CL:AIRE Technology Demonstration Project (TDP) Bulletins provide a summary of CL:AIRE approved Technology Demonstration Projects. TDPs have passed through the CL:AIRE application and review process, and represent demonstration for the specific conditions in which they are applied. This bulletin describes the development of a decision support tool for whether to use *in situ* chemical oxidation for the remediation of contaminated groundwater and guidance on which oxidants to use.

Decision support tool for innovative *in situ* multi-contaminant groundwater remediation

1. INTRODUCTION

Remediation of contaminated groundwater is of key importance in the regeneration of brownfield sites. In many instances the primary contaminants are organic compounds and the diversity of contaminant types, together with the variability of hydrogeological regime, has led to the use of a wide variety of physical, chemical and biological approaches to achieve remediation. In recent years, the introduction of chemical oxidation compounds, injected into the subsurface in order to treat contamination, has been increasingly utilised (USEPA, 2006; Crimi & Taylor, 2007; ESTCP, 2008). However, such treatments have frequently been undertaken on the basis of field experience without an underpinning of documented scientific analysis. For this reason, a research programme has been undertaken to investigate the use of *in situ* chemical oxidation (ISCO) remediation technologies in a systematic manner using comparative field trials supplemented by laboratory analyses and modelling studies, with a view to providing practical support for consultants, regulators and stakeholders using this technology.

2. OBJECTIVES

The primary objective of the research programme was to develop a decision support framework to assist practitioners and other stakeholders on the decision of whether to use ISCO for the remediation of contaminated groundwater and, if deemed appropriate, guidance on the selection of chemical oxidants and their application in the subsurface. A consortium comprising WorleyParsons, Imperial College London, National Grid Property Ltd, the Environment Agency and the City of Bradford Metropolitan District Council was formed. A programme of work was undertaken to investigate the performance of various oxidation technologies in the field. Laboratory studies were used to quantify and assess the controls on the chemical reaction rates of the oxidants used in the field trials. In addition, modelling tools were developed to assist in the interpretation of the field data and to help formulate a decision support framework, which would integrate the results, enabling them to be applied more generally. The knowledge gained from the project was also incorporated into an Excel Decision Support Tool.

3. FIELD TRIALS

The site selected for the experimental facility was a former gasworks where there was a history of contamination and where the availability of the site and its physical and chemical characteristics were suitable for the planned fieldwork. Three *in situ* groundwater remediation techniques were successfully trialled at the site. Two of these, catalysed hydrogen peroxide (CHP) (also known as Fenton's Reagent) and sodium persulphate (SP), were aggressive chemical oxidants typically used in ISCO remediation. The third was the injection of super-saturated oxygenated water, obtained using the Groundwater Pressurized Remediation Optimizer (gPRO®) system (www.gPROinfo.com). Although not strictly a chemical oxidation method, it falls in between the aggressive methods being investigated and natural attenuation, as it provides a large input of dissolved oxygen to the subsurface that can enhance naturally occurring biological attenuation
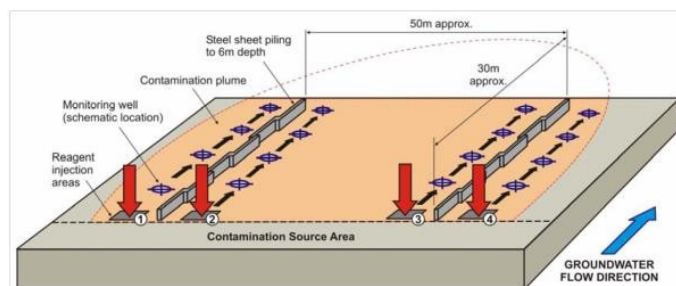

Figure 1: Conceptual design of the field investigation programme.

methods. To our knowledge, this is the first time that such comparative trials have been undertaken at a former gasworks site. Of these techniques, only CHP is a well-documented and tested treatment technology (Greenberg *et al.*, 1998; Watts & Teel, 2006; Watts *et al.*, 2007). The gPRO® technique had not been used in the UK before and relatively few published field trials of SP exist (Liang *et al.*, 2003; Liang *et al.*, 2008), although its use in the field has increased in recent years.

Field trials were undertaken by constructing isolated areas within the site for the injection and monitoring of each reagent. This involved the installation of steel sheet piles to create four hydraulically isolated treatment corridors within which different treatment technologies could be applied (Figure 1). The investigation area, where the bulk of the contaminated groundwater was located, had three metres of backfilled and compacted clay and engineered granular fill material (placed in the unsaturated zone during previous soil remediation works) overlying a sand and gravel aquifer. Following installation of the sheet piles, 24 additional groundwater monitoring wells were installed to monitor contaminant degradation in the treatment corridors.

Hydraulic conductivity tests were carried out on these wells to provide a more accurate estimate of permeability in the treatment corridors. A programme of groundwater sampling was then undertaken to obtain baseline contaminant concentrations. Following the completion of the facility setup, three of the corridors (A, B and C) were selected for the field trials. Corridor D was used as a control. Two different aggressive oxidants, CHP and SP, were trialled in corridors A and B, respectively. Details of the installed infrastructure associated with these are shown in Figures 2 and 3. The treatment involving the injection of super-saturated oxygenated water (gPRO®) was trialled in Corridor C.

Comparative trials of the two aggressive chemical oxidants, CHP and SP, were successfully undertaken. CHP was injected as 4 m³ 2.5% v/v aqueous solution over a period of 2.2 hrs. SP was injected as 4 m³ 2% v/v aqueous solution over a period of 1.33 hrs. Both treatments included a lithium bromide tracer.
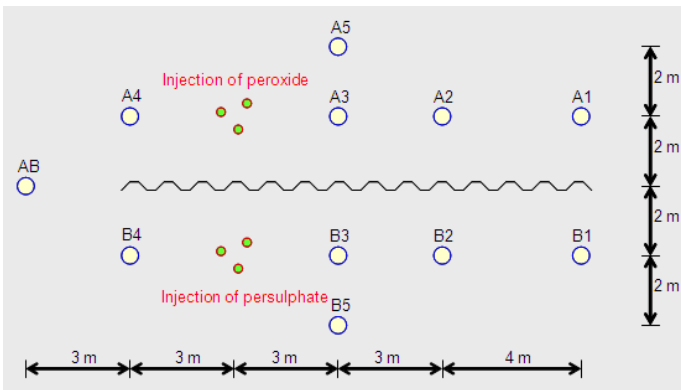
Figure 2: Infrastructure associated with CHP and SP trials in Corridors A & B
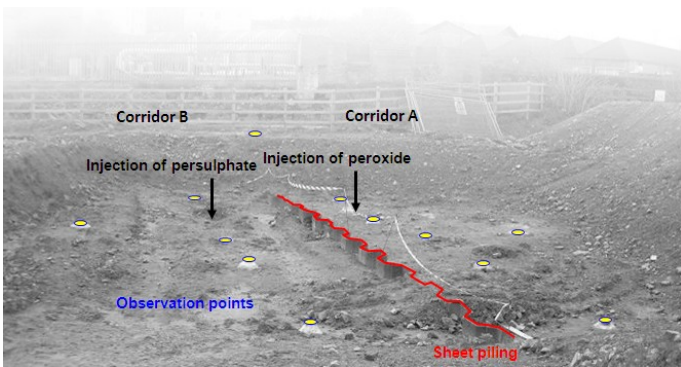

Figure 3: Annotated photograph of Corridors A & B showing completions of monitoring wells

Throughout the injection periods a range of variables were monitored in each of the observation points (A1 to A5 and B1 to B5 as shown in Fig. 2). These included water level, electrical conductivity, dissolved oxygen, pH and redox potential. Water level measurements were recorded using automatic loggers. The results confirmed that SP and CHP are powerful oxidants but that their lifespan is limited, in this case 3 days and 1 day, respectively. This was due to a combination of dispersion, arising from the injection process and natural groundwater flow, and oxidation. Good contact between the oxidant and the target zone is therefore vital to achieve a successful treatment. An important observation from the field trials was the rapid breakthrough of oxidant (within 30 minutes from start of injection) at monitoring wells close to the injection points (Figure 4).
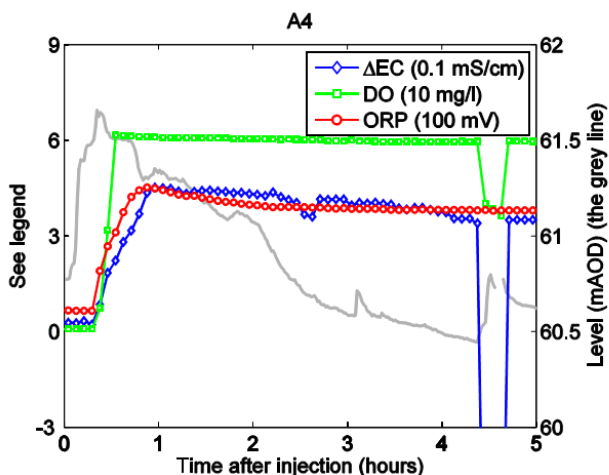

Figure 4: Plot of variables observed at monitoring well A4 during injection of hydrogen peroxide (EC-electrical conductivity, DO-dissolved oxygen, ORP-redox potential).

Colorimetric kits were used to determine oxidant concentrations in the field. In the case of SP, the sodium present in the oxidant and, in particular, its effect on electrical conductivity could also be used to detect oxidant breakthrough using conductivity logging. The pH of the groundwater became acidic during both trials although the magnitude and duration of the change was greater in the SP trial. The pH drop caused by the injection of SP was significant (pH fell to less than 3 for prolonged periods) and microbial activity would have been adversely affected. CHP reactions are more efficient at lower pH conditions. Work has been undertaken to investigate the use of neutral-pH catalysts or pre-treating areas with acid to ensure optimum conditions are produced prior to injection with hydrogen peroxide (USEPA, 2006). Hydrogen peroxide has also been used to catalyse SP in order to generate sulphate free-radicals (Block et al., 2004; Ko et al., 2012). Significant increases in dissolved iron (SP and CHP), sulphate (SP), sodium (SP), copper (SP) and nickel (SP) concentrations were detected in groundwater. In the case of sodium and sulphate part or all of this increase is due to the composition of the oxidant. It is also possible that some of the increase in dissolved nickel and copper is due to impurities present in the oxidant. Clearly, in the case of CHP some of the increase in dissolved iron is because of its presence as a catalyst. However, it is likely that the bulk of the increase in dissolved iron (and a possible contributing cause in the case of copper and nickel) is from dissolution caused by the acidic conditions. This effect has been noted elsewhere, particularly at former gasworks sites (Shields et al., 2006). The concentrations of these were all increased to above their respective guideline values (i.e. Drinking Water Standard or Environmental Quality Standard). Careful planning is therefore required during the design stage to ensure that these adverse effects do not have a significant impact on controlled waters. It is recommended that where drinking water abstractions are at risk, pilot-scale trials should be undertaken, in conjunction with groundwater modelling.

In the gPRO® trial, oxygen was delivered into the subsurface at relatively high concentrations (up to 60 mg/L), which is well above the breakthrough values of 5 mg/L observed in the monitoring wells following injection. This was due to dilution, possibly combined with some degassing following injection. Within a day of injection dissolved oxygen concentrations had fallen to below 1 mg/L, in addition, negative oxidising redox potentials were prevalent. This showed that optimum conditions for aerobic degradation of the organic contaminants were not achieved. Injections were repeated weekly, but although the oxidising redox potentials increased, this was not sufficient to produce aerobic conditions. As no obvious signs of aerobic degradation were identified, it is likely that injections had stimulated inorganic oxidation, a process previously observed at a contaminated gasworks site (Shields et al., 2006). Evidence for nitrate, iron, manganese and sulphate reduction was documented. However, in some cases the bioavailability of these terminal electron acceptors became limited. In order for degradation to continue using these terminal electron acceptors, additional nutrient injection would be required. No noticeable contaminant destruction was recorded from the analytical data taken during the trial. This is likely to be due to the mobilisation of soil / sorbed contaminants disguising any trends in the data in the dissolved phase. However, although this process could be used to remove soil / residual phase NAPL contaminant mass its use may not be cost effective. Therefore, it is concluded that the gPRO® technique has limited use for gasworks remediation. Particularly where soil contaminant mass / residual-phase NAPL is present, the reagent target zone is an unconfined aquifer (which results in excessive oxygen loss), treatment timescales are short (less than a year) or the subsurface / groundwater is nutrient deficient (although, in this case, terminal electron acceptors could be introduced with the oxygen saturated water). Where gPRO® may have a use is as a final polishing stage in a remediation project where nearby sensitive receptors prevent the use of more aggressive and potentially hazardous substances (such as chemical oxidation).

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4. LABORATORY TRIALS

Laboratory studies provided complementary data to the field trials. The application of additives to the groundwater has an impact upon the chemical forms of the contamination and the natural subsurface chemical species. A quantitative investigation of these effects was made, including controls on the reaction rate, the amount of additive dosed and the fate of the contaminants. These were studied using controlled batch reaction testing of the subsurface and surface soil types, uncontaminated groundwater and separate contaminants present in the site groundwater following the treatment additives applied in the field. The consumption of the additives in each system was monitored and the fate of the contaminants and chemical changes to the soils were evaluated using Gas Chromatography (organics) and Inductively Coupled Plasma Atomic Emission Spectrophotometry (metals) and Ion Chromatography (anions), Scanning Electron Microscopy and Fourier Transform Infrared Spectroscopy (soil surface chemistry). Other chemical parameters monitored were dissolved oxygen, electrical conductivity, pH and redox potential. Data from the laboratory tests provided important information on the degradation mechanisms for the contaminants and the effects of subsurface physiochemical conditions upon these processes.

The laboratory work concentrated on investigating in detail the two chemical oxidants trialled in the field (i.e. SP and CHP). Their performance was assessed in terms of oxidant and contaminant half-life, and the effects of varying levels of dissolved iron to catalyse the reaction. The half-life values, in days, were obtained by averaging a first order decay constant obtained from an exponential fit to the experimental data. The results for SP are shown in Table 1 and those for CHP in Table 2.

Table 1. Summary of half-lives [days] for sodium persulphate experiments (DI = deionised water, GW = groundwater from site, SOIL sediment and groundwater from site)

Sodium Persulphate				
Catalyst: Oxidant		$t_{1/2}$[d] DI	$t_{1/2}$[d] GW	$t_{1/2}$[d] SOIL
Fe(1:0)	SP	stable	stable	N/A
	Benzene	27.7	25.4±13.6	N/A
	Toluene	18.9	15.8±8.6	N/A
Fe (1:1250)	SP	stable	stable	35.1±2.3
	Benzene	7.8±2.4	21.9±11.4	15.6
	Toluene	6.6±0.8	16.5±9.9	9.8
Fe(1:250)	SP	210.1	173.29	38±7
	Benzene slow	6.9±0.6	12.5±4.4	13.4±1.9
	Toluene slow	5.3±0.5	9.2±2.8	10.3±1.7
	Benzene fast	0.81	Not observed	Not observed
	Toluene fast	0.62	Not observed	Not observed
Fe(1:50)	SP	61.3	31±11	36.0±1.5
	Benzene slow	2.15±0.02	2.0±1.8	11.9±0.3
	Toluene slow	1.75±0.06	1.5±1.4	9.6±0.4
	Benzene fast	0.098	Not observed	Not observed
	Toluene fast	0.081	Not observed	Not observed

Note: "Fast" initial reaction rate; "slow" subsequent reaction rate

Table 2. Summary of half-lives [hours] for hydrogen peroxide experiments (DI = deionised water, GW = groundwater from site, SOIL sediment and groundwater from site).

Hydrogen Peroxide				
Catalyst: Oxidant		$t_{1/2}$[d] DI	$t_{1/2}$[d] GW	$t_{1/2}$[d] SOIL
Fe (0:1)	Oxidant	13860	N/A	N/A
	Benzene	973	N/A	N/A
	Toluene	876	N/A	N/A
Fe (1:1250)	Oxidant slow	N/A	N/A	4.21
	Oxidant fast	N/A	N/A	0.60
	Benzene	0.69	9.60	3.50
	Toluene	0.81	10.23	2.58
Fe(1:250)	Oxidant slow	1.87±0.17	2.38±0.02	2.09
	Oxidant fast	Not observed	Not observed	0.63
	Benzene slow	0.17±0.05	0.176±0.006	5.74
	Toluene slow	0.22±0.7	0.207±0.006	3.38
	Benzene fast	0.042±0.015	0.08±0.01	Not observed
	Toluene fast	0.048±0.017	0.08±0.01	Not observed
Fe(1:50)	Oxidant slow	12.48±2.6	4.62	0.89
	Oxidant fast	0.40±0.05	0.57	0.50
	Benzene slow	0.075	0.080	1.77
	Toluene slow	0.086	0.094	1.38
	Benzene fast	0.012	0.013	Not observed
	Toluene fast	0.013	0.014	Not observed

Note: "Fast" initial reaction rate; "slow" subsequent reaction rate

SP decay was observed to be faster in the presence of toluene than benzene, which was consistent with previous work (Liang et al., 2008). The very long half-life of pure SP in deionised water is in accord with the known stability for this oxidant (Liang et al. 2003, Watts & Tell 2006). The contaminant half-life in groundwater dosed with oxidant tends to be longer than that for deionised water due to the presence of dissolved natural organic matter, which competes for oxidant. Oxidant and contaminant (benzene & toluene) half-lives were found to be effectively independent of catalyst concentration for experiments conducted in aquifer sediments. This is thought to be due to a greater amount of naturally occurring iron compared to the amount of catalyst. In addition, the slowing of the degradation reaction was compensated by an increment in the free radical production due to a larger catalyst concentration. For activated SP in soil sediments, the oxidant half-life was about 35 days, whereas the contaminant half-life was about 10 days. In the treatments using higher iron catalyst:oxidant ratios (1:250 and 1:50) oxidant degradation was found to occur at two different rates, an initial fast rate and a subsequent slow rate. This was thought to be due to the rapid consumption of free radicals during the initial reaction phase.

From the *in situ* oxidation point of view, the results for the experiments using sediment are the most interesting. Even though there is some scatter in the results, possibly due to sample heterogeneity, the methodology was able to overcome one of the main challenges when measuring the oxidation of organics, namely the build-up of considerable pressures in the vials due to the large amount of oxygen produced from the reaction between the hydrogen peroxide and the iron catalyst. The degradation rates for Fe:hydrogen peroxide (1:1250) and Fe:hydrogen peroxide (1:250) were very close, indicating that the naturally occurring (mineral) iron drove the reaction kinetics. Moreover, the initial fast consumption of hydrogen peroxide considerably reduced its concentration in solution by about 90% over the first 1.5-2 hours. This was not accompanied by rapid contaminant degradation, possibly due to the strong competition with natural organic matter for free radicals. The remaining contaminant was only partially degraded since the reaction seemed to halt due to the very low amount of hydrogen peroxide in solution. In soil sediments CHP half-life was between 1 and 4 hours, while the degradation half-lives for benzene and toluene were between 1.5 and 5 hours.

The results of these experiments highlight the need to balance the timescale when the oxidant is active with the time required for the injected fluid to interact with the ambient groundwater. The slower decomposing SP ($t_{1/2}$ = 30 days) in soil was, nevertheless, able to degrade benzene and toluene over comparable time periods ($t_{1/2}$ about 10 days). The catalyst concentration was not important due to the amount of natural mineral iron (and manganese) oxides in the soil. Due to its slow decay rate, SP is able to migrate to contaminated areas reasonably far from its point of injection. On the other hand, the kinetics for CHP were much faster, taking hours rather than days. Even if contaminant degradation was similarly fast, it appeared that the CHP oxidant was decomposing so quickly that complete degradation was not always achieved. As the rate of hydrogen peroxide decay is catalysed by natural iron and manganese oxides, its instability may be a serious issue, even without the addition of an iron catalyst. This might mean that its effectiveness in the field could be affected if the delivery of oxidant to contaminated groundwater is not able to ensure complete mixing within a timescale of a few hours.

The values obtained for the half-lives of these processes using different catalyst concentrations are generally in agreement with previous findings (Kang & Hua, 2005; Liang *et al.*, 2008). However, given the variability between soils (due to different amounts of metallic oxides and organic matter), the results obtained here are specific to the research field site. Therefore, an important outcome from the research is the value that laboratory measurement of reaction kinetics provides in terms of optimum catalyst concentrations and oxidant delivery methods in the field.

5.   MODELLING

The aim of the modelling programme was to investigate the processes governing the transport of oxidant to the contaminated region within the aquifer. Although it was originally envisaged that the main focus of the work would be the development of a reactive transport model, as the project progressed it became clear that a more fundamental issue was to understand the manner in which chemical oxidants move through the subsurface during and over the period following injection. This is important as it governs how effective the remediation process is likely to be, particularly for oxidants with fast reaction rates, such as CHP. By seeking to maximise the contact between the injected oxidant and contaminated groundwater a more efficient, and therefore cost-effective, procedure can be developed, which has important implications for remediation selection and design. The field programme gave rise to an important set of data on the processes associated with, and the effects of, injecting chemical oxidants into the subsurface. Interpretation of the experimental results, in particular physical and chemical responses in observation wells during injection trials, led to the conclusion that the speed and extent of oxidant breakthrough in the vicinity of the injection site was due to hydraulic fracturing on a scale of at least a few metres. This has a dramatic effect of the flow field and consequently on where and when the oxidant interacts with contaminated groundwater.

A review of the literature showed that there were limitations with existing model representations of this process, in particular the representation of leak-off[1] from the fracture. However, this is an extremely important process as it not only controls the radial extent of the fracture but also the delivery of oxidant to the subsurface. Therefore, a major outcome from the project has been the development of new fracture leak-off model (Figure 5). This has led to improved process understanding and new tools to assist in the application of this understanding to field problems (Mathias and van Reeuwijk, 2009). Initial investigations using a simplified model for hydraulic fracturing developed during the project and applied to the field data, gave encouraging results in terms of the extent and dynamics of hydraulic fracturing. It demonstrated the dependence of fracturing on the mechanical properties of the soil/rock and the pressure of the injected fluid. The model also provides a means of determining the flow field associated with the hydraulic fracturing, which can be used to investigate the effects of oxidant behaviour (Figure 6).
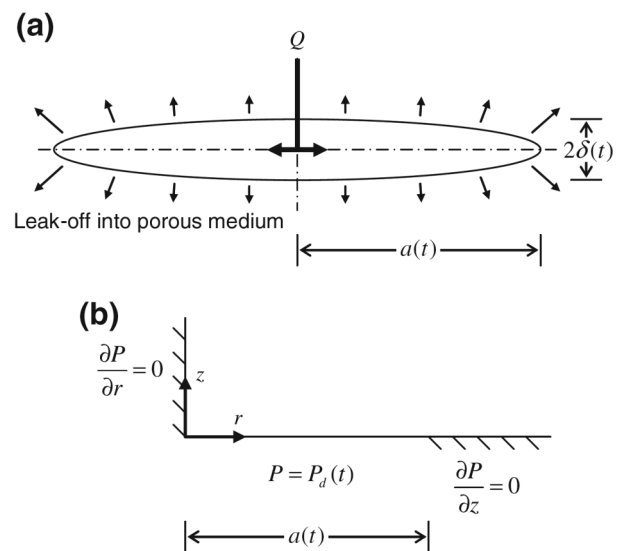

Figure 5. Schematic diagrams showing the geometry of leak-off model: a) Section through centre of fracture, b) Schematic of boundary value problem
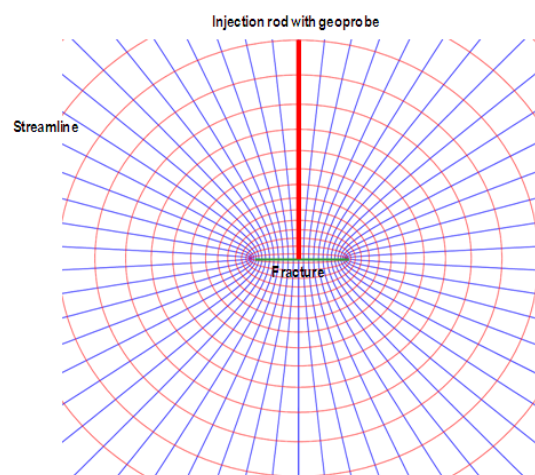

Figure 6: Equipotentials and streamlines associated with leak-off from a hydraulic fracture

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[1] denotes fluid loss from hydraulic fractures

6.   DECISION SUPPORT TOOL

A further output from the research was a Decision Support Tool (DST), which took the form of an Excel spreadsheet incorporating Visual Basic macros. Given the types of contaminant present, it helps the user to decide whether ISCO would be suitable as a remediation technology and, if so, which oxidants would be appropriate for evaluation in a pilot trial. It is underpinned by a comprehensive review of the literature, supplemented by outcomes from the current project. The tool is hosted on CL:AIRE's website and is freely available to download (see *Further Information* on page 6). It is a key component of the main deliverable from the project, which is a framework for decisions regarding ISCO.

The framework is shown diagrammatically in Figure 7 and comprises three main decisions / actions. The first is to decide whether ISCO is a suitable remediation technology for a given site. Support in answering this question is provided by the first stage in the Decision Support Tool (DST:1). This checks the suitability for using chemical oxidants in light of the types of contaminant requiring remediation. If ISCO is a plausible remedial approach, the next key decision is to evaluate the various chemical oxidants that are available in order to provide a ranked shortlist. Support for this is also provided by the Decision Support Tool (DST:2) on the basis of the ability of a particular oxidant to degrade the specific types of contaminant present. Finally, owing to the site-specific nature of the problem there is a need to evaluate the selected oxidants using pilot-scale field trials. Here, the other outcomes from the project provide important and essential guidance for the design and optimisation of such field trials, in particular:

- the design of a field monitoring and investigation programme;
- the implementation of field ISCO trials;
- a protocol for laboratory analyses to evaluate site specific degradation rates; and
- the potential for hydraulic fracturing to aid oxidant delivery and to assist in the design of the injection system.

7.   CONCLUSIONS

The project has delivered an integrated programme of work in which ISCO has been investigated using experimental field trials, supported by laboratory and modelling analyses, to provide a framework to assist regulators and practitioners in deciding whether the technology can be used at a contaminated site, and if so, which oxidants are the most appropriate for the contaminants present.

It was found that a single preferred chemical oxidant cannot be recommended for use at former gasworks sites. Results from the project suggest that there are advantages and disadvantages to both the oxidants used and site-specific constraints affect oxidant selection. The work has suggested that the use of sodium persulphate together with hydrogen peroxide could have added benefits, not only for contaminant destruction but to limit the magnitude of the adverse effects observed following the use of the oxidants individually. A further important finding is that the pressures generally used to inject oxidants are likely, given the mechanical properties of many subsurface sediments, to lead to rapid migration of oxidant through artificially enhanced flow pathways. This explains why efficient oxidant delivery can be made over large areas despite the rapid decay timescales that were identified in the laboratory analyses.

The outcomes of the work have been made available through a Decision Support Tool, which is available to download from the CL:AIRE website, along with supporting documentation on the main outcomes from the project. It is anticipated that, through the use of this tool along with associated reports and papers, the body of information available on ISCO will be enhanced and its success as a remediation technique will be strengthened. This will allow ISCO to tackle, in a more appropriate and cost-effective manner, the challenges that remain in developing and regenerating brownfield sites and facilitating urban development and renewal in the UK and elsewhere in the world.
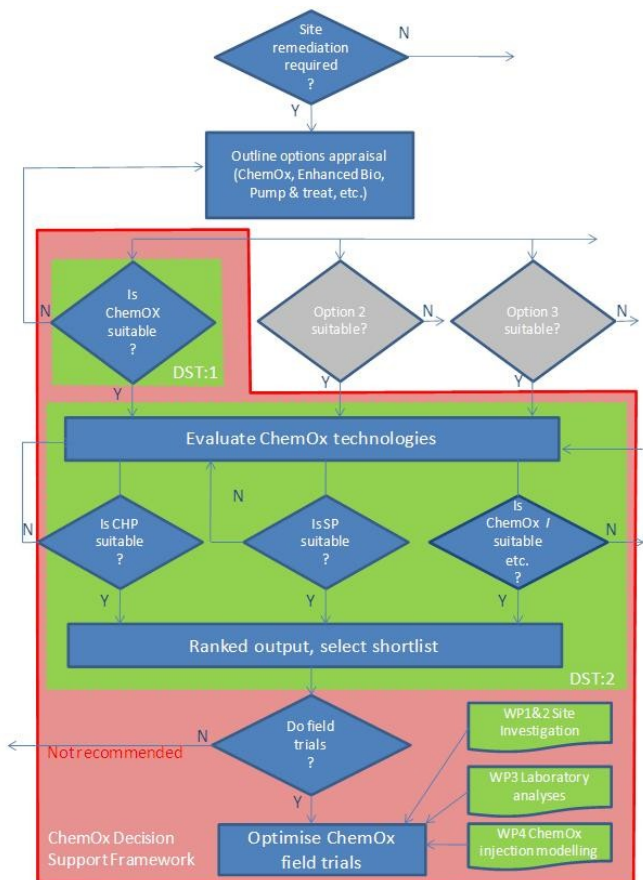

Figure 7. Overview of ChemOx Decision Support Framework (DST1/2 refer to sheets in the Excel Decision Support Tool and WP# refers to Project Work Package reports)

ACKNOWLEDGEMENTS

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