

Electrochemical remediation of CCA polluted soil

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Introduction

Heavy metal polluted soils pose a risk to water quality and to health of humans at normal site use, and thus remediation of these sites is highly relevant. Remediation of heavy metal polluted fine-grained soils is difficult or even impossible by more traditional methods like pump and treat technologies or soil washing methods. Electrochemical remediation methods, on the other hand, are very effective in fine-grained soils, e.g. (Ho et al. 1995) or (Reed and Berg, 1994) and this paper deals with such method.

Electrochemical soil remediation methods are based on the transport processes that occur when an electric current is passed through a soil. The electric current tends to pass the soil in the micropores due to the lower resistance here, and this means that the current is acting exactly where the heavy metals are mainly adsorbed in the soil. Thus the electrochemical methods are especially suitable for fine-grained soils.

The transport mechanisms of major importance to the electrochemical soil remediation methods are electromigration and electro osmosis (e.g. Acar, 1993). In the soil the current is carried by ions, and the anions will be transported toward the anode and the cations toward the cathode. This transport of ions in the electric field is electromigration. Electro osmosis is movement of pore water in the soil in the applied electric field. The negative charge on the surface of most soil particles causes an accumulation of positively charged cations near the surface in the diffuse electric double layer. Under the action of the electric field these cations will give a net flow of ions in the direction of the cathode, and by this water is forced to move towards the cathode.

Form wood preservation there has over the years been some serious pollution of soil by Cu, Cr and As. The present paper is focused on the remediation of these soils by electrochemical methods.

Development of electrochemical soil remediation methods

During the 1930s the effects of applying an electric potential to a soil were first used. Puri and Anand (1936) tested the value of applying an electric potential across a soil for removing sodium ions in order to improve the soil quality. Casagrande (1948) was the pioneer who started to use the electro osmotic effect for dewatering fine-grained soils. He used the technique to stabilize earth masses where the classical methods had failed.

Years later Bruch and Lewis (1973) proposed to extend the usefulness of the electrokinetic phenomenon to being a tool in ground water pollution control in soils of low hydraulic permeability. They based the proposal on theoretical considerations. In 1980 the movement of heavy metals in a soil in an applied electric field was first reported by Segall et al. (1980), who tried to dewater a dredged material disposal site and its embankment foundations in order to increase the capacity of the disposal site. In the liquid samples from the process they found various heavy metals. Even though this was just noted in the actual work, this finding may have given different researchers the idea of using an applied electric field for soil remediation, because in the end of the 1980s more teams started to develop electrochemical remediation methods. The majority of the published research conducted in the late 1980s and early 1990s originated from the Dutch company Geokinetics (Lageman 1989), and research teams from Louisiana State University (Acar et al. 1990), Lehigh University (Pamucku et al. 1990) and Massachusetts Institute of Technology (Probstein 1993).

The number of papers published in the late 1980s in the field of electrochemical remediation was less than 20, but during the 1990s more than 400 papers have been published, and this high rate is continuing. The increasing number of papers in the field indicates an increasing interest. Among the scientists and companies working in the field a network has been established, and in this network a common reference list is made (Ottosen and Hansen, 1999). The numbers given above about the publications are taken from this list. In 2003 this network constituted of 85 teams mainly from Europe and Northern America, but also teams from Asia, Southern America and Australia have joined the network.

The electrochemical remediation methods have in common that they are based on the transport processes occurring when an electric field is applied to a soil. The methods differ though in several aspects as e.g. design of electrode units and organization in the soil and target pollutants. The methods where an electric field is applied to a soil to remove chemical species are variably called e.g. electrokinetic remediation, electroreclamation, electrokinetic extraction, electrokinetic soil processing, electrochemical decontamination or electrodialytic remediation.

Quite extensive work has been performed on laboratory scale with the aim of increasing the knowledge of the processes of importance to the electrochemical methods. Some laboratory studies have been performed with soils polluted with CCA from wood preservation industry and these studies are outlined in chapter 3 below. Some methods are now in the state of field implementation while the method that is in focus in chapter 4 and 5 in this paper is fully implemented as a field method.

Laboratory results of electrochemical remediation of soil polluted from wood preservation

To test the feasibility of in-situ removal of arsenic from soil by electro-reclamation several laboratory tests were conducted by the Dutch company Geokinetics during the late 1980's and early 1990's. The samples originated from wood preservation sites in the Netherlands, Denmark, and Sweden. One test was done with arsenic contaminated soil from a chemical plant at Rijsbergen (NL), where pesticides were produced. An overview of the results of some of these tests is presented in the following table.

Location	Soil type	pH at start	Conc. at start (mg/kg)	Conc. at end (mg/kg)	Decrease (%)	Energy induced (kWh/m ³)	Decrease (% per kWh)	Duration of test (days)
Ter Apel (NL)	clay	6.1	410	190	54	50	1,07	52
Rijsbergen (NL)	fine sand	4.5	150	60	60	80	0,75	11
Hjältevad (DK)	sand	6.7	1140	780	32	73	0,43	21
Stenholtsvang (DK)	fine sand	6.5	2580	1500	42	270	0,16	83
Nissafors (SE)	fine sand	6.2	425	< 20	95	500	0,19	125

Table 1. Some results of electro-reclamation laboratory tests with soil contaminated with arsenic

Remediation companies like Geokinetics (which was acquired by Hak Milieutechniek BV at the end of the 1990s) run these laboratory tests on request of clients, who are looking for ways to remediate their polluted sites. Important parameters of these tests are therefore concentration decreases as a function of energy inducement and/or remediation time. There are two types of tests:

- Standard test, with 15 - 20 kg of soil sample, which is run for 2 to 4 months. In the standard test, all soil material is treated in one laboratory set-up. This set-up is used when the client has supplied ample data on origin of the sample and its contaminants. The test gives accurate data on optimum pH, energy requirements and remediation time. Sampling frequency amounts to 1 week. At the time of analysis, five samples are taken and analyzed: anode side, middle, and cathode side and precipitates in cathode and anode compartments.
- Turbo test, with 300 gr of soil sample, which is run for 1 to 3 weeks. In the turbo test the soil material is divided over six small containers, each of which is getting a different energy supply and/or electrolyte solution (different inorganic or organic acids). This kind of test is used if none or only scanty data about origin and type of contaminants are known or the client wants to have a quick scan of the possibilities of deploying electro-reclamation. It gives less accurate data on energy requirements and remediation time, but gives an indication whether the contaminants can be mobilized at all, using a specific kind of electrolyte solution. One week of testing is also possible when relatively high current densities are used. Every container is sampled once at the end of the test. From each soil compartment 5 samples are taken and analyzed: anode side, anode-middle, middle, cathode-middle and cathode side. From both cathode and anode compartments precipitates are made and analyzed.

The most reliable results are obtained when the two tests are combined. A turbo test followed by a standard test, which is run using the electrolyte solutions, which came out best during the turbo test.

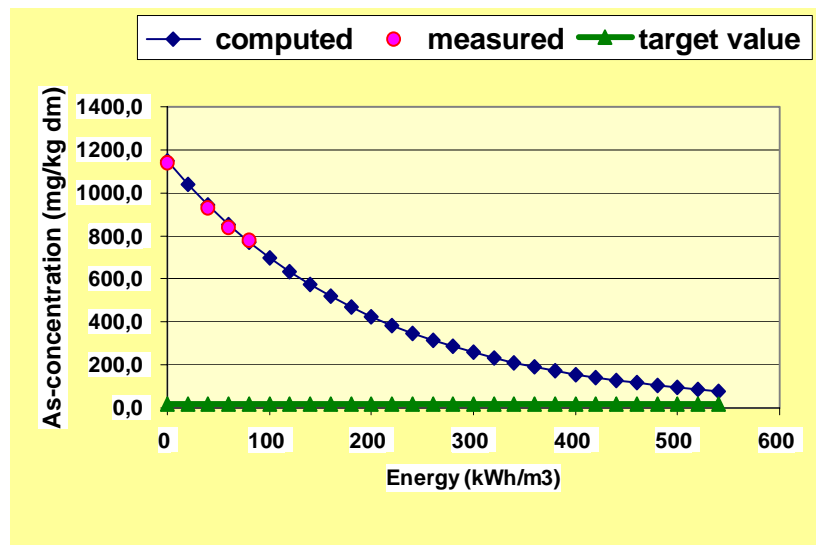


Figure 1: Example of result with combined laboratory results and computer calculations

From table 1 it can be observed that the sample from Ter Apel shows the best removal efficiency, followed by Rijsbergen, while the tests with samples from Stenholtsvang and Nissafors show very low efficiencies.

Other conclusions from these tests were that at low pHs (< 2.5) arsenic behaves, virtually without exception as a cation. All other heavy metals present in the samples showed high removal efficiencies. Although the sample from Nissafors had a low cation exchange capacity (CEC), it took 500 kWh/m³ to reduce arsenic concentrations below 20 mg/kg. In this case the arsenic species was not very mobile, even at low pH

Experimental results in the two thesis Ottosen (1995) and Ribeiro (1998) both underlined the fact that As is not always very mobile in an applied electric field in soils polluted from wood preservation, and that the removal of Cu and Cr was faster in these soils. Especially Cu removal can be very efficient (Ottosen et al. 1997), (Ribeiro & Mexia, 1998). To enhance the remediation of As it was tested in laboratory scale if an addition of ammonia prior to the application of electric current would improve the remediation (Ottosen et al. 2000). The idea was that As should be mobile in the alkaline environment and that Cu could form the mobile $[\text{Cu}(\text{NH}_3)_4]^{2+}$ complexes (the actual soil for this investigation was not polluted with Cr). The results confirmed the expectations and As and Cu was removed at the same time. When the soil is polluted with As, Cu and Cr ammonia is not a good enhancement solution, since Cr is not mobilized. In this case ammoniumcitrate can be used in stead, which was shown in laboratory scale in Ottosen and Villumsen, 2001.

Electroreclamation

Principles

It is difficult to recover ionic contamination from ground and groundwater because soil is a powerful ion exchange medium. Ions of, for example, heavy metals, cyanide, arsenic or other polar species are adsorbed on contact by soil particles. Flushing alone will not recover this contamination: to desorb and mobilise it, the pH must be changed. However, flushing with strong acids usually destroys the basic soil structure. An elegant solution is electrokinetic acidification that uses the technique of electro-reclamation.

Electro-reclamation belongs to the group of so-called physicochemical remediation techniques. Under the influence of an electric field, three electrokinetic phenomena occur:

1. Electromigration, i.e. the movement of ionic species in pore water or groundwater.
2. Electro osmosis, i.e. the movement of water from anode to cathode.
3. Electrophoresis, i.e. the movement of charged particles.

In the following sections, these phenomena will be described in more detail.

Fundamentals of electromigration

When inert electrodes are placed in water and a direct current is passed through them, changes occur at the anode and cathode according to the scheme presented in equation 1.



At the anode or positive electrode, electrons are stripped from the water molecules, oxygen is evolved, and protons (H^+) are formed and travel through the electrolyte towards the cathode. Meanwhile, the cathode is donating electrons to water molecules, creating hydroxyl ions and liberating hydrogen gas (Eq 2).



Note that the two reactions (the generation of protons at the anode and of hydroxyl ions at the cathode) are in balance and there is no net change in the pH of the electrolyte. In a liquid electrolyte, so much mixing occurs that the local changes in pH around the anode and cathode are difficult to detect unless the electrodes are well separated. There is little or no mixing in soil, sludge, concrete and gels, so because the remixing of the electrolyte is inhibited, the area around

the anode becomes acidic and the area around the cathode becomes alkaline. This has a significant impact on the chemical process in soil.

Soil as an electrolyte

Most soils are conductive because they contain dissolved ions, such as calcium, magnesium, sodium, potassium, (bi)carbonate, some soluble fatty acids, nitrate, phosphate, sulfate and chloride ions. Soils that seem to be dry usually have a moisture content of more than 5%, which is sufficient to provide a continuous path for these ions to move along. This is essential for plants, as the roots need access to these nutrients and they extract the nutrients by ion transport across membranes.

In terms of their contamination and subsequent remediation, the most significant feature of natural soils is their high ion exchange capacity. In their metallic state, heavy metals such as cadmium, lead, iron, and zinc corrode and form salts and bases, which occupy cationic sites on soil particles. In some cases, land is contaminated by aqueous spills of heavy metal ions directly from plating shop wastes or by airborne pollution from metal smelters. Soil has the capacity to immobilize significant quantities of heavy metal ions; heavy metal concentrations of as much as 2-3% have been found in the topsoil around lead smelters.

Soil is made up of several components derived from the weathering of rocks and the accumulation of organic matter from the growth and decay of plants and other organisms. The more permanent background assemblage of organic materials in soil is supplied by humus, a complex mixture of humic and fulvic acids with a highly functionalized aromatic structure. These materials have a very high ion exchange capacity and are usually closely bound to clay particles. Clays have an ion exchange capacity; they derive this capacity from the basic silicate structure, which acts like a Lewis acid. Note that the basic unit contains excess oxygen atoms, which are able to form chains, sheets or three-dimensional networks. The end groups in such networks provide vacant sites for cations. Silicates as complex as this behave as ionic exchangers for metal cations. Multivalent cations attached to silicates can also provide sites for the attachment of anions such as arsenites, sulphates, cyanide, and carbonates and hydroxide ions.

The interaction of electrochemical and electrokinetic phenomena

When anodes are placed in soil and a direct current is passed between the electrodes, the amount of available H^+ is raised significantly. The H^+ ion travels much faster than other ions and carries a disproportionate share of the total current. The average mobility of an ion is $5 \cdot 10^{-8} \text{ m}^2/\text{U.s}$, where U = drop in potential, in Volts. Along the gradient in potential between the electrode pairs a hydrogen ion moves almost twice as fast ($33 \cdot 10^{-8} \text{ m}^2/\text{U.s}$) as a hydroxyl ion ($18 \cdot 10^{-8} \text{ m}^2/\text{U.s}$) and 5 -6 times

faster than metal ions. In soil however, the hydrogen ions are quickly mopped up by the ion exchange sites on the soil particles; as a result, the metal ions are displaced into the electrolyte. Even if the soil picks up the metal ion on a new ion exchange site, there is a net drift towards the cathode, as the ion exchange sites fill up with protons emanating from the anode. Metal ions will be in equilibrium with other available sites on the soil in front of the proton sweep.

A similar but slower drift of hydroxyl ions moves from the cathode, displacing anions adsorbed onto the soil particles. Free anions in the water electrolyte will be in equilibrium with available sites on the soil. Note, however, that these sites compete with OH^- ions created at the cathode, and so there will be a net drift of displaced anions to the anode due to electromigration. As a result, the soil acidifies from the anode towards the cathode and if no measures are taken, practically all the energy will be used for the transport of H^+ ions. Likewise, there is increased alkalinity towards the anode, ultimately leading to the precipitation of metal hydroxides in the soil.

Electromigration will occur with any species that will form ions in aqueous environments. The process can therefore be applied to contaminants such as:

- Inorganic anions and cations.
- Organic carboxylic acids, phenols.
- Sulfonated aliphatic and aromatic compounds, such as certain dyestuffs.
- Detergents and some pesticides, such as paraquat and diquat.

Electro osmosis

Electro osmosis is the movement of pore water or groundwater under the influence of a DC field. With electro osmosis the direction of flow is from anode to cathode. In some cases one can also observe a flow from cathode to anode. This phenomenon is known as electro-endosmosis. Electro osmosis is influenced by the following factors:

- The mobility of the ions and charged particles present in the pore fluid, including those ions and particles entering the pore fluid via ion exchange.
- The hydration of ions and charged particles present in the pore fluid.
- The electrical charge and direction of movement of ions and particles, resulting in net water transport.
- The ionic strength or ion concentration.

- The viscosity of the pore fluid.
- The temperature.

The average electro osmotic mobility is $5 \cdot 10^{-9}$ m²/U.s, where U = drop in potential, in Volts. To calculate the electrical power required in order to remove water from a certain volume of soil, the following should be known:

- The soil porosity.
- The desired final water content.
- The specific conductivity of the soil.
- The intended use of the treated soil.
- The time available for the treatment.
- The safety measures required given the maximum permitted electrical potential.

Note that the volume of water removed per unit of time is directly proportional to the electrical power used per unit of volume of removed water. Thus, the faster the water transport, the more power is necessary to remove the same volume of water.

Electrophoresis

Electrophoresis, or cataphoresis, is to do with the movement of particles under the influence of a DC field. The term “particles” includes all charged particles, such as the colloidal, clay and organic matter particles suspended in the pore fluid. The movement of these particles is similar to the movement of ions. In the pore fluid of clay soils, the particles participate in the transfer of electrical charges and influence the electrical conductivity and the electro osmotic flow.

Clay minerals can polarise in two ways. The first is the permanent dipole moment, which results from the structure and depends on the atomic masses. It is oriented parallel to the long axis of the clay particles. The second polarity is perpendicular to the first one and is a result of the external electrical field. It depends on the polarisation capacity of the electrical double layer. Thus, the mobility of clay particles depends on the combined action of these two moments and is therefore low, varying between $1 \cdot 10^{-10}$ and $3 \cdot 10^{-9}$ m²/U.s.

In-situ arsenic removal

Carrying out electro-remediation in the field

The key elements of an electrokinetic installation are as follows:

- Ion-permeable electrolyte casings (100-120 mm diameter) are placed in the contaminated medium and connected to a centralised electrolyte management system (EMS). Each casing has an electrode inside. The result is alternating rows of anodes and cathodes. Electrolytes are circulated in a closed loop between the electrode casings and the EMS. Via these electrolytes, pH is maintained at a predetermined value. The electrodes are then energised. The water in the electrolytes is electrolysed, forming H^+ ions and $O_2\uparrow$ at the anodes, and OH^- ions and $H_2\uparrow$ at the cathodes. These ions are then made to migrate through the casing into soil to generate a temporary and localised pH shift, which desorbs the contaminating ions. No acids are pumped directly into the soil.
- Once desorbed, the contaminating ions migrate to their respective electrodes, under the influence of the potential applied (electromigration). The anions migrate to the anodes, the cations to the cathodes. Here they pass through the electrode casing walls and are taken up by the circulating electrolytes.
- Critical for the control of system performance is the careful management of the pH and other electrolyte conditions within the electrode casings.
- Contaminants are recovered from the circulating electrolytes by both precipitation and filtration, by electrochemical ion exchange (EIX), or by other ion-collecting systems.

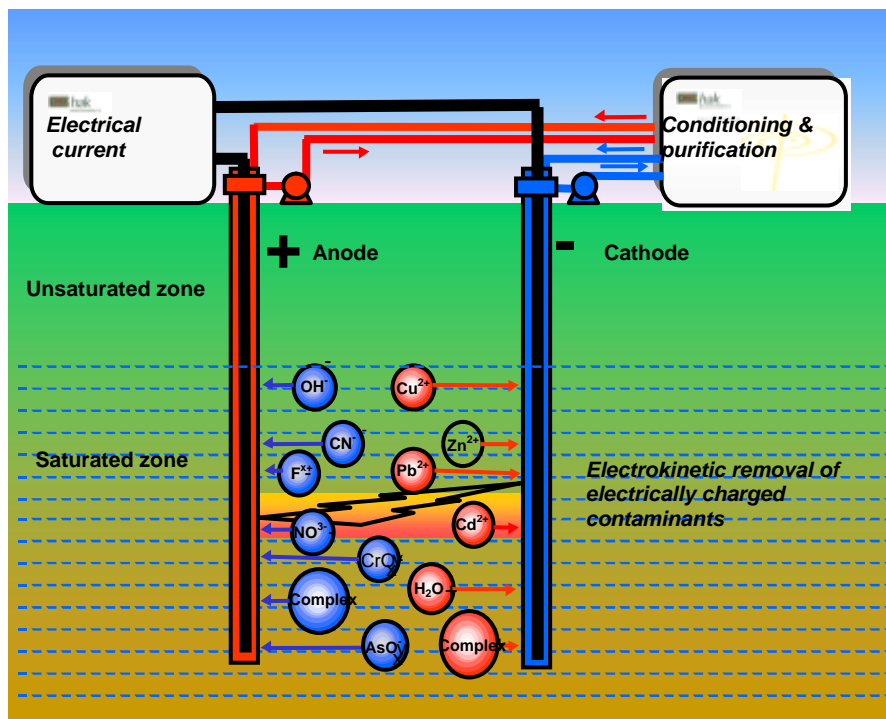


Figure 2: Schematic presentation of in-situ electro reclamation

When the electrical conductivity of the electrolytes has reached approximately 20 S/m, the electrolytes are pumped into a connected treatment installation. Depending on the type of contaminants, they are treated:

- With lye (NaOH), to precipitate metal hydroxides, which are then removed by filter press. The amount of filter cake produced depends not only on the concentrations of the heavy metals but also on concentrations of iron, alkaline earth and alkaline ions, carbonate, bicarbonate, magnesium etc. As a rule of thumb, 0.05 to 0.1% of the total volume of treated soil is ultimately collected as waste.
- By electrochemical ion exchange (EIX®), a process developed by AEA Technology (Harwell) that uses electrochemically driven adsorption and desorption to remove ions from dilute electrolytes
- By using a so-called EnViroCell® for specific heavy metals and cyanide.

The electrolyte management and purification systems are housed in containers, together with the energy supply. When necessary, electricity cables, circulation ducts and pipes can be installed underground.

Arsenic remediation project

A full scale commercial in-situ arsenic remediation was carried out in 1989 by Geokinetics BV at the site of a former wood impregnation plant in Loppersum a small city in the Northern part of the Netherlands. The site was underlain by heavy clay soil containing arsenic levels up to 400-500 mg/kg to a maximum depth of 2 m below ground surface (bgs). The source of the pollution was attributed to $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ or "Super Wolmansalt D", used for preservation of timber and wooden poles. The pollution was confined to an area of 10 x 10 m, contaminated to a depth of 2 m bgs, and an adjoining area 10 x 5 m, contaminated to a depth of 1 m bgs.

When this project began, the resistivity of the clay was 10 Ohmm and soil temperature at a depth of 0.5 m was 7 °C. After 3 to 4 weeks, temperature had risen to an average of 50 °C, while soil resistivity decreased to 5 Ohmm. The original drop in potential of 40 V/m decreased subsequently to 20 V/m with an average current strength of 4 A/m² (the total cross-sectional area being 110 m²).

Changes in arsenic concentration were monitored at 10 fixed sampling locations and numerous randomly distributed points. After 65 days roughly three-quarters of the area showed arsenic concentrations below the target value of 30 mg/kg. During the remediation process it had been

observed that in the remaining one quarter of the area the decrease in arsenic concentration proceeded much more slowly than at other locations. Electro-reclamation continued for 15 more days, but no significant changes were observed. Because of time constraints related to the planned building activities on the site, it was decided to stop the electro-reclamation process and excavate the remainder of the soil, which showed arsenic concentrations above the target level. During excavation, many metal objects such as tins, barrels and reinforcing rods were found - shiny without any rust, and in many cases partly dissolved. These objects functioned as preferential flow paths for the electrical current, thereby delaying the movement of the pollutants in their vicinity, until the objects had been dissolved completely. Some 40 m³ of soil had to be excavated. Periodic treatment of the electrolyte solutions yielded 800 kg of filtrate, which contained 38 kg of arsenic. The excavated soil contained 14 kg of arsenic. In the underlying table, results of this project have been summarized.

Soil type	<i>Heavy clay</i>	Average concentration at end	<i>10 mg/kg</i>
Depth of pollution	<i>2 m</i>	Arsenic removed by electro-reclamation	<i>38 kg</i>
Maximum concentration at beginning	<i>500 mg/kg</i>	Arsenic removed by excavation	<i>14 kg</i>
Average concentration at beginning	<i>115 mg/kg</i>	Energy consumption	<i>150 kWh/ton</i>
Maximum concentration at end	<i>29 mg/kg</i>	Remediation period	<i>80 days of 18 hrs.</i>
Cost (Turn key)	<i>€ 64,000</i>		

Table 2. Results in-situ electroreclamation of inorganics at Loppersum

An important observation during remediation was that hydraulic permeability of the clay had increased. Normally it took several days for the groundwater to fill up a monitoring well pumped dry. After approximately one month of electro-reclamation, it did not take more than a few hours. During later projects this was observed again. At first it was thought that a reorientation of the clay minerals was responsible for this phenomenon, but as it also occurred in fine sandy sediments, it had to be attributed to the decrease in viscosity of the groundwater. Measurements showed that for every 20 °C increase in temperature, permeability almost doubles.



Figure 3: The plant in Loppersum

Discussion and conclusion

The foregoing chapters have shown that in-situ electro-reclamation of present and former wood preservation sites contaminated with arsenic and other heavy metals is feasible. However, there are pronounced differences in terms of energy supply and remediation time, mainly as a result of the buffering capacity of the soil and speciation of arsenic during changes in pH. At $\text{pH} < 2.5$ arsenic behaves like a cation and will move together with the other heavy metals towards the cathode. At higher pH, arsenic species might behave as an anion and move into the opposite direction. In cases of soils with a low to medium cation exchange or buffering capacity (CEC), pH may be lowered electro kinetically to the required pH. The cost of energy will amount to 15 - 25 % of total remediation cost, which is normal for in-situ electro-reclamation projects. Soils with a high CEC require significantly more energy to lower pH electro kinetically. In that case pre-acidification of the soil material might be considered. This option means that the soil cannot be treated in-situ, but should be excavated, mixed with acidified water and remediated on site within a temporary lagoon. Within the lagoon electrodes are installed in the same manner as with in-situ electro-reclamation. After clean-up the soil can then be returned to its original location.

The final choice to remediate a site by electro-reclamation will ultimately depend on a comparison between this technique and other techniques, if these are available. For in-situ remediation of clay, or soils rich in argillaceous material (> 20 %) contaminated with arsenic and other heavy metals, there are no remediation options other than electro-reclamation. For sandy soils, ex situ soil washing is an option. The soil at Nissafors for example (mentioned in chapter 3) has been cleaned using a mobile soil washing installation. In this case the required 500 kWh/m³ needed to remediate the site by in-situ electroreclamation, could not compete with the cost for ex situ soil washing.

For the site at Rijsbergen (also mentioned in chapter 3), the client choose for excavation of 25,200 m³ of soil up to 9 m bgs and installation of a bentonite/cement wall from 0 to 23 m bgs. Two industrial buildings and a private house had to be demolished and several gardens had te be excavated and filled in with clean soil. Groundwater is still being remediated with pump and treat. Total cost have already reached 15 million euro, while the original proposal of Geokinetics for in-situ electro-reclamation of the site up to a depth of 18 m bgs amounted to 8 million Euro.

We may finally mention that apart from the arsenic remediation project at Loppersum, several electro-reclamation projects with other heavy metals, like lead, copper, cadmium, chromium, nickel and zinc, have been completed during the past 15 years. At present an in-situ electro-reclamation project is running underneath a galvanizing plant, where ground and groundwater has been polluted with nickel up to 6 m bgs. Electrodes, cables and ducts for the electrolyte management system have been installed inside the production building, flush with the liquid tight floor, enabling undisturbed production.

Contrary to the general opinion that electro-reclamation is an emerging technique, 15 years of applied electro-reclamation in the Netherlands, and to a lesser extent in the USA, have proven that electrokinetic remediation is a viable clean-up technique, which not only can compete with other techniques, but is more often than not, the only effective clean-up option.

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