rates and endpoints
- Determine any reduction in contaminant levels during the test

4.3.1.3 Methodology

The slurry biodegradation tests were carried out on four different material samples consisting of TP344 and 13A from the waste tip and 108A and 311 from the plant area. Fifty grams of material from each sample were screened to < 5 mm and placed in 500 ml dark glass flasks which were sealed with a silicone septum to prevent volatilisation of contaminants. The test was carried out over a treatment period of 17 weeks. The test was performed under aerobic conditions so it was necessary to ensure that sufficient O₂ remained in the flask. The gas phase in the flasks was regularly sampled through a self-sealing septum and analysed for O₂ and CO₂ using a gas chromatograph / thermal conductivity detector. O₂ depletion and CO₂ generation were used as indicators of aerobic biodegradation. During the test each flask was agitated on a shaking table.

The tests on each of the samples were carried out in duplicate under a range of conditions including: abiotic and biotic controls; different nutrient mixtures; and the addition of a bacterial inoculum. Each condition was identified by a letter code (see Table 4.5). The inoculated condition involved the addition of activated sludge from an effluent plant at a nearby coking works.

Table 4.5: Nutrient code and composition used in the slurry biodegradation tests

Code	Condition	Description
B	Abiotic (dead) control	No additives except for sodium azide as a microbial inhibitor
C	Biotic (live) control	No additives except for pH adjustment when necessary
NP	Nutrient mixture 1	Ammonium chloride and pyrophosphate, dosed to achieve a C:N:P ^a ratio = 100/10/1
T	Nutrient mixture 2	A commercial nutrient formulation
NPI	Nutrient mixture 1 + bacterial inoculum	Nutrient mixture 1 supplemented with a bacterial inoculum: an activated sludge sample from a nearby coking plant

Note:

a. nutrient ratio of carbon to nitrogen to phosphorus

Source: DEC NV (2001)

Degradation kinetics for different test treatments were monitored weekly by measuring the uptake of O₂ and the formation of CO₂ in the headspace of the flasks. The slurries were aerated whenever the concentration of O₂ dropped below a threshold value of 10 % in the flask headspace. At the end of the incubation period, the slurries for each treatment were combined and centrifuged to separate solid from liquid phases. The solid phase was analysed for a range of parameters including total PAHs, phenols, BTEX and mineral oil. The liquid phase was analysed for phenols and BTEX in selected samples.

4.3.1.4 Conclusions

No particular treatment was more successful than another in promoting biodegradation. Waste tip samples 13A and TP344 and sample 108A from the plant area demonstrated the greatest potential for biodegradation. Biodegradation was still occurring in both waste tip samples when the test stopped, so it was not possible to say what the final end concentrations might have been. Respiration data for the plant area (108A) sample showed that the bioavailable and biodegradable fraction was nearly exhausted at the end of the test. Total PAH concentrations had reduced. Despite this reduction a significant contaminant concentration remained. The waste tip (TP344) sample only showed naphthalene degradation but this was possibly due to the high initial concentrations. Waste tip sample

(13A) had much lower initial naphthalene concentrations and it did show degradation of three ring PAHs after eight weeks.

The results from the slurry biodegradation tests were used to select the waste tip (TP344) and plant area (108A) samples for further testing in bioreactors. Both samples exhibited higher respiration activities compared to those for the waste tip (13A) and plant area (311) samples. In addition, the waste tip (TP344) sample had higher initial contaminant concentrations compared to the waste tip (13A) sample, illustrating the inherent heterogeneity in the source material.

4.3.2 SOLID PHASE BIOREACTOR TESTS

4.3.2.1 Introduction

A solid phase bioreactor test involves placing a sample of contaminated material (70 % to 85 % dry solids) in a bioreactor in which rotating paddles mechanically mix the material and keep it aerated, in a similar manner to the mixing that occurs in a field-scale biopile. Soil and made ground material are naturally heterogeneous and large sample bioreactors can help overcome the limitations inherent in using only the very small amounts of material that are used in slurry phase biodegradation tests. The bioreactor provides a controlled environment in which the internal atmosphere can be analysed for temperature, VOCs, CO₂ and O₂. This can help verify that any reduction in the concentration of the contaminants is due to biodegradation. The biodegradation rate is enhanced relative to that which occurs in a slurry biodegradation test because the bioavailability is improved by breaking up the soil aggregates during the slurring process and therefore allowing an estimate to be made of the time needed for biodegradation to occur in a field-scale biopile.

4.3.2.2 Objectives

The objectives of carrying out the solid phase bioreactor tests were to:

- Scale up the chemical and physical processes that occurred in the slurry biodegradation test by using much larger sample sizes (3 kg to 50 kg)
- Determine the biodegradation kinetics at a larger scale than occurred in the slurry biodegradation test
- Evaluate the technical feasibility of solid phase treatment

Whilst a bioreactor test can be performed successfully in 28 days, if PAHs with more than three rings are predominant, a much longer test period of up to 80 days is recommended.

Following a bioreactor test, an analysis of the material samples and gas phase results allows a mass balance to be calculated to demonstrate the efficiency of biodegradation in reducing the initial contaminant concentration.

4.3.2.3 Methodology

After screening to remove material larger than 75 mm, between 20 kg and 30 kg of the waste tip (TP344) and plant area (108A) samples were treated in separate solid phase bioreactors each with a capacity of 50 kg of dry matter. The tests took place over 80 days between July 3rd and September 21st 2001. Sample characteristics and chemical analyses are described in Tables 4.6 and 4.7.

Table 4.6: Characteristics of samples used in the bioreactor tests

Sample	No. of samples	Total wet wt. (kg)	Total wet wt. after screening (kg)	% wt. loss on ignition 105 °C	% wt. loss on ignition 550 °C
Plant area 108A	2	44.5	26.6	16.73	20.7
Waste tip TP344	1	44.9	21.87	25.5	20.7

Source: DEC NV (2001)

Table 4.7: Chemical analyses at time t=0 of the material samples used in the bioreactor tests (mg/kg dry weight)

Contaminant	Concentration (mg/kg)	
	Waste tip TP344 t=0 days	Plant area 108A t=0 days
Naphthalene	5600	1600
Benzo(a)pyrene	Value not known	380
Total PAH	27848	6112
BTEX	92	NA
Phenol index	12	0.57
Phenols	<0.5	<0.5
Mineral oil	29000	6300

Source: DEC NV (2001)

An aqueous solution of a commercial nutrient formula was added to each screened material sample to obtain a C:N:P ratio of 100/10/1 before each sample was placed in the bioreactor. The contents of the reactor vessels were mixed every three hours for 15 minutes and the bioreactors were aerated continuously, although this was suspended for a short period of time each week to allow the respiration activity in the bioreactor to be measured.

The waste tip (TP344) sample would often aggregate into large pieces up to 10 cm in diameter when stirred in the bioreactor; this may have been due to the high tar content of the sample. Water was added to the bioreactor at day 34 of the test period to mitigate this. Similar problems did not occur with the plant area (108A) sample.

The atmosphere within each bioreactor was analysed for CO₂ and O₂ to monitor respiration activity during the test. Off-gas emissions consisting of VOCs were analysed with photoionisation detectors (PID). Hydrogen cyanide and benzene were measured with specific electrochemical analysers and Draeger CMS-chip analysers respectively.

Approximately 500 g of solid phase material were collected for analysis through an airlock on the bioreactor at intervals of between 3 days and 18 days. Residual contaminant levels in this material were measured frequently over the 80 days of the test for the following parameters: pH, weight loss on ignition at 105 °C and 550 °C, total PAHs, TPH, phenols, BTEX, as well as bacterial counts at the beginning and end of the test period.

4.3.2.4 Conclusions

The bioreactor tests confirmed the results obtained for the waste tip (TP344) and plant area (108A) samples during the slurry biodegradation tests. During the bioreactor tests volatilisation was significant for the waste tip (TP344) sample where 10 % of the contaminant removal was estimated to be due to volatilisation. It should be remembered that volatile contaminants will be in equilibrium between the headspace and the sorbed phase. This does not reflect the condition in biopiles where volatile contaminants released from soil during the mixing process are lost to the atmosphere.

The slurry and bioreactor laboratory tests demonstrated that both the waste tip and the plant area materials were suitable for bioremediation and therefore those materials were used in the field-scale biopile.

5. TECHNOLOGY DEMONSTRATION SUPPORT ISSUES

5.1 INTRODUCTION

This section discusses supporting issues associated with the design, construction and operation of the biopile including:

- Regulatory approval and compliance
- Contract agreement and health and safety
- Method statement
- Sampling plan
- Laboratory analytical methods
- Quality assurance / quality control
- Role of CL:AIRE

5.2 REGULATORY APPROVAL AND COMPLIANCE

The remediation trial was undertaken by DEC NV. The local authority confirmed that the trial would not be subject to any requirement for planning permission. The Environment Agency (EA) confirmed that the trial satisfied the requirements for an exemption from waste management licensing under the Waste Management Licensing Regulations 1994. The exemption assumed compliance with the then draft EA document "Guidance on Trial Waste Management Operations", which restricted the volume of material subject to treatment to 500 m³.

5.3 CONTRACT AGREEMENT AND HEALTH AND SAFETY

An Official Journal of the European Community (OJEC) notice was advertised on the 26th August 2000 inviting expressions of interest to undertake remediation trials at the site. Tender documents were then issued, submissions assessed and a contract for solid phase bioremediation was awarded in late spring 2001 to DEC NV, who opted to use biopile technology for the trial.

DEC NV were also awarded a contract for slurry phase remediation on tarry sediments from Lagoon 2 on the site. The results from the laboratory tests on this material showed that bioremediation was not a viable option and the slurry phase field demonstration was cancelled with the agreement of EMDA.

The construction of the field biopile was undertaken under Construction (Design and Management) Regulations 1994 (CDM). Under CDM the designers (Jacobs Babbie) were required to develop the pre-tender health and safety plan (HSP) for the biopile trial. DEC NV as the contractor, developed method statements and risk assessments taking into account issues raised in the pre-tender HSP.

Principal parties covered under CDM were:

- Client – East Midlands Development Agency
- Designer – Jacobs Babbie
- Planning Supervisor – Jacobs Babbie
- Principal Contractor – Turner and Townsend Health, Safety and Environment
- Contractor – DEC NV
- Sub-contractor – VHE Construction plc

Turner and Townsend developed a construction phase HSP. The plan included general site procedures, information for contractors, risk assessments, information and training for site personnel and monitoring arrangements.

5.4 METHOD STATEMENT

The work plan for the bioremediation trials was agreed jointly between DEC NV, Jacobs Babbie and EMDA and comprised a detailed method statement. This outlined a phased approach to the remediation trials consisting of laboratory-based slurry biodegradation tests and bench-scale bioreactor tests followed by a field-based biopile.

5.5 SAMPLING

Sampling was undertaken to characterise the contaminated material before, during and after both the laboratory-based tests and the field-scale biopile.

5.5.1 SITE SAMPLING

Only limited information on site characterisation was available during the design phase of the biopile field trial. Therefore, samples of contaminated material were collected by Jacobs Babbie in April 2001 from parts of the site considered to be most contaminated using an excavator and stored on site in drums for use in the laboratory phase trials. DEC NV received the following material samples from these drums for the laboratory tests.

- Sample 311 from the plant area (Zone 3)
- Sample 108A from the plant area (Zone 3)
- Sample TP344 from the waste tip (Zone 4)
- Sample 13A from the waste tip (Zone 4)

5.5.2 BIOPILE SAMPLING

The field-based biopile was constructed from 200 m³ of contaminated source material and was divided into two sections: one was constructed of material from the tip and one from plant area material. For sampling purposes each section was divided into zones A, B and C (see Figure 5.1).

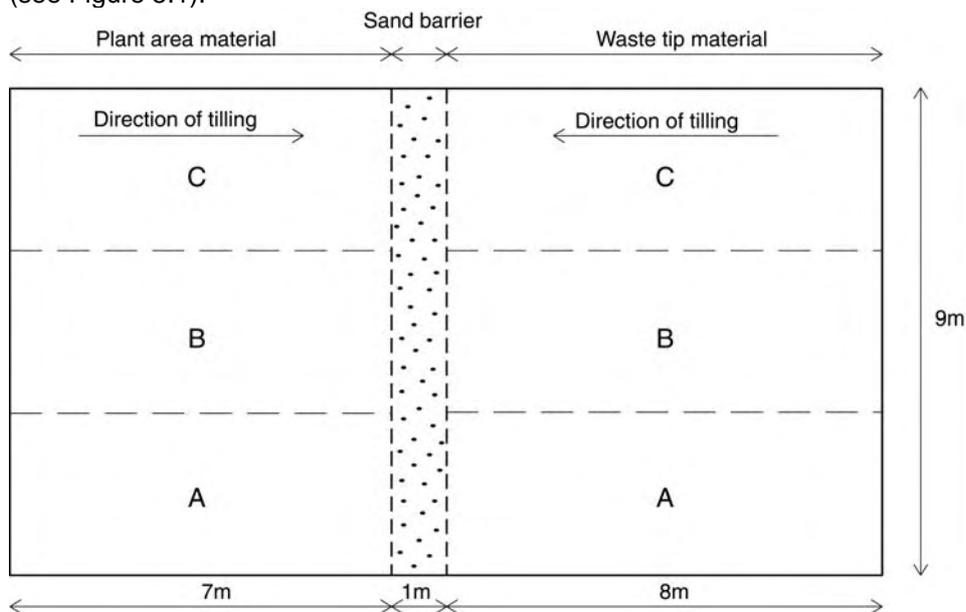


Figure 5.1: Plan view of the biopile illustrating sampling zones

During mixing of the biopile, a trowel was used to collect material samples at between five and nine random locations at different heights in the exposed working face of each zone.

The samples from each zone were then combined to produce a single composite sample which was representative of each zone from the waste tip and plant area sections. A total of six composite samples (2 sections x 3 zones) were then sent for laboratory analysis. After two months of operation, duplicate samples were sent to a second laboratory for analysis. During mixing, volatile emissions from the biopile (BTEX, VOCs, phenols and naphthalene) were measured with Draeger tubes at the surface of the biopile and at the outlet to the activated carbon filter (see section 6).

5.6 LABORATORY ANALYTICAL METHODS

The laboratory-based tests were undertaken at the laboratories of Vito (Flemish Institute for Technological Research), DEC NV's partner organisation, located in Mol, Belgium. All material and liquid samples collected during the laboratory tests and from the field-based biopile were analysed by ALcontrol Laboratories in the UK and the Netherlands. All results are reported on a dry weight basis.

Vito were responsible for on-site gaseous analyses (e.g. CO₂, O₂, VOCs) during the laboratory tests and field-based biopile demonstration. DEC NV analysed selected samples for basic physical parameters such as grain size.

ALcontrol undertook organic and inorganic analyses using the following approved techniques for the parameters listed below:

•	PAHs	High Performance Liquid Chromatography Ultra Violet (HPLC UV)
•	Total cyanide	Distillation/spectrophotometry
•	Phenol index	4-amino antipyrine method NEN 6670 Standard
•	Phenol	Gas Chromatography Mass Spectrometry (GCMS) or HPLC
•	Cresols	HPLC
•	C2-alkylphenols	HPLC
•	Total BTEX	Headspace Gas Chromatography Flame Ionisation detection (GC-FID)
•	Mineral oil	GC-FID
•	Phosphate	Spectrophotometric
•	Total nitrogen	Distillation followed by spectrophotometric
•	Kjeldahl nitrogen	Distillation followed by spectrophotometric
•	pH	Electrode / meter
•	Organic matter	Combustion then infrared
•	Dry matter	Gravimetric
•	TOC	Combustion then infrared

DEC NV undertook gaseous analyses for the parameters below:

•	Off-gas: VOCs	Photoionisation detection (PID) instrument
•	Off-gas: cyanide	Specific electrochemical analyser
•	Off-gas: benzene	Draeger CMS-chip analyser

PAH samples were initially sent to the ALcontrol laboratory in Chester for analysis using GCMS. After two months, duplicate samples were also sent to the ALcontrol laboratory in Rotterdam for analysis using HPLC/UV. Due to inconsistencies, DEC NV decided to use the latter for the purpose of their trial and conclusions. All other samples from the Avenue Coking Works were analysed by ALcontrol in Chester.

5.7 QUALITY ASSURANCE / QUALITY CONTROL (QA / QC)

QA/QC for laboratory testing and field sampling is discussed below.

5.7.1 LABORATORY QA / QC

Both the UK and Dutch ALcontrol laboratories are accredited under their respective national schemes: in the UK this is the United Kingdom Accreditation Scheme (UKAS) and in the Netherlands it is the Dutch Council for Accreditation (AvR).

Laboratory analysis complied with industry best practice for analytical methods. All analyses were run against standard solutions, usually with a five point calibration (all inorganics), plus internal standardisation for most organic analyses. GCMS analyses included surrogate standards and multi-component standards as a check on recovery and performance.

5.7.2 FIELD QA / QC

When samples were being taken from the biopile, the trowel was brushed clean between each episode of sampling to avoid cross contamination.

5.8 ROLE OF CL:AIRE

CL:AIRE was invited by EMDA to assess the results of the field trials. The method statement describing the trial methodology was evaluated and approved outside the normal CL:AIRE process. The contractor was required to write a report following the CL:AIRE format. CL:AIRE personnel made several visits to the site during the trial to observe how the trial was being conducted.

6. BIOPILE CONSTRUCTION AND DEMONSTRATION

6.1 INTRODUCTION

This chapter describes the biopile field trial which was performed between July 9th 2001 and October 8th 2001.

6.2 SHED DESIGN AND CONSTRUCTION

The biopile was contained within a purpose-built weatherproof enclosure, which was constructed on the concrete base of the High Level Stocking Area (HLSA) located within Zone 4 of the site (see Plate 6.1).



Plate 6.1: View of the biopile shed

The use of an enclosed shed design allowed greater control over material moisture levels, the rate of injection of air into the biopile and also allowed gaseous emissions to be monitored. The shed was equipped with an air extraction system consisting of a network of polyvinyl chloride (PVC) pipes. Air, including any gaseous emissions from the biopile, was collected and treated through an activated carbon filter before being discharged to the atmosphere (see Plate 6.2).

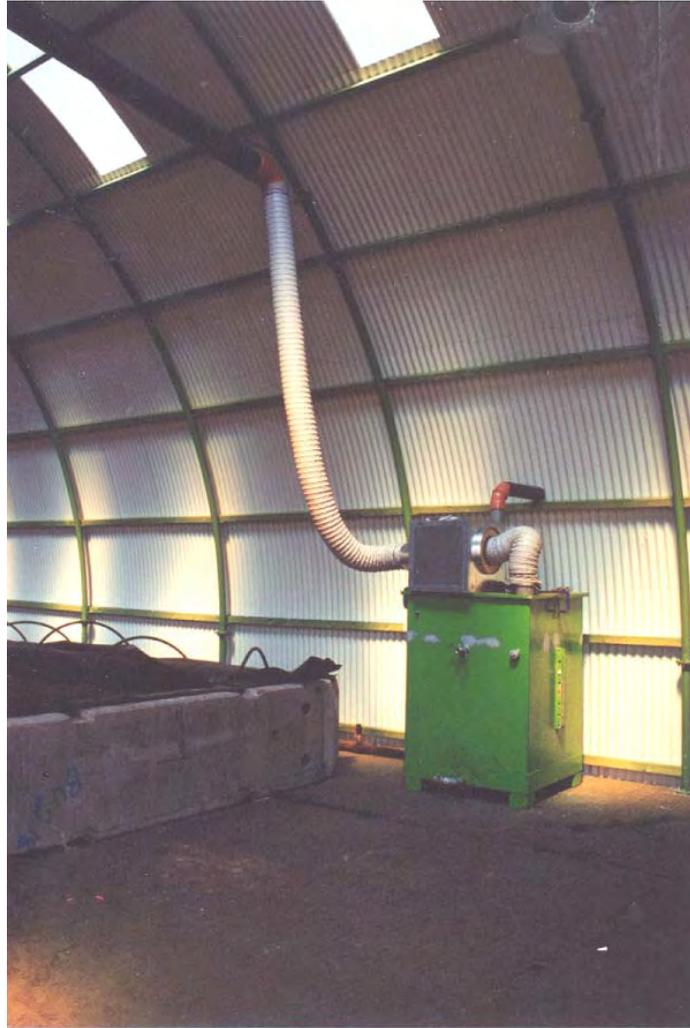


Plate 6.2: Activated carbon filter and the leachate collection sump

6.3

BIOPILE DESIGN AND CONSTRUCTION

The base of the biopile was constructed of a 0.5 m thick layer of coarse drainage sand overlying a 0.3 mm thick low density polyethylene (LDPE) construction liner. Leachate drains and air injection pipes were installed in the sand layer. Approximately 200 m³ of contaminated material was taken from stockpiles of waste tip and plant area material and was placed in layers on top of the basal sand layer to a thickness of approximately 1 m using a mechanical excavator. Stockpiled material had been screened to less than 75 mm. The roof of the shed was approximately 5 m above the surface of the biopile. The different source material in the biopile was segregated: one side of the biopile was constructed of waste tip material and the other side of plant area material, separated by 1 m wide by 0.5 m high vertical barrier of clean sand. The material from each source was divided into zones A, B and C for sampling purposes. Zone A was located closest to the main air distribution pipe or manifold, B was located in the middle of the biopile and C was located at the end of the aeration pipes (see Figure 5.1). A plan view and cross section through the biopile are shown in Figures 6.1 and 6.2. A view of the biopile after construction is shown in Plate 6.3.

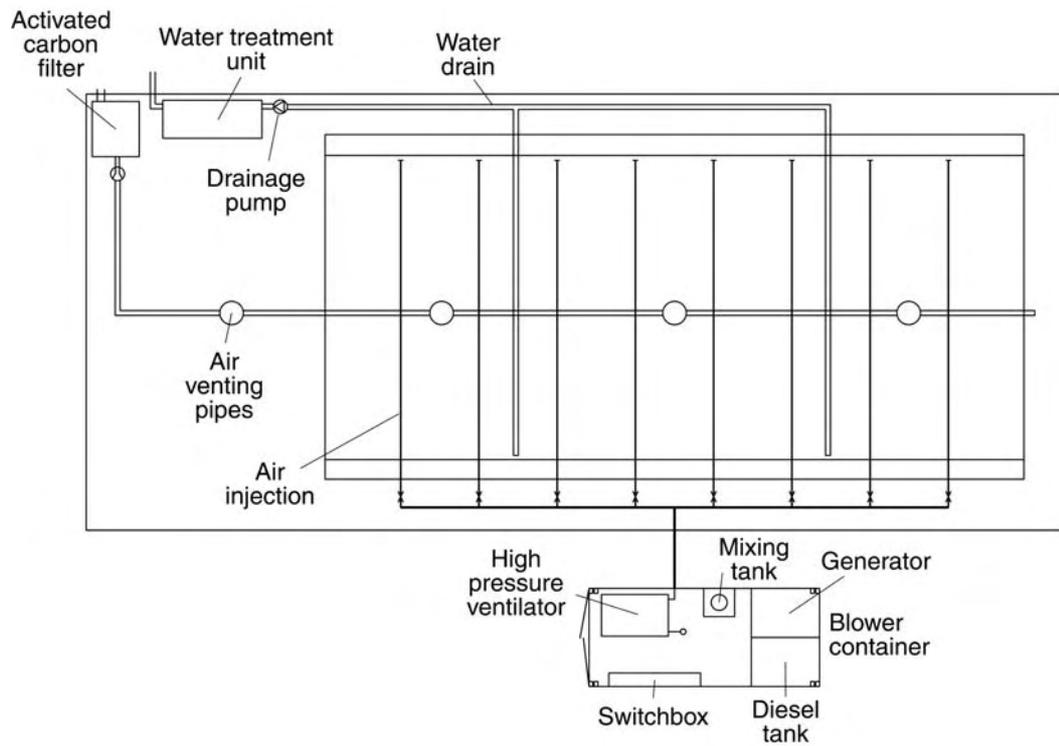


Figure 6.1: Plan view of the biopile

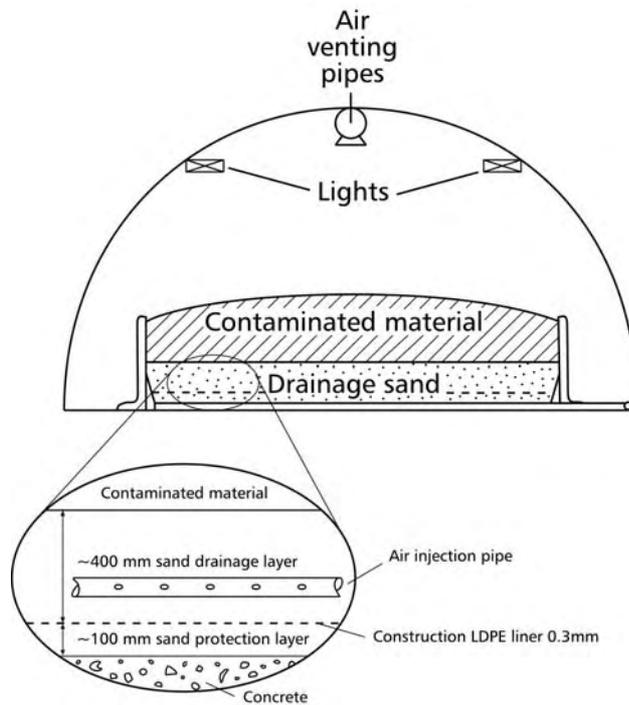


Figure 6.2: Biopile hut cross section

A sump pit approximately 1 m^3 in volume and lined with high density polyethylene (HDPE) was constructed at one corner of the biopile to collect any leachate that might be generated. A submersible pump was installed in the sump so that any leachate could be pumped to a

small mobile water treatment unit. The water treatment unit consisted of an oil-water separator and an activated carbon filter. The treatment unit was judged to be most efficient for dealing with the few cubic metres which were expected to arise daily from the biopile. However, during operation of the biopile no leachate accumulated in the sump.

The air injection for the biopile was supplied using a blower unit located within a 6 m long container located outside the shed. The container also housed a 25 kVA diesel generator, a 2000 litre diesel tank and a water-nutrient mixing tank. The air was pre-heated to a temperature of 30 ± 5 °C before being injected into the biopile at a flow rate of $1 \text{ m}^3/\text{h}/\text{m}^2$.



Plate 6.3: The biopile at the start of the trial showing plant area (background) and waste tip (foreground) material separated by a sand layer

6.3.1 CHEMICAL CHARACTERISTICS OF TEST MATERIALS

The plant area and waste tip stockpiles consisted of contaminated material collected from the vicinity of boreholes 311 and 13A respectively.

The material was mixed in each source area using a backhoe, working from the edge of the biopile towards the clean sand layer separating the two source areas. During this operation water was often sprayed onto the material to keep the biopile moist and to help suppress dust.

The waste tip material used in the field-scale trial was significantly less contaminated than the material from the same source used in the laboratory tests (see Chapter 4) and this illustrates the heterogeneity of the material. The total PAH concentrations for waste tip material TP344 and 13A that were used in the laboratory tests were 23,860 mg/kg and 11,936 mg/kg compared with a mean concentration of 6,400 mg/kg for the material from the field-scale trial used to construct the biopile. BTEX, phenol and mineral oil concentrations were also less than those in the material used for the laboratory tests.

The plant area material used in the field trial had similar contamination levels to the material used for the laboratory tests. The mean total PAH concentration for the plant area materials 108A and 311 was 11,615 mg/kg compared with an mean concentration of 13,916 mg/kg for the plant area material used to construct the biopile. BTEX, phenol and mineral oil compounds were at similar levels to those in the material used for the laboratory tests.

The extent of VOC loss from both plant area and waste tip material during the construction of the stockpiles and biopile is not known.

The contaminants of major concern in both source materials were PAHs. Both materials also showed slightly elevated levels of total phenols and mineral oil. The plant area material contained significant levels of VOCs and the waste tip material contained slightly elevated levels of As, Cu, Pb and Zn.

6.4

METHODOLOGY

After placement in the appropriate section of the biopile, the contaminated material was pre-treated with a commercial nutrient formula to achieve a C:N:P ratio of 100/10/1. The nutrient and contaminated material were mixed using an excavator following which three composite material samples were collected from each section of the biopile and sent for analysis. This was taken to be the time $t=0$ sampling just before bioremediation commenced. The biopile was mixed every two weeks and during this operation samples of the material were collected for analysis. From week 2 onwards, the biopile was sprayed with water three times a week to keep the moisture content at approximately 70 % (Plate 6.4).



Plate 6.4: Spraying the biopile with water

During the trial the average air temperature in the shed was 25 °C. After 6 weeks some of the plant area material in Zone C was mixed with sludge from a nearby effluent treatment plant, located on a coke works, in approximate proportions of 10 litres of sludge to 1 tonne of material. This was to try and replicate the results from the slurry biodegradation test in a similar composition plant area sample. During this test the most promising biodegradation results were those achieved when the sample was mixed with nutrient mixture 1 and inoculated with sludge from the same wastewater treatment plant.

After the biopile had been operating for 10 weeks, sampling indicated that both sections of the biopile were becoming depleted in nutrients. Urea and diammonium phosphate fertilisers were added to restore the C:N:P ratio to 100/10/1. The choice of these nutrients was based on the results obtained during the earlier laboratory tests. After discussions between DEC NV and EMDA it was agreed to stop the mixing operation of the biopile after 13 weeks, although further sampling continued until no further degradation was observed.

6.5

CL:AIRE OBSERVATIONS

Personnel from CL:AIRE visited the site during the trial to ensure that operations were being carried out in accordance with the method statement.

7. PERFORMANCE EVALUATION

7.1 INTRODUCTION

The current best practice approach to the management of contaminated land in the UK involves a site specific risk assessment which comprises an assessment of the risks to human health and the environment from particular contaminants using a source-pathway-receptor approach. As part of an agreed remedial strategy, particular contaminants may require treatment to reduce their concentration or availability to an “acceptable level”. The drivers for a remedial strategy usually include:

- Redevelopment of the site through the planning regime
- A statutory designation of “contaminated land” under Part IIA of the Environmental Protection Act 1990
- Owner / occupier requirements

It therefore follows that performance evaluation of the capacity of a remedial technique (in this case a biopile) to reduce critical contaminants to an acceptable target level is necessary.

7.2 CONTAMINANTS OF CONCERN

The following contaminants, all associated with coke works and other carbonisation plants at the Avenue Coking Works site, are potentially capable of undergoing bioremediation.

- PAHs
- BTEX compounds
- Phenols

The effect of bioremediation on mineral oils and VOCs in the biopile was also considered.

7.3 TARGET LEVELS

Whilst a site investigation at the Avenue site had taken place, a formal quantitative site risk assessment had not been completed and remedial targets for critical contaminants had not been agreed. In the spring of 2003, following the completion of quantitative risk assessments, preliminary soil remedial targets for residential and open space land use were calculated by Jacobs Babbie. These values are used in this chapter to assess the performance of the biopile and its potential as a remedial solution at the site. Residential target levels were calculated using the Contaminated Land Exposure Assessment (CLEA) Model (DEFRA, 2002) and open space targets using the ‘SNIFFER Methodology’ (SNIFFER, 2000). Toxicological data for benzo(a)pyrene were taken from the CLEA Research and Development Publication TOX 2 (DEFRA, 2002) and other toxicological data used in the modelling were sourced from the literature. Both target levels for human health and controlled waters were only indicative and have since been reviewed following finalisation of the intended land use and proposed restoration of the site.

Target levels were calculated for naphthalene, benzo(a)pyrene, benzene and total phenols. These compounds were used as marker compounds to represent other related contaminants that exhibit similar chemical behaviour and risks to human health due to toxicity and/or prevalence on site.

7.4 BIOPILE PERFORMANCE

7.4.1 SUMMARY OF RESULTS

Selected analytical results from the biopile field trial along with target levels are shown in Table 7.1. The concentrations given are the mean values from zones A, B and C. The table shows that, in general, the plant area material had higher levels of contamination than the waste tip material. For more detailed analytical results refer to Appendices 1 and 2.

Table 7.2 illustrates which target levels were met for specific contaminants in the waste tip and plant area material after bioremediation.

Table 7.1: Summary of results from the biopile field trial including target levels

Contaminant	Waste tip (mg/kg)		Plant area (mg/kg)		Target level (mg/kg)	
	Trial start	Trial end	Trial start	Trial end	Residential	Open space
Naphthalene	133	46	4,233	397	110	325
Benzo(a)pyrene	213	183	773	753	1	24
Total PAH	6,400	3,346	13,916	8,155	NA	NA
Benzene	0.43	0.020	7.9	0.072	2.5	19
Total BTEX	2.50	0.036	101.4	0.754	NA	NA
Total phenols	9.82	0.64	4.30	1.04	50	1,950
Mineral oil	1,037	1,037	1,644	1,417	NA	NA

Note:
NA = Not available

Table 7.2: Assessment of final contaminant concentrations against the target levels

Contaminant	Waste tip		Plant area	
	Residential	Open space	Residential	Open space
Naphthalene	✓	✓	✗	✗
Benzo(a)pyrene	✗	✗	✗	✗
Benzene	✓	✓	✓	✓
Total phenols	✓	✓	✓	✓

Sections 7.4.2 - 7.4.7 provide further details on the results of the biopile field trial with respect to the degradation of PAH, BTEX, mineral oil and total phenols, and gaseous emissions.

7.4.2 PAH DEGRADATION

At the end of the trial, mean total PAH concentrations had decreased from approximately 6,400 mg/kg to 3,346 mg/kg (48 % reduction) in the waste tip material and from approximately 13,916 mg/kg to 8,155 mg/kg (41 % reduction) in the plant area material. However, the degradation potential of individual PAH species varied considerably depending on their chemical structure. The changes in 16 individual PAH concentrations between the beginning and end of the field trial are represented graphically in Figures 7.1 and 7.2. These graphs show the concentrations measured in each of the three zones (A, B and C) and give an indication of the range of concentrations recorded across the three zones. The horizontal bars represent the mean values.

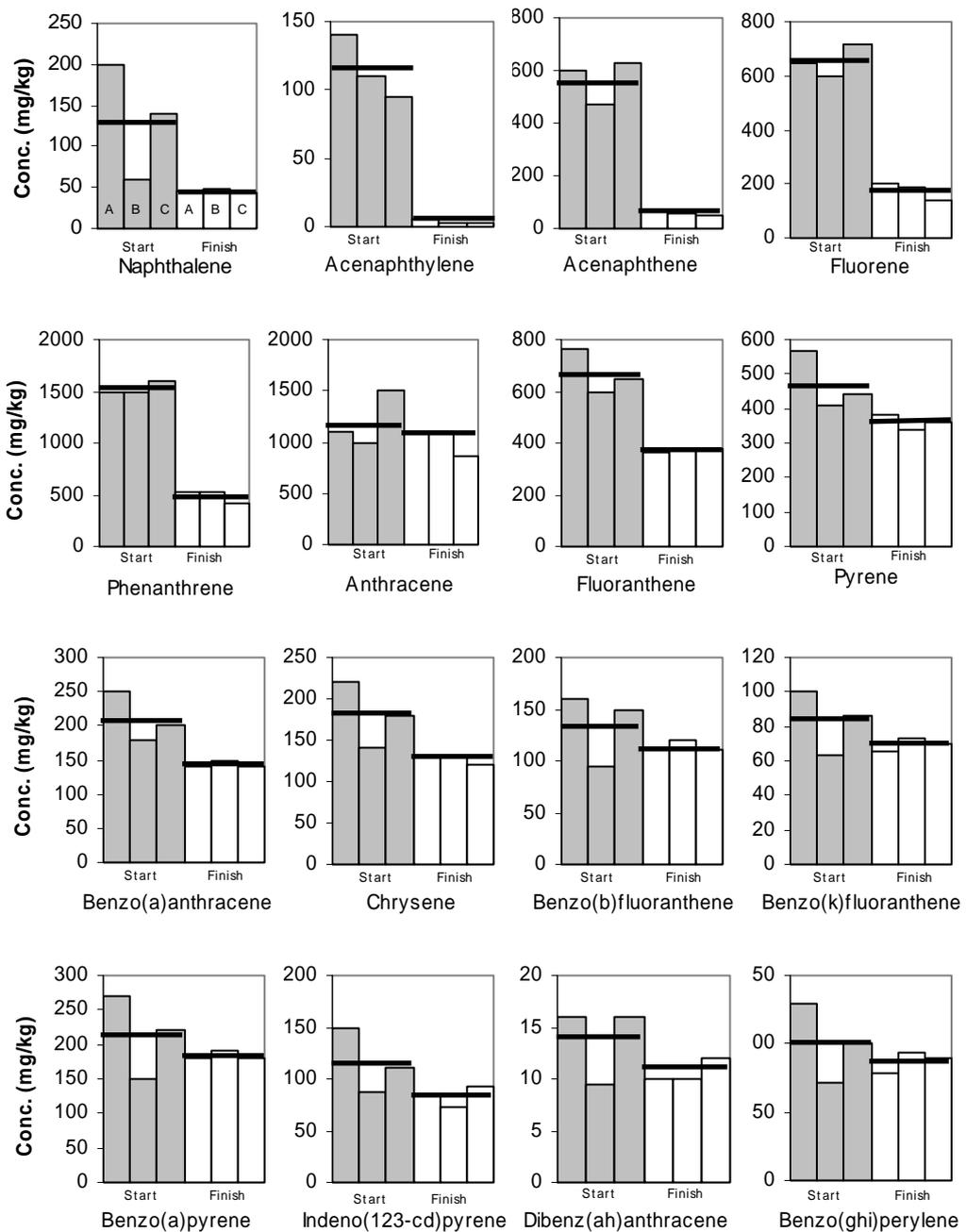


Figure 7.1: Individual PAH concentrations in zones A, B and C at the start and end of the field trial for waste tip material. Horizontal bars represent the mean value.

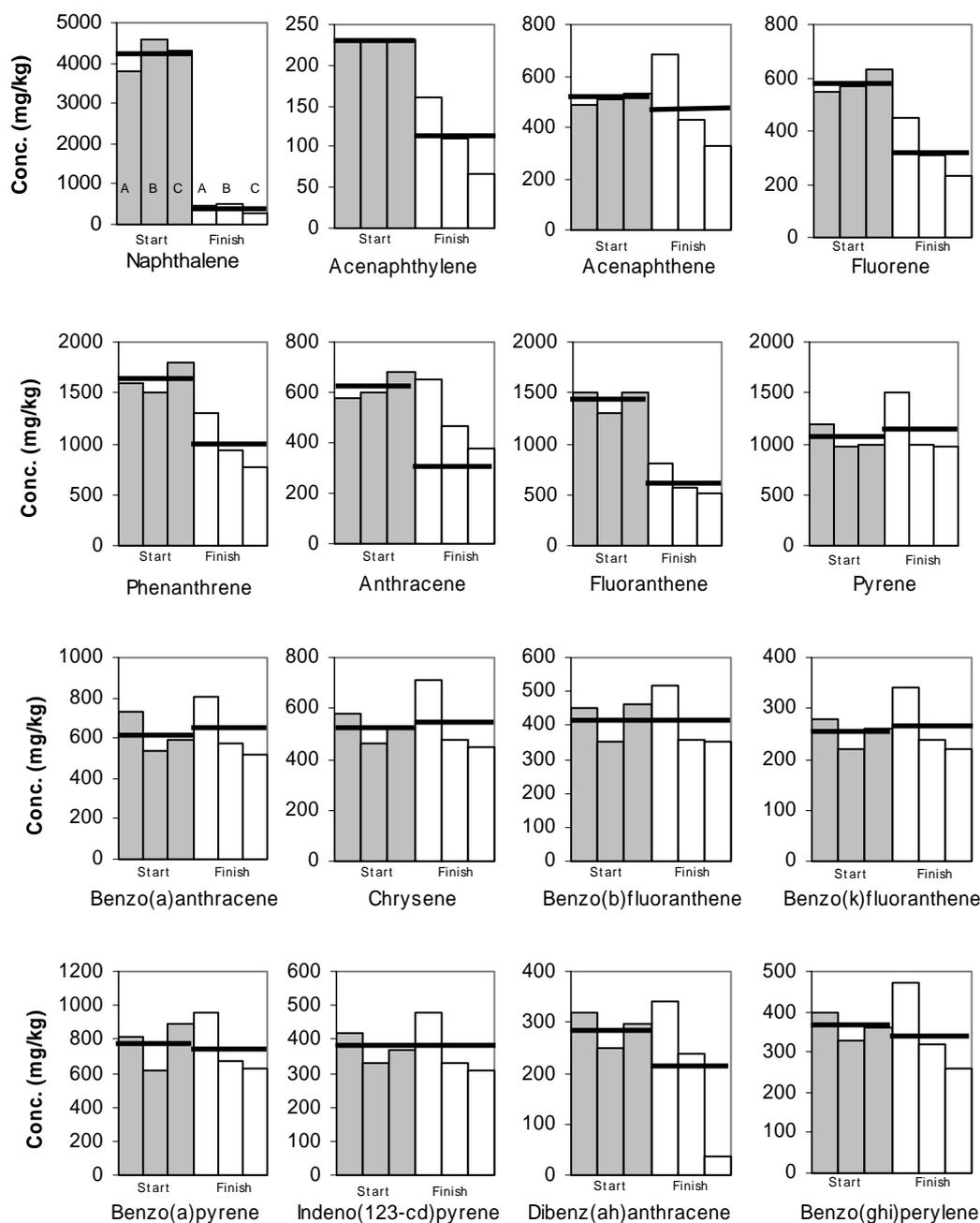


Figure 7.2: Individual PAH concentrations in zones A, B and C at the start and end of the field trial for plant area material. Horizontal bars represent the mean value.

For both the waste tip and plant area materials, degradation was generally much greater for “light” PAHs such as naphthalene and acenaphthylene, which contain two and three aromatic rings respectively, compared to “heavy”, five ring PAHs such as benzo(a)pyrene. The exception to this trend is the four-ringed fluoranthene, which exhibited significant degradation in both materials.

With regard to the target levels shown in Table 7.2, at the end of the trial, only the naphthalene concentrations (133 mg/kg) in the waste tip material had reduced to below the target level concentrations of 110 mg/kg for residential use and 325 mg/kg for open space. Levels in the plant area material had reduced from 4,233 mg/kg to 397 mg/kg. Benzo(a)pyrene concentrations for both materials (183 mg/kg and 753 mg/kg) remained well in excess of the target levels of 1 mg/kg and 24 mg/kg. Bioremediation of the waste tip and

plant area material using an active biopile did not reduce PAH contaminant levels to values that would meet the target values for this site. If bioremediation had continued beyond the 13 week trial it is likely that contaminant concentrations would have reduced further. However, a significant quantity of the organic contamination may have been bound up in tar and pitch particles and therefore would not be bioavailable.

7.4.3 BTEX DEGRADATION

Initial benzene and total BTEX contaminant levels were extremely low in the waste tip material. During the trial, the mean benzene concentration reduced from a mean of 0.43 mg/kg to 0.02 mg/kg (a 95 % reduction) and the total BTEX concentration reduced from 2.5 mg/kg to 0.036 mg/kg (98.6 %).

In the plant area material, initial benzene and BTEX concentrations were significantly higher than their waste tip analogues, but similar percentage reductions in concentrations were observed. The mean benzene concentration reduced from 7.9 mg/kg to 0.072 mg/kg (99.1 %) and the mean total BTEX concentration reduced from 101 mg/kg to 0.75 mg/kg (99.3 %).

For both materials, the final benzene concentrations easily met the target levels of 2.5 mg/kg and 19 mg/kg set for residential use and open space respectively. However, initial contaminant levels for benzene in the waste tip material of 0.43 mg/kg would have met the target level without any bioremediation. The initial contaminant level for benzene in the plant area material of 7.9 mg/kg met the target level for open spaces but not for residential use. Whilst BTEX compounds are considered as being suitable for bioremediation, a distinction has to be made between reductions in contaminant concentrations that are due to bioremediation and those that occur from volatilisation during the construction and operation of the biopile. Table 7.3 shows a comparison of the percentage reductions in BTEX concentrations in plant area and waste tip material during the two laboratory tests and the field demonstration biopile.

Table 7.3: Percentage reductions in BTEX concentrations for contaminated material during the laboratory tests and biopile field demonstration

Method of bioremediation	Percentage reductions in BTEX	
	Waste tip material (%)	Plant area material (%)
Slurry biodegradation test (TP344)	50-82	NA
Solid phase bioreactor test (TP344)	97	NA
Field biopile - stockpile	99	99

Note: NA = Not available

The loss of organic chemicals by volatilisation from soil can be significant, and a consideration of the Henry's Law constant (H_C) and the octanol-water partition coefficient (K_{OW}) for specific chemicals can give a qualitative indication of their potential to volatilise from soil. Henry's Law states that when a liquid and gas are in contact at any given temperature, the weight of the gas that dissolves in a given quantity of liquid is proportional to the pressure of the gas above the liquid. The value of H_C can give a qualitative indication as to whether volatilisation is significant for a specific contaminant. If H_C is less than $10^7 \text{ atm}\cdot\text{m}^3/\text{mol}$, the H_C for water, the contaminant is less volatile than water and as water evaporates the concentration of the contaminant in the soil will increase. Volatilisation will be rapid for chemicals that have an H_C value $> 10^3 \text{ atm}\cdot\text{m}^3/\text{mol}$.

K_{OW} is the octanol-water partition coefficient and is the ratio of the concentration of a substance in octanol to its concentration in water at equilibrium. It is an indication of the tendency of a chemical to leave the aqueous phase and become sorbed to organic matter in the soil. The following empirically defined categories based on H_C and K_{OW} the octanol-water partition coefficient can give an indication of volatilisation potential for different chemicals (SNIFFER, 1999). Some examples are given in Table 7.4.

$H_C > 1 \times 10^{-4}$ and $H_C/K_{OW} > 1 \times 10^{-9}$ high volatilisation potential
 $H_C < 1 \times 10^{-4}$ and $H_C/K_{OW} < 1 \times 10^{-9}$ low volatilisation potential

Table 7.4: H_C and K_{OW} values for selected organic chemicals (US EPA, 1990)

	H_C (atm-m ³ /mol)	K_{OW}	Volatilisation Potential
naphthalene	1.15×10^{-3}	1.30×10^3	High
benzo(a)pyrene	1.55×10^{-6}	5.50×10^6	Low
benzene	5.59×10^{-3}	8.30	High

Volatilisation was considered to be negligible for the plant area (108A) sample (except for naphthalene) during the bioreactor test but significant for the waste tip (TP344) sample. The waste tip (TP344) material used in the bioreactor had an mean BTEX concentration of 92 mg/kg compared with 4.7 mg/kg for the plant area (108) material. However, this variation in contamination levels was reversed for the biopile where the initial BTEX concentration in the waste tip material was 2.5 mg/kg compared with 101.4 mg/kg in the plant area material. Therefore, when looking at volatile losses, the assumption cannot be made that the waste tip and plant area material will behave like their predecessor samples in the bioreactor tests. Notwithstanding this, it is likely that some of the reductions in BTEX concentrations that occurred during the field trial are likely to be due to volatilisation and regular mixing of the material will have exacerbated this.

7.4.4 TOTAL PHENOLS DEGRADATION

The initial concentrations of total phenols in the waste tip and plant area materials (9.82 mg/kg and 4.30 mg/kg) were below the target levels of 50 mg/kg and 1,950 mg/kg for residential use and open space. During bioremediation these concentrations decreased to 0.64 mg/kg and 1.04 mg/kg, reductions of 93 % and 76 % respectively.

7.4.5 MINERAL OIL DEGRADATION

Mean mineral oil concentrations in the waste tip material showed no reduction during the trial with the mean concentration remaining at 1,037 mg/kg. In the plant area material mean mineral oil concentrations showed a slight reduction from a mean of 1,644 mg/kg to 1,417 mg/kg (14 %).

7.4.6 OTHER SOIL ANALYSIS RESULTS

In the waste tip material, solvent extractable matter (SEM) levels fell from 45,256 mg/kg to 15,651 mg/kg (65 %); total non volatile aromatic (TNVA) levels from 13,024 mg/kg to 6,245 mg/kg (52 %) and nitrogen-sulphur-oxygen (NSO) compound levels from 13,106 mg/kg to 5,915 mg/kg (55 %). Reductions were also observed for the plant area material: SEM levels fell from 80,459 mg/kg to 12,837 mg/kg (84 %); total non volatiles from 27,425 mg/kg to 13,024 mg/kg (52 %); and NSO levels from 88,449 mg/kg to 3,202 mg/kg (96 %).

The addition of activated sludge to part of the biopile that was constructed of plant area material showed no difference in degradation behaviour compared to those areas that did not receive this treatment.

7.4.7 GASEOUS EMISSIONS

When mixing of the biopile occurred, gaseous emission levels were measured using Draeger tubes. Although strong odours were observed in the shed during the first four weeks of the trial, all measurements for BTEX, VOC, naphthalene and total phenols were below detection limits and hence were not identified. Therefore, any losses of VOCs from the shed could not be quantified.

8. ECONOMIC CONSIDERATIONS

The breakdown of costs for the field biopile trial at the Avenue Coking Works is detailed in Table 8.1.

Table 8.1: Cost breakdown for the field-scale biopile demonstration

Activity	Cost
Insurance	£379.00
Preparation of method statements	£500.00
Mobilisation / erection of the biopile shed	£36,034.00
Management and staff costs	£3,418.00
Site accommodation	£2,515.00
Water charges	£1,509.00
Compliance with health and safety	£4,795.00
Running costs (electricity, operating plant, etc)	£35,577.00
Air analysis and material sampling ^a	£1,804.00
Production of reports	£1,868.00
Dismantling and demobilisation of equipment	£2,870.00
Total	£91,269.00
Price per tonne (based on 500 tonnes)	£183.00

Note: a. Does not include cost of laboratory analysis which was paid for by the client EMDA. These costs are not known.

The following assumptions were taken into account when arriving at these figures:

- Costs associated with site characterisation at the site were not included. These would normally be incurred on any contaminated site as a precursor to any remedial technique being employed or waste being consigned to landfill.
- When the field-scale demonstration occurred remedial targets had not been agreed for contaminated material at the site. Therefore, the time scale for treating material in the biopile may be longer or shorter than the 13 weeks allowed for during this demonstration.
- An impermeable surface (the HLSA) on which the biopile was constructed already existed on site. Therefore no construction costs were incurred with this aspect of the biopile design.

The cost of £183.00 per tonne (excluding off-site analytical costs) is very high considering that only 500 tonnes of material were treated. It is very high as it was a trial and the volume of material being treated was small and was treated in a purpose-built shed. If a biopile was located on a contaminated site it would not usually be constructed inside a shed. Instead it would be covered with a tarpaulin to help maintain an optimum temperature and prevent dust nuisance and water infiltration into the pile. However, if the contaminated material were transported to a fixed location treatment facility, treatment would then usually occur in a permanent shed or building. In fixed location treatment centres contaminated material similar in composition to that found at the Avenue site can be treated using biopile technology for about £15 - £20 per tonne. On-site treatment costs would be slightly less than this. However, if material had to be treated on site in a shed to prevent odour, then the costs would rise to about £25 - £35 tonne.

The cost of disposing of this type of material to landfill was running at about £30-40 tonne including landfill tax before the implementation of the Landfill Directive (July 2004). Since

July 2004, these costs are likely to rise considerably as the landfill tax rate increases, and the options for disposing of hazardous and non-hazardous material from contaminated sites to landfill significantly reduces due to the limited volume of hazardous landfill space available.

9. CONCLUSIONS

1. Laboratory-based treatability studies were used to help design the biopile field trial. Four test materials representing the waste tip and plant areas of the site were characterised to determine if they were suitable for bioremediation and this was followed by slurry biodegradation tests and solid phase bioreactor tests.
2. No particular nutrient treatment was more successful than another in promoting biodegradation. Data from the respiration measurements indicated that waste tip (TP344) and plant area (108A) samples had the greatest potential for biodegradation and these samples were chosen for further testing in bench-scale bioreactors.
3. The bioreactor tests on the waste tip (TP344) and plant area (108A) materials confirmed the results obtained during the slurry biodegradation tests. Volatilisation of some contaminants was significant for the waste tip (TP344) material.
4. A field-based active biopile demonstration was performed between July 9th and October 8th 2001. Approximately 200 m³ in total of waste tip and plant area material were used to construct separate sections of the biopile which was located inside a specially constructed shed. Operating inside a shed allowed greater control over moisture levels and the rate of injection of air into the biopile and also gave the ability to monitor gaseous emissions from the biopile.
5. At the start of the biopile demonstration, the contaminated material was pre-treated with a commercial nutrient formula and then mixed using a mechanical excavator. Samples were collected for analysis (t=0) and every two weeks the biopile was mixed and further samples collected. After two weeks, the biopile was sprayed with water three times a week to keep the moisture content at approximately 70 %.
6. At the end of the field trial, total PAHs in the waste tip material had reduced in concentration from 6,400 mg/kg to 3,346 mg/kg (48 %). Biodegradation was more pronounced for 'light' PAHs than for 'heavy' PAHs like benzo(a)pyrene. BTEX concentrations reduced from 2.5 mg/kg to 0.036 mg/kg (98.6 % reduction). Total phenol concentrations reduced from 9.82 mg/kg to 0.64 mg/kg (93 %). Mineral oil concentrations showed no reduction during the trial.
7. At the end of the field trial, total PAHs in the plant area material had reduced in concentration from 13,916 mg/kg to 8,155 mg/kg (41 %). Similarly to the waste tip material, biodegradation was more pronounced for 'light' PAHs than for 'heavy' PAHs. BTEX concentrations reduced from 101 mg/kg to 0.75 mg/kg (99 % reduction). Total phenols concentrations reduced from 4.3 mg/kg to 1.04 mg/kg (76 %). Mineral oil concentrations showed a slight reduction from 1,644 mg/kg to 1,417 mg/kg (14 %).
8. The biopile was enclosed by a shed and volatiles were captured and treated by activated carbon. During the trial all measurements for BTEX, VOC, naphthalene and total phenols were below detection limits, both above the biopile and in the air discharged from the shed.
9. The addition of activated sludge to part of the biopile that was constructed of plant area material resulted in no difference in degradation compared to those areas that did not receive this treatment, suggesting that the soils contained sufficient contaminant degrading microbes.

10. LESSONS LEARNED

1. A respiration test is the ideal test to determine whether bioremediation has stopped. A respiration test within the laboratory tests therefore gives a good idea of when further biodegradation can no longer be expected within the full-scale test. However, within the respiration test this end concentration is achieved within a shorter timeframe, due to more favourable conditions.
2. To achieve maximum reductions in contaminant concentrations may require the biopile to be operated for longer than the 13 weeks duration of the field trial described in this report.
3. Techniques used for laboratory analyses should be chosen that are reliable and which produce consistent and reproducible results. Thought needs to be given to choosing appropriate techniques prior to the start of trials.
4. A statistically coherent sampling programme should be designed to adequately characterise material used in the laboratory tests and in the field trial before any operations commence. Ensure a large stockpile of material is available from which samples can be retrieved.
5. The interpretation of the results from the laboratory tests is necessary before a design for a field biopile is agreed.
6. The addition of activated sludge to part of the biopile that was constructed of plant area material resulted in no difference in degradation compared to those areas that did not receive this treatment. Inoculation of the biopile with sludge from a wastewater treatment plant did not lead to an improvement in performance.

GLOSSARY OF TERMS

Abiotic

Not biotic; not formed by biologic processes.

Aliphatic hydrocarbon

Straight chained hydrocarbons without benzene rings (C₆H₆)

Anaerobic

Able to live, grow, or take place where free oxygen is not present

Aromatic hydrocarbon

Hydrocarbons containing benzene rings (C₆H₆)

Biodegradation

The consumption (degradation) of matter by microorganisms

Bioremediation

Processes that use living organisms (usually naturally occurring) such as plants, bacteria, yeast, and fungi to break down hazardous substances into less toxic or non-toxic substances.

Heterogeneous

Varying in structure or composition at different locations in space.

Homogeneous

Uniform in structure or composition at all locations in space.

Hydrocarbon

Chemical compounds composed only of carbon and hydrogen.

Inoculate

To implant microorganisms onto or into a culture medium.

Microorganisms

Microscopic organisms including bacteria, protozoans, yeast, fungi, mold, viruses, and algae.

Polycyclic Aromatic Hydrocarbon

Hydrocarbon compound with multiple benzene rings. PAH are typical components of tars, asphalts, fuels, oils and greases. These are also called Polynuclear Aromatic Hydrocarbons.

Recalcitrant

Unreactive, nondegradable; refractory.

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APPENDICES

Appendix 1: Biopile – Analytical Results Before Treatment

Appendix 2: Biopile – Analytical Results After Treatment

APPENDIX 1

BIOPILE – ANALYTICAL RESULTS BEFORE TREATMENT

Contaminant	Waste tip (mg/kg)				Plant area (mg/kg)			
	Zone A	Zone B	Zone C	Mean	Zone A	Zone B	Zone C	Mean
Naphthalene	200	60	140	133	3,800	4,600	4,300	4,233
Acenaphthylene	140	110	95	115	230	230	231	230
Acenaphthene	600	470	630	567	490	510	530	510
Fluorene	650	600	720	657	550	570	630	583
Phenanthrene	1,500	1,500	1,600	1,533	1,600	1,500	1,800	1,633
Anthracene	1,100	1,000	1,500	1,200	580	600	680	620
Fluoranthene	760	600	650	670	1,500	1,300	1,500	1,433
Pyrene	570	410	440	473	1,200	980	1,000	1,060
Benzo(a)anthracene	250	180	200	210	730	540	590	620
Chrysene	220	140	180	180	580	460	520	520
Benzo(b)fluoranthene	160	95	150	135	450	350	460	420
Benzo(k)fluoranthene	100	63	86	83	280	220	260	253
Benzo(a)pyrene	270	150	220	213	810	620	890	773
Indeno(1,2,3-c,d)pyrene	150	87	110	116	420	330	370	373
Dibenz(a,h)anthracene	16	9.4	16	14	320	250	298	289
Benzo(g,h,i)perylene	130	72	100	101	400	330	360	363
Total PAH	6,816	5,546	6,837	6,400	13,940	13,390	14,419	13,916
Benzene	0.23	0.15	0.91	0.43	9.2	6.0	8.5	7.9
Toluene	0.12	0.09	1.34	0.52	21.3	16.1	24.3	20.6
Ethylbenzene	0.04	0.0	0.18	0.07	8.7	6.5	10.3	8.5
Xylene	0.42	0.17	3.86	1.48	61.8	57.2	74.2	64.4
Total BTEX	0.81	0.41	6.29	2.50	101.0	85.8	117.3	101.4
Total phenols	3.82	17.43	8.21	9.82	2.11	3.48	7.32	4.30
Mineral oil	1,037	1,184	889	1,037	1,860	1,694	1,379	1,644

Adapted from DEC NV (2001)

APPENDIX 2

BIOPILE – ANALYTICAL RESULTS AFTER TREATMENT

Contaminant	Waste tip (mg/kg)				Plant area (mg/kg)			
	Zone A	Zone B	Zone C	Mean	Zone A	Zone B	Zone C	Mean
Naphthalene	47	48	43	46	440	490	260	397
acenaphthylene	5.1	2.5	2.5	3	160	110	67	112
acenapthene	70	54	53	59	680	430	330	480
fluorene	200	190	140	177	450	310	230	330
phenanthrene	530	520	420	490	1,300	940	770	1,003
anthracene	1,100	1,100	870	1,023	650	470	380	500
fluoranthene	360	370	380	370	810	570	520	633
pyrene	380	340	360	360	1,500	1,000	980	1,160
benzo(a)anthracene	140	150	140	143	810	570	520	633
chrysene	130	130	120	127	710	480	450	547
benzo(b)fluoranthene	110	120	110	113	520	360	350	410
benzo(k)fluoranthene	66	73	70	70	340	240	220	267
benzo(a)pyrene	180	190	180	183	960	670	630	753
indeno(123-cd)pyrene	86	73	92	84	480	330	310	373
dibenz(ah)anthracene	10	10	12	11	340	240	37	206
benzo(ghi)perylene	79	94	89	87	470	320	260	350
Total PAH	3,493	3,465	3,081	3,346	10,620	7,530	6,314	8,155
benzene	0.014	0.022	0.025	0.020	0.087	0.07	0.059	0.072
Total BTEX	0.024	0.038	0.045	0.036	0.958	0.696	0.608	0.754
Total phenols	0.39	0.55	0.97	0.64	1.18	1.22	0.73	1.04
Mineral oil	1,216	1,046	848	1,037	1,113	1,912	1,225	1,417

Adapted from DEC NV (2001)