A METHOD OF APPLYING AN ELECTRIC FIELD TO CONTAMINATED SOIL CONTAINING DEGRADING BACTERIA

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Abstract— This research paper addresses the problem of bioavailability of contaminants in soils, and has investigated the use of electro kinetic phenomena as tools to bring about an increase in this factor, leading to improved biodegradation. Soil contaminated with pentachlorophenol (sodium salt) was subjected to an electric field in a number of experiments, with significant transport of the initially ionic chemical noted. The transport and fate of this chemical were tracked throughout each experiment, along with properties of the soil pore fluid. Significant changes in soil chemistry were noted (particularly pH or moisture content, depending on the experiment). The effect of pH change was found to be particularly important in this respect, with acidic conditions hindering both movement and bioavailability. A method of applying an electric field to contaminated soil containing degrading bacteria was developed which minimized the changes to these parameters within the soil. A significant increase in the effectiveness of the remediation was noted with this technique, with substantially faster degradation found to occur.

Index Terms— remediation, contaminant bioavailability, in situ remediation, pollutant, clay soi.

I. INTRODUCTION

The aims of the work described here were to assess the feasibility of directly increasing contaminant bioavailability using an electric field, thereby offering scope to improve the process of bioremediation by reducing remediation time and increasing the rate of degradation. The first objective was the design of simple, robust apparatus that would allow ease of use in complex experiments. Simple, safe protocols to prepare contaminated, inoculated soil specimens were also required, in order to ensure the repeatability of experiments. The next goal was the determination of the basic properties of the soil and contaminant, particularly with respect to the behaviour of the contaminant in the soil. This could then be extended with the introduction of the electric field, to establish the contaminant transport behaviour within the system –movement of PCP (on a micro- or macro-scale) was required to improve bioavailability. Once the contaminant behaviour was characterized, and a method of inducing movement had been found, the introduction of degrading bacteria and their effect on the pollutant could be studied with and without the electric field. Further studies on the effect of ageing (increased recalcitrance with time) of the contaminant in soil, the behaviour of other contaminants and the effect of the electric field and contaminant on bacteria were additional targets.

The ability of the electric field to move the contaminant molecules even a small distance will significantly increase access, by helping to move it out from inaccessible pores, by redistributing high concentrations and also by desorbing the contaminant from the soil. The alliance of these two techniques gives greater scope for *insitu* remediation in fine-grained soils. The problems of dealing with the often-strong association between pollutant and clay soil are usually overcome by removing either althea soil or at least the finer portions of it. This new technology would allow a more rapid and efficient remediation of fine-grained soils without the need to dig and dump.

Literature survey

To balance the charge on the pore walls, there will be an excess of positive ions in a region close to the face, together with 'bound' water molecules. The model used to describe the variation in electrical potential around a particle is the Gouy-Chapman-Stern model (Atkins, 1990), as shown on Figure 2-3. This was created by taking the Gouy-Chapman model, which provided a realistic model of the diffuse outer layer but led to unrealistically large surface concentrations of ions, and modifying it by combining it with Helmholtz' theory of the distribution of ionic species at a solid-solution boundary. The electrical potential around an object with surface charge such as this may be quantified using the zeta potential. This is the potential at the boundary between immobile water molecules sorbed to the soil and mobile water molecules in the free pore fluid. The species of captions present will vary depending on variables such as the soil constituents and history. When an electric field is applied, captions in the fluid flow towards the cathode and anions in the fluid flow towards the anode, but the excess of cations generates an overall friction on water molecules, creating a net flow of water, which is known as the *electro osmotic flow*. This flow is usually towards the cathode, due to the negative zeta potential and therefore excess of cations in the

surrounding double layer. However, under certain conditions (notably in highly acidic regions), an excess of small, positive ions can become tightly bound to the soil due to the electrostatic attraction. This has the net effect of reducing the effect of negative charges on the soil surface, and may even make the net charge positive. In this case, as the zeta potential will now be positive, an excess of negative ions will be required in the double layer for charge balance, and so application of an electric field will lead to flow towards the anode. This effect is described by Eykholt and Daniel (1994).

Electro osmosis is a phenomenon that occurs only in soils containing these particles with charged surfaces. It is a function of the excess of positively charged ions over negatively charged ones, or *vice versa*. It therefore does not occur in sands, silts or gravels unless an appreciable amount of clay particles is present. One of the first analytical models of electro osmotic behaviour was proposed by Helmholtz (1879) and modified by, amongst others, Smoluchowski (1921), which gave the velocity of fluid flow in such a porous medium. Objects such as pollutant molecules or bacteria in the soil are therefore subject to a range of conditions and forces. For example, bacteria may be affected by electrophoresis as well as the electro osmotic water flow; although they will naturally become attached top soil particles or organic matter and so may resist these forces.

Ballou (1955) made predictions of electro osmotic flow and the effect of various physicochemical properties of the soil and pore fluid. He prepared a variety of homo-ionic clays and subjected them to electric fields, measuring the initial electro osmotic flows (in time, the creation of hydrogen ions through electrolysis of water at the anode may have led to hydrogen ions exchanging onto the clay surfaces, thereby altering the zeta potential and hence electro osmotic flow). This allowed an investigation of the effect of different cations on electro osmosis; different cations led to different zeta potentials as expected (with hydrogen ions showing a strong shift from the natural value). However, the water flow noted with these different cations was not accounted for solely by water that would be expected to associate with the ion by hydration – other effects such as friction were expected to play a part. Electrochemical changes in soil have a marked long-term effect on the properties of the soil, with for example the introduction of different cations allowing changes in the soil structure, and possible improvements in soil behaviour. Substantial changes of pH, Atterberg Limits, water content and shear strength of illitic clay were shown by Esrig and Gemeinhardt (1967) in research into electro kinetic strengthening of soils. The introduction of calcium ions was used to illustrate the effect of cation exchange on electrochemical hardening. Variation of all these factors was noted across the sample, showing that the whole treated soil mass cannot be expected to attain the same levels of improvement, as position relative to the electrodes is an important factor. Indeed, shear strength and Atterberg Limits

increased from cathode to anode. This has implications for electro kinetic treatment of real sites, where suitable electrode positioning in three dimensions (rather than one dimension as in many laboratory tests) would be required. For example, in terms of electrokineticremediation, Maini *et al* (2000a) investigated positioning of electrodes in two dimensions, and showed that removal of contaminants was substantially greater in direct linebetween electrodes than it was away from this line. Alshawabkeh *et al* (1999) analyzed variety of different electrode configurations in terms of remediation efficiency, cost another parameters.

The fundamental role of ion exchange in electrochemical soil treatment was investigated by Gray and Schlocker (1969), who looked at the introduction of aluminum ions into arrange of clays. Often, the use of iron or aluminum electrodes will cause the introduction of the particular metal ions into the soil, and they examined the improvement in strength brought about by ion exchange rather than dewatering. The effect was found to be soil sensitive, with certain clays undergoing substantial changes of structure due to build up of hydroxy-aluminium inter-particle layers, whereas others were unaffected with aluminum remaining mainly as a constituent of the pore water.

More recent work by Lo *et al* (1991a) has concentrated on strengthening of soft clays in the laboratory with substantial increases in shear strength over short periods of time. They followed this up with fieldwork (Lo *et al*, 1991b), using a variety of electrode arrangements, and again showed an improvement in soil strength, although this was somewhat less than that achieved in the laboratory, an example of the problems of scaling up to prototype sizes. In a similar vein, Micic *et al* (2001) and Abdel-Meguid *et al* (1999) strengthened marine sediments using electro kinetics, in studies to develop a method for the improvement of offshore foundation capacity. In addition, the latter tested piled foundation capacities following electro kinetic treatment and found significant increases of up to 30% from a single treatment.

The effect of pH is extremely important in the soil, and can cause large changes in soil properties. Also, any pH change is likely to allow different species of soil flora and fauna to flourish (the distribution of micro-organisms particularly will tend to quickly adapt due to the relatively short reproductive period – see section 2.5). However, the effect of Ph change may be counteracted by any natural buffering capacity of the soil of hydrogen and / or hydroxyl ions, due to factors such as chemical reaction with or sorption to soil matter. The effect of pH on the zeta potential of soils (the electrical potential at the outer edge of the bound water layer on clay particles) was discussed by Eykholt and Daniel(1994), with reference to the effect that low pHs can have (described in section 2.1).Such effects are also described in depth by Yeung *et al* (1997), who particularly focus onzeta potential variation due to both pH and metal sorption, but also

describe the resistance of soil to changes of pH (acid / base buffering capacity).

Geo environmental Uses of Electro kinetics

Whereas the use of electro osmosis for stabilization of soft clays is at least well established, if not extensively used, the use of the range of electro kinetic phenomena tore mediate contaminated land is a relatively recent development. As with the geotechnical work, there are substantially more laboratory studies presented than work in the field. The attraction of this particular method of remediation is based on it being an *in situ* technique - it would be possible to decontaminate fine-grained soils without recourse to dig and dump or other methods.

Heavy metals are common pollutants in soils that have previously supported a wide range of industrial processes. Highly toxic species such as cadmium and arsenic pose a health hazard (Pearce, 2003), and so will have to be remediated. The process of electro migration is one choice for this task. However, the soil environment is a complex one; especially once the effects of electro kinetics are imposed (changes in the soil chemistry, such as the development of a pH gradient as discussed earlier, are particularly important). The simplicity of the basic technique then becomes vastly more complicated. There is a significant collection of research on the remediation of heavy metals, as the effects and processes will change from soil to soil. Açar and Alshawabkeh (1993) demonstrated and explained many of the effects that occur due to pH changes in soil subjected to an electric field. They describe lead and calcium removal from kaolinite and associated problems with precipitation of the contaminant at high pH near the cathode.

Accra *et al* (1992) demonstrated using phenol that there appears to be no retardation of the contaminant while it desorbs from the soil. This indicates that the electric field helps to make this an almost instantaneous process, although phenol is a relatively soluble organic compound. High levels of organic removal (this time using pentachlorophenol as well asphenol) were demonstrated by Kim *et al* (2000), again indicating that sorption and low solubility of pollutants does not appear to hinder substantially the electro kinetic remediation technique in the soil used.

EXPERIMENTAL APPARATUS AND MATERIALS REQUIRED

The second design retained many of the elements of the previous construction. The cartridge system was entirely retained, but new electrode chambers were constructed. Design diagrams are presented the electrode chambers were designed to be entirely self-contained and separate from one another. To perform a test, a soil specimen was placed on a base plate and the chambers plugged into either end of the cartridge. The protruding face on the chambers was permeated by approximately 30 small holes (approximately 4mm diameter) and covered by a semi-permeable membrane, and was long enough to ensure the membrane made good contact with the soil. The holes in this face allowed electrolyte fluid to make contact with the membrane over a substantial portion of its area, enabling a uniform water flow through the membrane. The membrane was a hydrophobic semi-permeable material called Dioramic (Daramic Inc., North Carolina, and USA). It was sufficiently hydrophobic that it did not permit the passage of water under working hydraulic gradients (i.e. when the chamber was filled with water)but allowed water to flow due to external forces (for example, that of electro osmosis).This was fixed in position using Araldite glue. Each chamber had several ports to allow fluid and pH control and the base plate had provision for voltage probes, as in the previous design.

Each experiment using this apparatus was performed with six soil specimens, all in cartridges and all prepared identically. Three were situated in the electro kinetic apparatus and subjected to an electric field, whilst the remaining three were used as control experiments. Testing in triplicate in this way provided replication of results.

Control of pH and Electrolyte Fluids

The electric field induced significant pH changes at both cathode and anode. In order to prevent, or control, the resulting changes in soil chemistry, the pH of either electrolyte could be controlled. Early attempts at this used manual addition of either sulphuric acid (at the cathode) or sodium hydroxide solution (at the anode). For the majority of experiments where pH control was implemented, however, commercial electronic pH controllers were used. Their design was such that when the pH was outside a specified range, the power supply was routed to an external device which could allow introduction of suitable chemicals (sulphuric acid or sodium hydroxide solution). The devices used were either solenoid valves or peristaltic pumps. This process took place in an external control chamber, as pH electrodes do not function well in the presence of an electric field.

Fluids were pumped into this chamber using peristaltic pumps, with the pH-adjusted fluid allowed to flow back to the electrode chambers under gravity. In experiments where the pH at both anode and cathode was required to be unchanged, all electrode fluids, both acolyte and catholyte, were pumped to the same chamber, with flow under gravity used to keep electrode chambers full. As the number of hydrogen ions produced equaled the number of hydroxyl ions, the combination of these fluids allowed recombination of these ions to reform water molecules (this method is similar to one used in Lee and Yang (2000)). This procedure also allows ions and other material removed from the soil under the electric field to be reintroduced.

Electro osmotic flow in a clay soil involves the flow of water from the anode to the cathode, except for under highly

acidic conditions. This may lead to a drop in fluid level at the anode, which, in addition to fluid loss by evaporation and
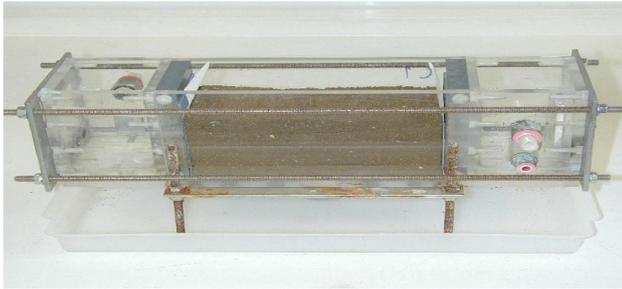

Figure 1: Photograph of small apparatus

electrolysis, cans cause significant water loss. In order to maintain water levels in anode chambers, a device known as a Mariotte bottle (Figure 3-10) was used.

Particle Size Distribution

The particle size distribution of soil has been calculated using two methods. The proportion of the soil particles falling within the larger grain sizes (above $63\mu m$) was determined by wet sieving through a range of sieve sizes (2mm, 600, 425, 300, 212, 125and $63\mu m$), using techniques described in Head, section 4.6.6 (1980a). The distribution of the finer clay and silt size particles was determined using a Cilas 920 automatic laser particle size analyzer (School of Geography and the Environment, University of Oxford).
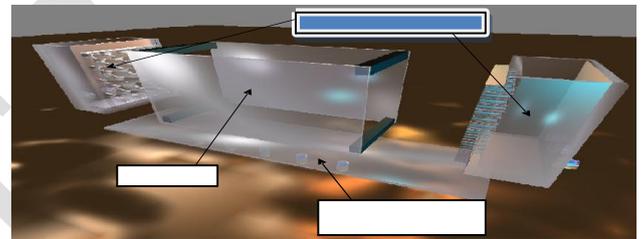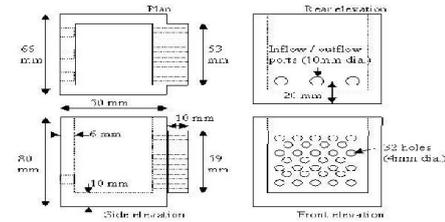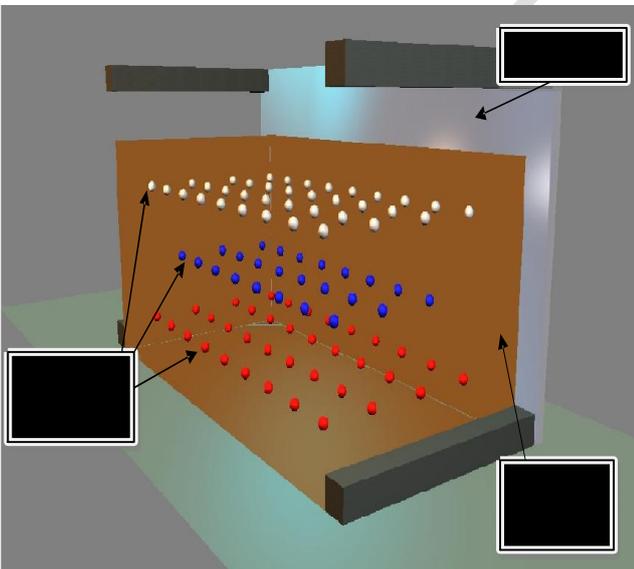

Figure 2: Second apparatus design
Maximum Dry Density / Optimum Moisture Content

The maximum dry density and corresponding optimum moisture content of this soil were determined using the Proctor Compaction Test (section 6.5.2, Head, 1980a). Soil was compacted into a mould of known volume using a manually applied ram, at a range of moisture contents. The variation in dry density achieved was plotted against moisture content. The maximum point on the trend line was taken to be the maximum dry density; the moisture content at which this

may be achieved was the optimum.

Pore Fluid Electrical Conductivity

The electrical conductivity of soil pore fluid was measured by preparing three soil cartridges as detailed in section 3.3, then contaminating them (see section 3.4) followed by centrifugation of soil samples from these cartridges at 4000rpm. Supernatant fluid was taken off, and the conductivity of this fluid was measured using a Hanna 8633conductivity meter.

Figure 3: Cut away diagram showing points of inoculation

II. EXPERIMENTAL PROGRAME AND RESULTS

PCP Extraction

In the majority of experiments, the total recovery of PCP, either with the ace to nitride extraction technique or the combined water / acetonitrile techniques used with radio labeled PCP, yielded on average approximately 80% of that thought to have been introduced. This compared PCP recovered from extractions on day 0 of an experiment with that calculated to be in the initial solution. It is likely that the remaining PCP was distributed between that strongly sorbed to the soil, and that which was 'lost' during contamination, for example due to association with the contamination apparatus(especially filter paper). Combustion of the soil following water and acetonitrile extractions recovered all the remaining PCP in soil, which was found to be only a small percentage of the missing contaminant. Therefore, it is thought that the majority of unaccounted-for PCP was lost during contamination, and soil extraction results give an accurate picture of soil conditions.

Effects of Electro kinetics on Soil

Data are presented that illustrate typical effects of

applying an electric field to soil on changes in soil properties (such as pH, moisture content and temperature) as well as voltage, current and electro osmotic flow.

Changes in pH

Changes in the pH of the electrolyte fluids were found in all experiments, due to water electrolysis at the electrodes. It was expected that the hydrogen and hydroxyl ions produced would migrate into the soil, primarily through electro migration and diffusion, changing the pH of the soil pore fluid, and this was observed many times. Typical results from a range of different experiment types are presented here. An example of uncontrolled pH changes is shown, followed by an investigation into the effect of manually controlling the pH through chemical addition. The effect of catholic pH control on the pH within the soil pore fluid is then demonstrated using results from two experiments, followed by the effect of two types of pH control at both electrodes - either mixing of electrolyte fluids or through current reversal. These results show the success of pH control (both chemical control and through current reversal) in later experiments in preventing substantial changes in the soil. The considerable changes that occurred without such controls illustrated the important effect of electrolysis in the use of electro kinetics.

Temperature

Changes in temperature within the soil mass during electro kinetic processing was monitored in experiments with non-radiolabel led PCP. It should be noted that the overall variability (including that of the ambient temperature / control) was due to the laboratory in which these tests were performed, which was not temperature controlled. It would be expected that the heating effect of the electric current would have large part to play in any increase in the temperature of electro kinetic specimens, and it was generally noted that the occurrence of temperatures higher than the ambient / control coincided with higher currents. In general, the highest temperatures were found to be associated with the anode end of soil specimens – this may be an effect of the current, Orit could possibly have been due to exothermic chemical reactions induced by pH changes there. Temperature measurements were discontinued in later experiments as it was

Figure 5: Electrolyte pH versus time

found that the constant current conditions at 10mA did not

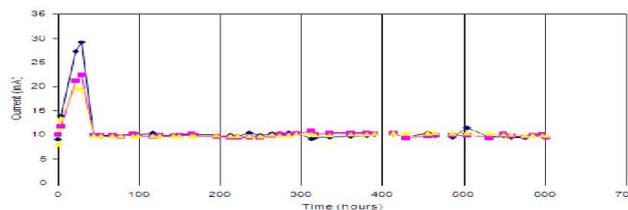induce noticeable temperature variations.

Figure 4: Electric current versus time I

Electro osmotic Flow

Flow induced by the presence of an electric field was found to have a certain amount of repeatability between experiments, in that flow would initially be slow to start, and then accelerate substantially, before reducing or stopping entirely.

The results of three different experiments are shown. Reasons for the slow initial flow may include lack of saturation of Vyon membranes (which was used in all the experiments presented) hindering flow. The reduction or cessation in flow is dealt with later in this section.
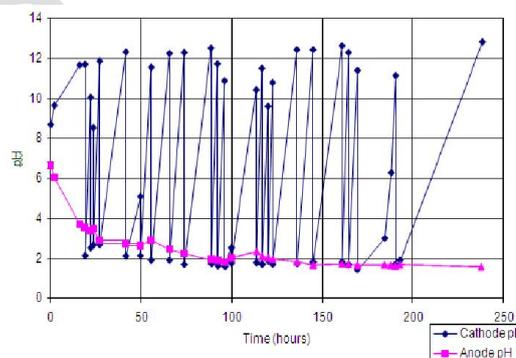
Figure 6: Electrolyte pH versus time

Moisture Content

An unsaturated soil state was considered important in these experiments as aerobic bacteria were being used to degrade the contaminant. The soil was designed to be un saturated at the start of each experiment, and so it was expected that changes in moisture content would be seen due to the action of electro osmotic water flow induced by the electric field. It is also possible that diffusion through membranes at the electrode chambers would have had an
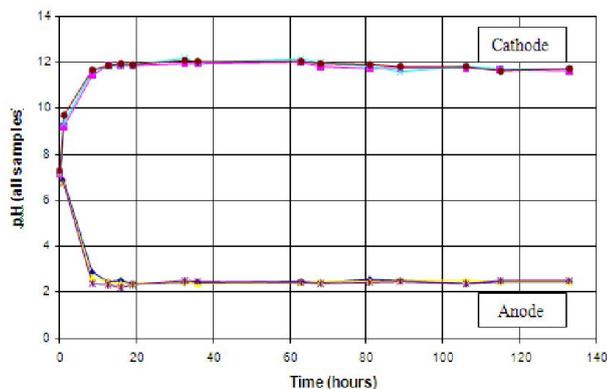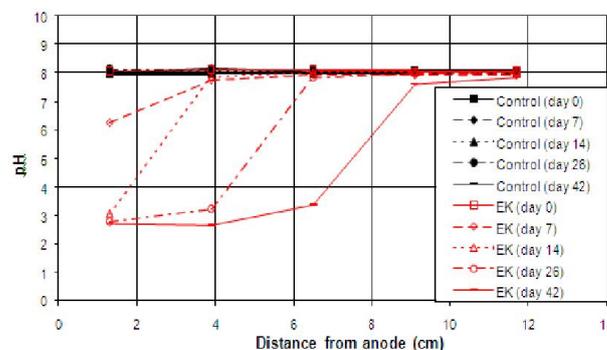
Figure 7: Variation of pH profile with time

effect (although this would have been hindered by the

extremely low hydraulic conductivity of the membrane material when Daramic was used). The suction present in the
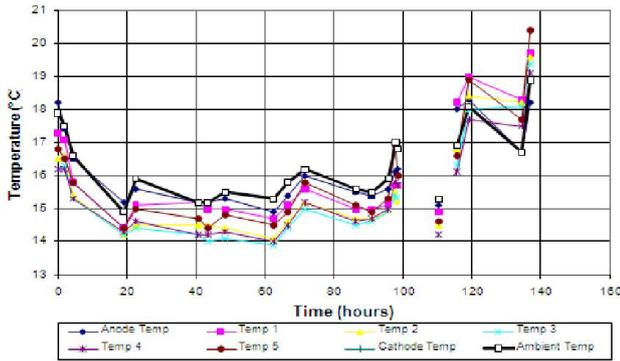

Figure 8: Temperature regime across specimen versus time

unsaturated soil would be expected to take up and hold the moisture presented by any of these methods, and so the moisture content would be expected to rise. Conflicting behaviour may have been generated, however, by such factors as the heating effect of an electric current or evaporation drying out the soil.

Pulsed and Low Current Experiments

Despite drier conditions initially in electro kinetic specimens compared to controls, pulsed-current electro kinetic processing in increased the moisture content in the former specimens. It was significantly higher ($p<0.002$) at positions 2 to 5 after 36 days than corresponding positions in control experiments.

Effect of pH control at both electrodes

In the experiments described above, the pH was controlled at the cathode chamber, but not at the anode. In experiments PI_18 and PI_20, it was controlled at both electrodes, and a constant current of 10mA was applied in each. Over the initial 66-day period in experiment PI_18 (during which time no field was applied), there was a slight increase in moisture content on average over all samples. With the application of an electric field for 29 days, however, a significant ($p<0.02$) increase of approximately 15% was seen across the electro kinetic specimens. The control of pH at both electrodes, therefore, can be seen to have been an important factor in large moisture content changes when an electric field was applied.

Control of Moisture Content and pH

This was done by removing the need for electrolyte fluids – electrodes were placed in direct contact with the soil. Despite regular additions of water to the soil adjacent to the electrodes in electro kinetic specimens (necessary to maintain current flow), no change in the initially identical control and electro kinetic specimens' moisture content profiles was seen after 56 days of testing.

The effect of an electric field on several aspects of the soil specimens, as well as PCP and its transport, has been modeled, in order to assess the validity of hypotheses for phenomena seen in experiments. The limited nature of the modeling meant that only physical behaviour was investigated. Changes in pH in the electrode chambers and soil due to water electrolysis, and transport and state of the PCP were the major factors modeled. The first aim was to compare the theoretical progression of an acid front into the soil with that seen in numerous experiments. The movement of PCP by electro migration and electro osmosis was also determined theoretically, based in part on the pH results. Comparison was made with experimental results, with particular attention paid to rate of transport and sorption. The hypothesis that a decrease in pH increased the prolongation of the PCP and thereby increased sorption to soil was investigated. The majority of this work has been performed in Microsoft® Excel. Although several of the equations used to model transport of chemical species have been taken from other sources, the majority of the model has been developed specifically for this project, with many relationships being determined empirically.

III. CONCLUSION

Preparation of samples for experiments was consistent throughout the project. The contamination procedure worked well, and generally satisfied the required criteria, in that it gave a sufficiently uniform distribution of PCP whilst allowing contamination of a pre-prepared soil specimen. The use of the sodium salt of PCP was justified in that it allowed the uniform contamination of ready-prepared samples in this way. Prorogated PCP could also not be dissolved sufficiently to allow a high enough level of contamination.

Inoculation of bacteria proved to be more problematic, with the number inoculated being more variable than hoped. In one or two cases, the number was much less than required. The target level of inoculation for earlier tests was 1×10^6 cfu / g dry soil. However, experiments with this number of bacteria did not always show degradation, possibly due to low survivability in some cases, so it was decided that this number was too low. Experiment PI_7 showed that there was little degradation of PCP in cartridges or flasks under these conditions, whilst the bacteria themselves died off within three weeks. From experiments PI_13i onwards, the target was 1×10^8 cfu / g dry soil and degradation was seen in each of the later tests.

The experiments were performed at laboratory scale, and as such, there might be expected to be significant differences between these experiments and conditions on a typical contaminated site. In this work, factors such as the homogeneity of the soil, temperature, uniform distribution of contaminant and degrading organism and electric field distribution could be controlled rather better than might be expected at field scale. For example, many contaminated sites can have a significant amount of made ground present, with Extreme heterogeneity in soil structure. The presence of metallic objects buried in the soil would be a significant hindrance to electro kinetic remediation, as the electric current would travel preferentially along these rather than through

pore water. Despite this, the electric current used in the majority of tests (10mA) was low, and therefore scaling up of the process might not be as difficult as if a higher current was employed. The principle of electro kinetic remediation will still hold at larger scale, and due to phenomena such as electro migration and electro osmosis, fine-grained soils at field scale are expected to show an improvement in bioremediation efficiency as was seen in these laboratory experiments.

IV. FUTURE WORK

An important step forwards would be the testing of the efficacy of this process with other contaminants and soils. With the knowledge gained from this project, it should be possible to determine a method of remediating a range of organic contaminants, given an understanding of the behaviour of these chemicals in soil. It may be that small changes in pH or moisture content could be implemented to great effect in order to best present a contaminant to degrading bacteria, and this could easily be performed using the techniques developed. It might also be of use to investigate the role of organic matter in this experimental system, in terms of sequestration of the contaminant, as well a transporting it (charged organic matter particles can be moved by electro kinetics). This could then be taken further, with the use of 'real' contaminated soils, possibly incorporating a 'cocktail' of different chemicals, both organics and metals. The optimum conditions for remediating such a mixture would be difficult to determine, as these two types of contaminant will behave very differently, and other factors such as heterogeneity and ageing will arise, but it may be that a combination of movement of metals and degradation of organic chemicals can be used.

Further testing without inoculated bacteria would be useful, to determine whether electro kinetics can enhance natural attenuation significantly. This would be an important step forwards as it can be difficult to ensure a good distribution of degrading bacteria and if existing soil organisms could be used then this would remove this problem.

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