

CHALLENGES AND HOPES FOR SCALING UP AN ELECTRODIALYTIC REMEDIATION METHOD FOR TREATING CCA CONTAMINATED SOIL

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INTRODUCTION

Soils polluted with heavy metals and arsenic compounds are a challenge for contaminated soil handling procedures. Traditionally, a remediation of such material involves excavation of the contaminated soil, followed by an immobilization of metal contaminants by solidification/stabilization technology prior to disposal of the material in a permitted landfill site or on-site. However those remedial actions based on solidification/stabilization technology are no longer considered a permanent solution because the pollutants are not removed from the contaminated media resulting in a need for future monitoring of a pollutants faith on site and questionable longevity of the solidified/stabilized materials (Dermont et al., 2008).

Therefore, there is a great need for a novel treatment technology that can efficiently separate heavy metals and arsenic from the soil. Among the technologies that have been in focus recently are: 1) flotation; 2) soil washing; 3) phytoremediation and 4) electrokinetic methods. All processes are affected by soil properties and speciation of the polluting compounds and process efficiency may have to be enhanced by chemicals addition, like surfactants, which were reported to improve washing and flotation (Reynier et al., 2013; Torres et al., 2012).

Another approach could be a combination of two technologies, like presented in this paper uniting soil washing and electrodialytic remediation. Application of the electrodialytic (ED) remediation developed at the Danish Technical University has been well described in last decades, but was mostly applied as laboratory and bench scale experiments with very limited mass of treated material (Ottosen & Hansen, 1992). The ED method combines the technique of electrodialysis with the electromigration of ions in the polluted soil. During the process it is possible to separate ions from soil with help of applied electric field to electrodes isolated from soil by ion-exchange membranes. Anions, like arsenite and arsenate, are removed to compartment with anode through a anion-exchange membrane and cations, of copper and chromium, are moved into a compartment with cathode through a cation-exchange membrane. Laboratory experiments proved that the ED remediation process applied on saturated soil was very effective for the removal of copper and to a certain extent chromium (Hansen et al., 1997). However As was not removed significantly in this system. This is mainly because the soil is acidified during the remediation, and in acidified soil As will mainly be present as uncharged species (H_3AsO_3 in case of As(III) and H_3AsO_4 in case of As(V)). Such uncharged species are not transported in the applied electric field. Faster remediation was showed to take place in a stirred suspension of soil than in the water saturated soil (Jensen et al., 2007; Ottosen et al., 2012). Moreover, increasing pH to pH=3-4 significantly increased arsenic species removal reaching a removal efficiency of 60% (Sun et al., 2012). It was also shown that remediation of the fine fraction of soil was faster and more energy efficient than remediation of the whole soil. Therefore if the contamination is primarily bound to the fine fraction of the soil, which is often the case, it may well be feasible to first implement soil washing to separate the less contaminated coarse fractions from the highly contaminated sludge, and then subject the fine fraction (sludge) to ED remediation, which would then be more efficient both due to the smaller mass to be treated and due to the higher efficiency of the treatment (Jensen et al., 2006). The concept was introduced earlier (Jensen et al., 2012). Another issue for application of the ED remediation might be membrane fouling, that is a major factor to consider when applying electrodialysis, especially in a full scale. Fouling is the accumulation of undesired deposits on the membrane surface which affects its performance. Among the causes of these deposits scaling (salt precipitation) and clogging (deposit of macromolecules, colloids) are anticipated to influence the ED process the most (Araya-Farias and Bazinet, 2006; Mondor et al., 2009). Therefore the use of any chemicals agents shall be limited, especially surfactants during the soil washing procedure as the

anion exchange membranes were reported to be sensitive to anionic organic compounds (Lee et al., 2008)

This work presents some results from laboratory and pilot plant investigations of the technology duet for decontamination of one of several former wood preservation plant left polluted by Cu, Cr and As (CCA) in Denmark. CCA was originally used in the impregnation process, and hot spot concentrations of copper, chromium and arsenic (1000-2000 mg Cu/kg of soil, 300-600 mg Cr/kg and 200-1200 mg As/kg) are clearly above the Danish clean soil criteria of 500 mg Cu/kg, 500 mg Cr/kg and 20 mg As/kg. The Collstrop site only poses minimal risk to the nearby recipients and the groundwater resource in the area, but it is a standard site for wood preservation. The site is therefore used by The Capital Region of Denmark for testing remediation methods. The objective is that these methods in the future can be applied on similar sites where remediation is required due to risks towards groundwater and recipients.

The aim of this study is to discuss the feasibility of the soil washing and ED process combination and its implementation for CCA contaminated soil treatment. The pilot scale investigations gave an opportunity to evaluate especially the ED process, to define challenges that it may face in the full scale and foremost outline directions for improvements to develop a cost-efficient soil remediation technology.

METHODOLOGY

Soil sampling

Soil samples were obtained from two locations at the Collstrop contaminated site in Hillerød, Denmark. The samples differed in contamination level. Soil samples from location A were mainly used for lab scale investigations, and soil fines were obtained by wet-sieving the original soil through a 63µm sieve with water and soil from location B was excavated and washed in a soil washing facility Umweltschutz Ost GmbH in Germany. The initial concentrations are shown in Table 1. The soil samples were air dried at room temperature prior laboratory experiments, while for pilot investigations it was used as obtained from the soil washing facility.

During the soil washing in the German facility a high molecular weight flocculant, Magnafloc 120 L, was used to treat recycled water. Therefore the soil from location B was also sieved in laboratory by wet-sieving and obtained fractions were mixed to get a fraction that mimics the soil washing without undesirable compound.

Table 1 Heavy metals and arsenic content in Soil samples from the two different locations

	Location A (the hot spot location) – for lab scale investigations		Location B – excavated and washed in Germany		
	Soil-A	Fine-A	Soil-B	Fine-B	Fine-M
Soil sieving results [% m/m]					
X > 4mm	2.5		0.6		
4mm > X > 1mm	1.9		1.5		
1mm > X > 250 µm	26.7		12.9	1.9	
250 µm > X > 125 µm	39.2		26.0	5.3	7.6
125 µm > X > 63 µm	16.7		23.4	17.3	9.7
63 µm > X	13.0	100	35.6	75.5	82.7
Heavy metals and arsenic content [mg/kg TS]					
Cu	1130 ±90	8500 ±1600	183 ± 80	260 ±10	480 ±4
Cr	300 ±20	1900 ± 200	10 ± 5	200 ±10	50 ±0.5
As	214 ±5	2080 ± 400	220 ± 80	1040 ±20	740 ±40

Analytical procedures

Solid samples of 1.0 g of dry soil and 20.0 mL (1:1) HNO₃ were heated at 200 kPa (120 °C) for 30 min according to Danish Standard 259. The liquid was separated from the solid particles by vacuum through a 0.45 µm filter and diluted to 100 mL. The concentrations of metals and As in prepared liquid samples were measured with ICP (Varian 720-ES, software version: 1.1.0). Examination of the pH-dependence of metals and arsenic mobilization from soil used the following procedure: 5.0 g of dry soil and 25 mL of acid, HNO₃ in various concentrations, or basic, NaOH in various concentrations, solutions were used to make soil suspensions that were mixed for 7 days to reach equilibrium. After this time the suspensions were filtered (0.45 µm) and the filtrates were analyzed.

Electrodialytic remediation – lab and pilot scale

The electrodialytic experiments in lab scale were conducted in cylindrical cells made from polymethyl methacrylate and were built from 3 compartments; two compartments with electrolytes on sides separated by ion exchange membranes from the middle compartment, where the soil slurry was

placed as shown on Figure 1. An anion exchange membrane (50 cm², AR204 SZRA MK III from Ionics, GE Water & Process Technologies) was placed between anode (+) and middle compartment and cation exchange membrane (50 cm², CR67 MK III from Ionics, GE Water & Process Technologies) was placed between cathode and middle compartment. Platinated rods from Permascand were used as electrodes. Electrolytes (500 mL of 0.1 M NaNO₃, pH= 2 ± 0.2 adjusted with HNO₃) were circulated through the electrode compartments and the soil slurry in the middle compartment remain suspended. Application of a DC electric field to electrodes immersed in the electrolytes (compartment I and III) or directly in the treated slurry (compartment II) induces water splitting reactions that depends on polarity of electrode:

- reaction on anode (+): $2 \text{H}_2\text{O} \rightarrow \text{O}_2(\text{g}) + 4\text{H}^+ + 4\text{e}^-$
- reaction on cathode (-): $2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2(\text{g}) + 2\text{OH}^-$

The ED cell can be used in two different setups, i.e. 3C and 2C that corresponds to use of all 3 compartments or only 2 compartments, when aiming to remove only one type of ions. Each concept has its advantages and disadvantages, as for examples 2C setup was reported to have lower energy consumption and shorter time for acidification when removing heavy metals from suspension of harbor sediments and ashes (Kirkelund et al., 2015; Pedersen et al., 2014). Therefore a placing of electrode in the slurries compartment enabled to apply current between the slurry and one electrolyte at a time, depending what kind of ions were aimed to remove. To remove cations compartments II and III were connected, where the electrode M was working as anode and the compartment I was inert in the process. Similarly connection of compartment I and II, where the electrode M was working as cathode was used to remove anions from the slurry.

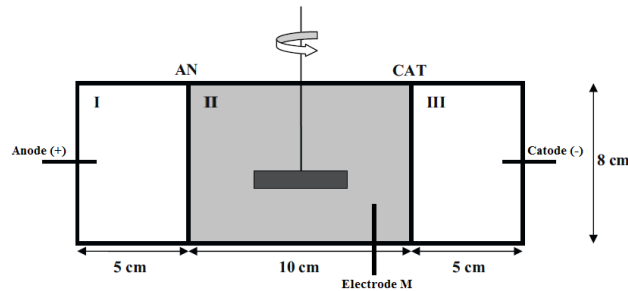


Figure 1 Principle of electrochemical remediation of suspended soil slurry (AN – anion exchange membrane, CAT – cation exchange membrane) (Sun et al., 2012)

The laboratory experiments were performed on 35 ± 0.2 g of soil material that was suspended in 350 ± 5 mL of water, resulting in liquid to solid (L/S) ratio 10. A current of 20 ± 0.1 mA was used resulting in a current density of 0.4 mA/cm^2 . After end of the experiments removal efficiency for each element was calculated as the mass of the actual heavy metal and arsenic in the electrode components (membranes, solutions in electrode compartments and on electrodes) divided by the total mass of the element found in all parts of the cell. Therefore the final results were divided into 4 groups that are defined as following:

- Anode Comp. (+) – sum of the element masses from electrolyte, anode and anion exchange membrane;
- Cathode Comp. (-) – sum of the element masses from electrolyte, cathode and cation exchange membrane;
- Mobilized from soil – mass of the element found in filtrate after passing $0.45 \mu\text{m}$ filter;
- Left in soil – mass of the element in dried soil after filtration with $0.45 \mu\text{m}$ filter.

The upscaled electrochemical remediation was following the same principles as the laboratory cell, but enabled to treat 1000L of soil slurry. The compartments with ion exchange membranes were submerged in the slurry that were mixed with help of compressed air applied from the bottom of the tank, as shown on Figure 2. The upscaled plant used bigger membrane area, i.e. 2200 cm^2 each, and 25L of electrolytes were circulated. There were used different initial volumes of slurry, but the L/S ratio was the same as in the lab experiments.

The energy consumption of the ED process can be calculated by equation $E_x = \int V I dt / W$, where E is the power consumption per milligram of removed element ($\text{Wh/mg}_{\text{removed } x}$); V - voltage between working electrodes (V); I - current (A); t - time (h); W - the mass of the element found in the electrolytes compartment (mg). For the upscaled treatment plant power meters (SparOmeter – kWh meter from Elma Instruments) were used to measure a factual energy consumption of all electrical equipment, also for electrolytes pumps and stirring systems.

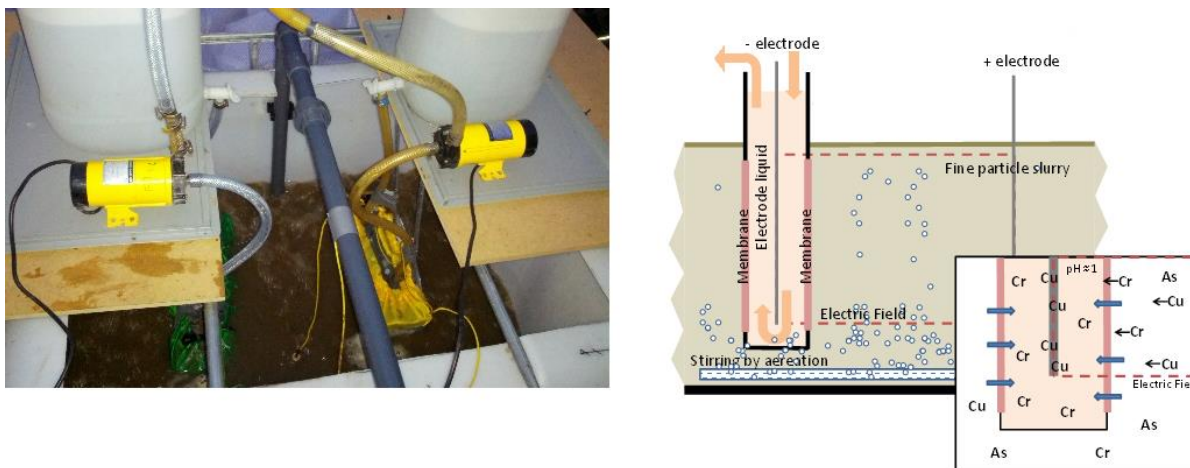


Figure 2 Left: The prototype bench scale electrochemical treatment with aeration system (in a bottom), electrode cassettes and an electrode liquid recycling system (green with cation exchange membranes and catholyte and yellow with anion-exchange membranes and anolyte). Right: Conceptual model of electrochemical batch. Only the setup for removal of cations, Cr and Cu, is shown. For removal of As, the electrodes and membrane are shifted.

RESULTS

Soil characteristic and implications on the treatment procedure

The particle size distribution obtained by wet-sieving of soil A indicates that the soil consisted mostly of size fraction between <1-0.125 mm (Figure 3). However these fractions are the least contaminated by As, Cr and Cu. The highest contents of Cu, exceeding the Danish clean soil criteria of 500 mg/kg, were in sand (> 1mm) and finest fractions (<0.063 mm). All fractions surpass As the criteria that is 20 mg/kg, but the highest As content was found in the finest fractions. Chromium exceeding the clean soil criteria was found only in the finest fraction. The presence of the contaminants in coarse fractions can be a consequence of organic material occurrence, especially preserved wood particles. Though accumulation of contaminants in the finest fraction of soil brings two advantages, enable to reduce mass of treated material and increase initial contaminants content. Therefore washing of soil B was performed on 14 tonnes of material and was aiming to obtain the finest fraction that would have been used in the pilot scale investigations. The outcome of soil washing was production of 4.4 tonnes of fine fraction (fine-B), which properties can be found in Table 1. The fine-B material was only exceeding the clean soil criteria for arsenic content, as the soil-B did not have high Cr and Cu concentration when compared to soil-A.

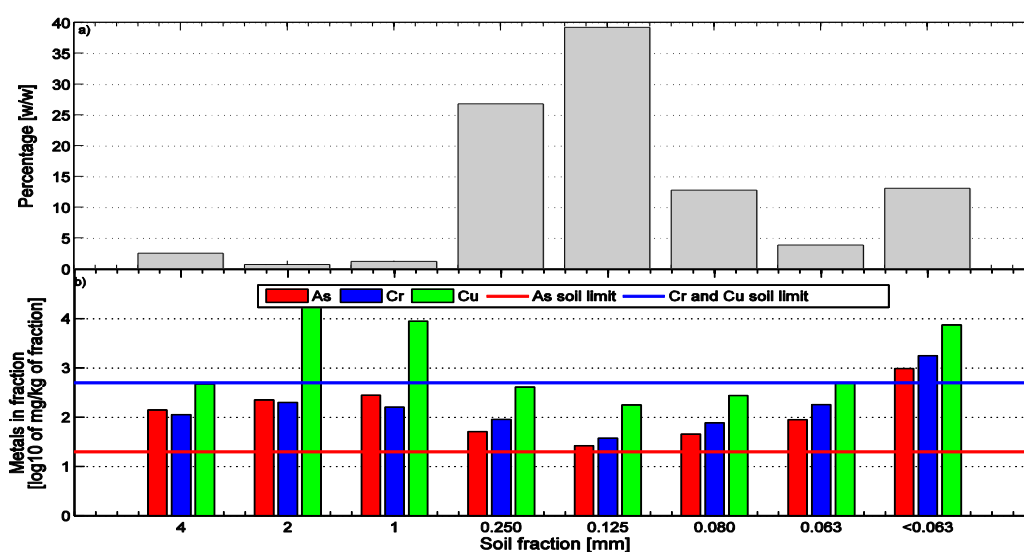


Figure 3 Particle size distribution (a) and concentration of contaminants in each fraction size for soil sample A (As, Cr and Cu soil limits refer to Danish clean soil criteria)

The pH of slurry is a crucial parameter for electrodialytic remediation as it affects heavy metals and arsenic release. The As mobilization dependences on pH for fine fractions, obtained from the soil washing facility and from wet sieving to mimic the one from the washing facility, are presented on Figure 4. The differences in the As mobilization are significant and are most probably caused by the fact that the fine fraction from the washing facility was mixed with settled material from treating recirculated water used for washing. The recirculating water treatment was based on flocculation with commercially available agent, Magnafloc 120. Unfortunately the use of the flocculation could have not been avoided. However results obtained from fine-M material show more efficient As mobilization from the fraction depending on the pH. To achieve more than 80% of As mobilization it is necessary to use either very low pH <1 or high pH, i.e. above 12. The mobilization of arsenic compounds at low pH may be due to dissolution of contaminant bearing minerals and aluminum and iron hydroxides that are known for their adsorption capacities (co-precipitation). However the low pH conditions are limiting occurrence of arsenic anions, as its species starts to dissociate at pH above 2 (pK_{a1} of arsenate- H_3AsO_4 is 2.2 and pK_{a1} of arsenite- H_3AsO_3 is 9.3). Applying the basic conditions, especially pH above 12, reveals a different pattern for contaminants mobilization. At high pH the arsenic distinguishes itself as the most mobilized. The phenomena may be explained with desorption of As anions from amorphous iron oxide that starts at pH above 9 (Dixit and Hering, 2003).

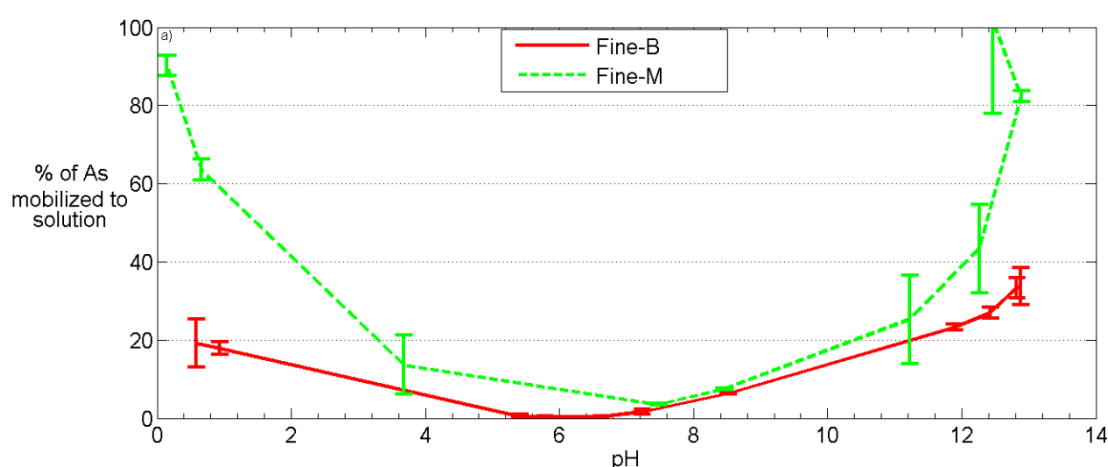


Figure 4 Dependency of pH on As mobilization from fine fractions B (from soil washing facility) and M (from wet sieving in lab)

ED - lab and pilot results

An example of laboratory results of all three contaminants removal from soil fraction from the hot spot site, fine-A, were obtained in a two phase experiment (Figure 5). The ED experiment, where during first 2days aimed to remove cations reaching pH 1.5 and 2nd phase, lasting 15 days, where anions were removed at pH 10-11 enabled to achieve 90% of Cu removal and 50% of As removal efficiency. However it was not possible to remove chromium compounds. The ED laboratory processes were improved with regards to As removal efficiency, as the element was the only one exceeding the clean soil criteria in the fine-B material used in further laboratory and pilot scale investigations. Application of 2-compartment setup cell for removal of arsenic from fine-B and fine-M materials revealed that it was possible to remove above 70% of arsenic, Figure 6, which are a better result than achieved in this and other studies, when soil fines suspension was treated in acidic conditions (Sun et al., 2012). The application of the same ED conditions on fine materials B and M exposed an electrodialytic process advantage. It was possible to achieve comparable As removal efficiency even though As was barely mobilized from the fine-B material containing the flocculant agent that affected pH desorption as discussed above.

Similarly to the laboratory investigations on the fine-B material the ED processes have been performed only at high pH conditions that can be reached in two different ways: by producing hydroxide anions with help of reaction on a cathode submerged in soil slurry or by dosing hydroxide to the slurry. As expected from pH desorption results the addition of NaOH increased pH immediately from 7.5 to 10.8 and resulted in some arsenic mobilization. Production of sufficient amount of OH^- on cathode requires time and energy before mobilizing arsenic species from suspended material. In other hand addition of other anions, like when dosing NaOH, may have a consequence in long-term process efficiency as hydroxide and arsenic anions will compete to pass through anion exchange membrane. An evolution of main parameters during pilot electrodialytic treatment of fine-B suspensions, with and

without initial pH fixing with NaOH, is presented on Figure 7. From a beginning of the treatment no significant As removal to electrolyte was observed in the experiment without initial pH adjustment. An initial addition of hydroxides to the soil slurry increased pH and resulted in arsenic release to water phase enabling their immediate removal through anion exchange membrane driven by applied current. Moreover addition of NaOH increased suspensions conductivity and as a consequence enabled to apply higher current that was limited by maximum voltage given by a power supply. The treatment of the fine material slurry without initial pH fixing showed to be a slow process, where first 700 hours were used to reach the same pH as achieved with NaOH addition. Besides the increase in pH did not enable to achieve to reduce suspension resistance and limited the current applied in the ED, resulting in much lower arsenic uptake from slurry.

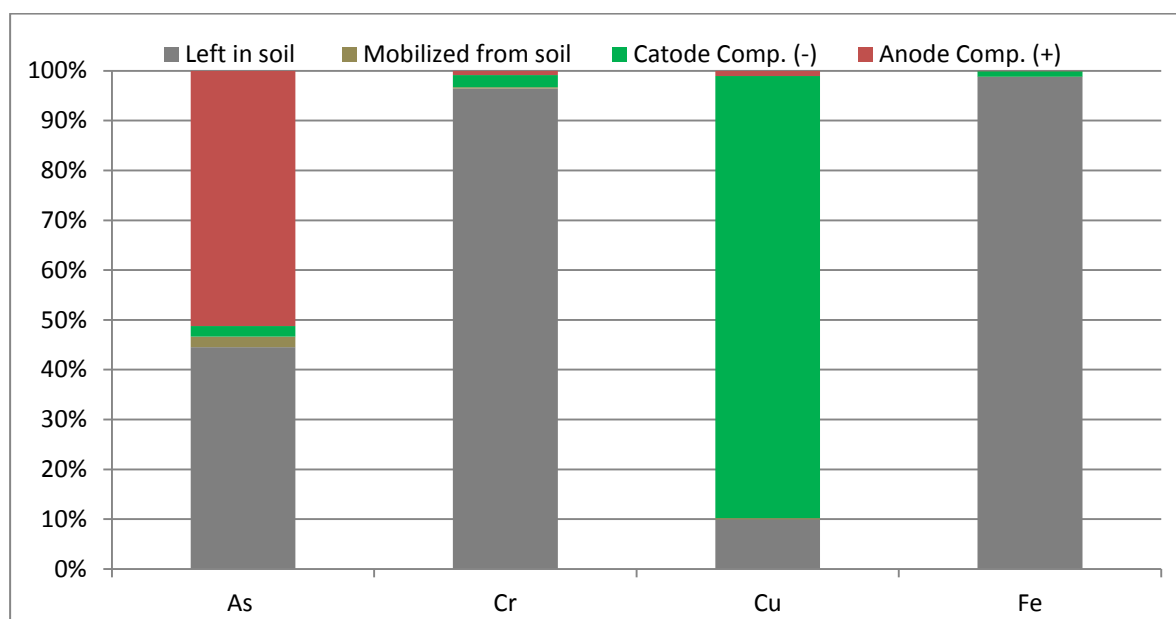


Figure 5 Performance of ED laboratory experiment, where cations were removed at low pH, in a first phase lasting for 2 days, and anions at high pH, in a second phase lasting for 15 days

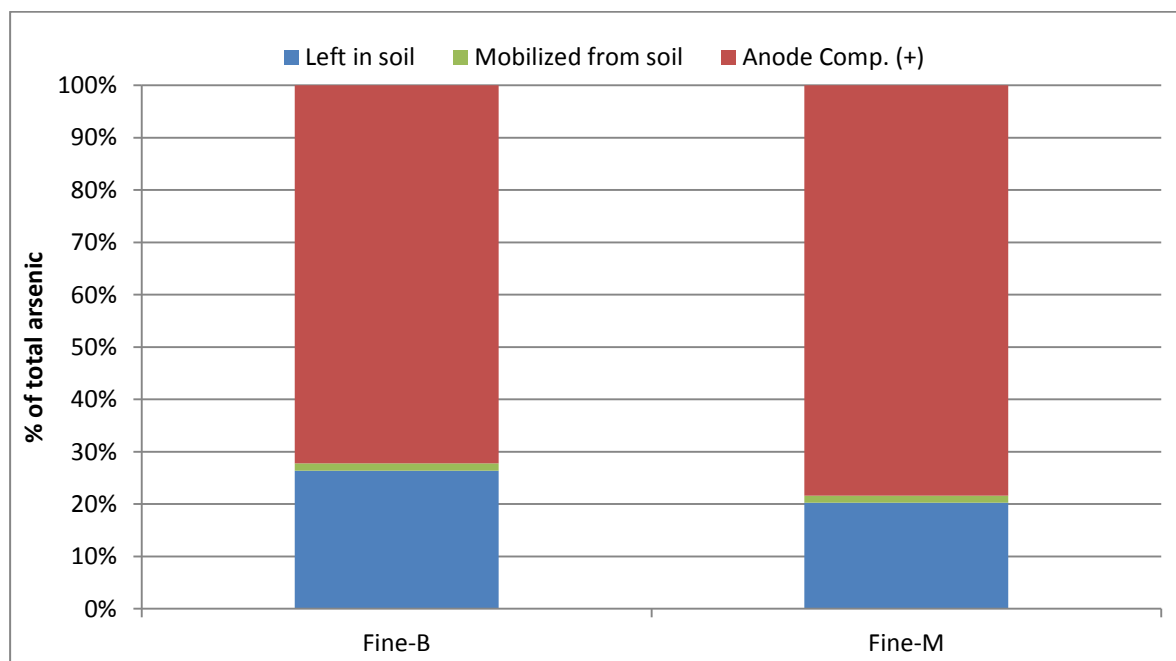


Figure 6 Comparison of ED laboratory experiment performed on fine-B, fine fraction from soil washing facility, and fine-M, fine fraction from laboratory sieving. The experiments were performed in 2C cell setup, where slurry were kept at high pH 10-11

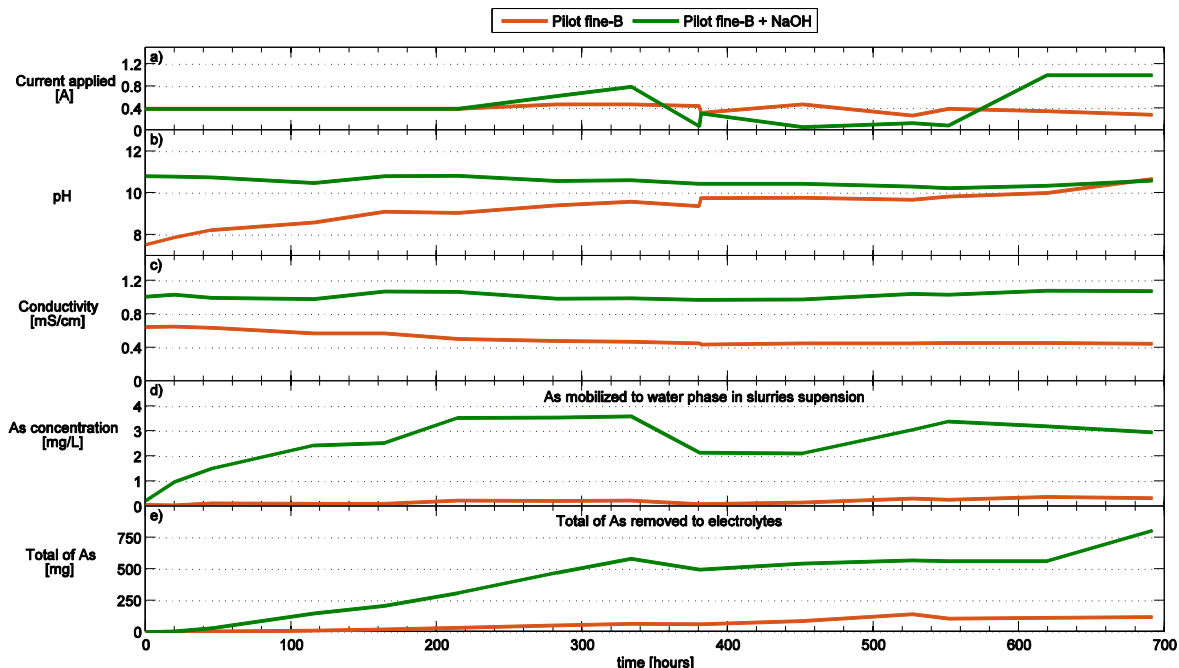


Figure 7 Variations of main parameters during ED pilot remediation applied for fine-B suspensions (90kg of soil fraction in 880 L of water) with and without NaOH initial addition: applied current (a) to electric field; pH (b) and conductivity (c) of suspensions; concentration of As mobilized to suspensions water phase (d) and total of As removed to electrolytes (e)

ED – experiences from upscaling

Comparison of processes efficiency, with regards to energy used for removal of As ($\text{Wh/mg}_{\text{removed As}}$) is presented on Figure 8. Three different pilot treatments are compared with the ED laboratory experiment on fine-material. The laboratory experiment was the most efficient with regards of energy used for ED, i.e. energy of power supply to apply current between the electrodes, which can be explained by the small size of the laboratory cell and amount of material used. The pilot treatment required 10-100 fold higher energy to remove copper and arsenic from slurry, which indicates that electric current was not as efficiently distributed in the slurry container as it was in the laboratory cell. However the differences in power used for maintaining suspension reveals that in the laboratory experiment a low cost of energy used for ED is only a minor part of total energy spent on the treatment, as a major part of the energy was consumed by a stirring system. On the contrary the pilot setups with the fine-B material have used a very low energy for stirring, as it was applied only twice a week for period of 15 minutes, which seemed to be sufficient to keep a material in suspension to some degree. More energy was used in case of treatment of soil-A suspension, as the slurry were constantly mixed, but it has not been enough to keep a coarse fraction of soil suspended. However the energy used for ED itself is much alike for treatment of acidified slurry of soil-A and basic slurry of fine-B, which can be explained by immediate arsenic mobilization and its availability for removal. As for pilot treatment of fine-B without initial pH change the process started at pH 7.5, where less As is mobilized, resulting in much lower energy efficiency for its removal. The processes are continued to reveal mechanisms of As removal and built a ground for process optimization.

Beside a proper stirring regime application in the pilot plant ED process some experience about scaling issues was also learned. The cause of the disruptions in applied current, that were experienced and can be noticed by current drop on Figure 7, seemed to have their source in fouling of the membrane. One of the reasons could have been a choice of the electrolyte solution due to scaling of magnesium and calcium carbonates on the membrane surface, as tap water with significant amounts of those cations and bicarbonates was used as basis for electrolyte medium in the pilot runs. Another source of membrane fouling could have been a presence of the chemical agent used in the soil washing facility, a high molecular weight (several millions) anionic polyacrylamide flocculant, which due to its properties may also be attracted by electric current and deposit on the membrane surface limiting its use. However, when applying the soil washing in a full scale and on-site, the use of flocculant can be avoided as the produced fine fraction slurry would be used directly in the ED process and there will be no need for water recirculation. Therefore the process improvement shall focus on

finding and maintaining the electrolyte solution together with application of membrane cleaning procedures to overcome fouling issues caused by scaling.

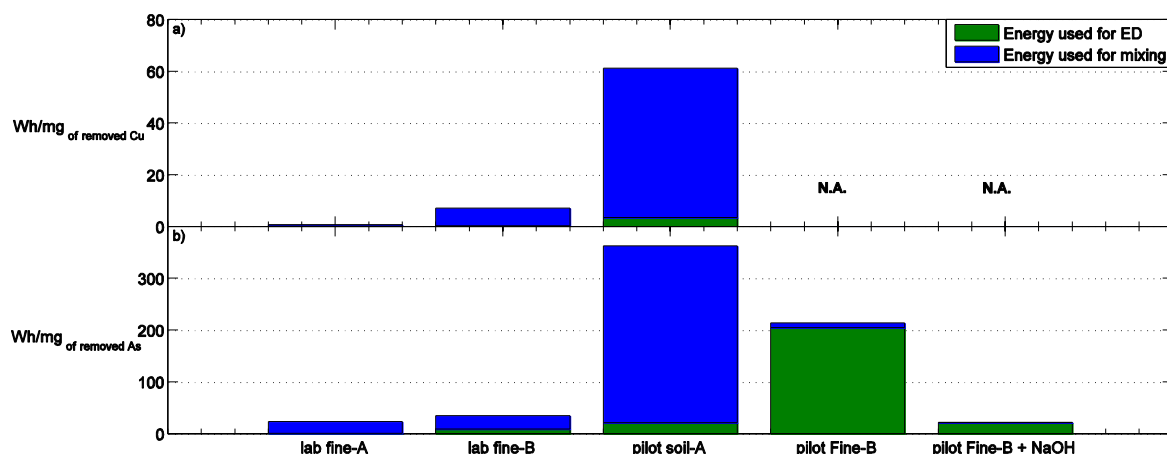


Figure 8 Comparison of energy of the ED processes used for removal of Cu (a) and As (b) from soil material suspensions in laboratory cell („Lab fine-A” and “Lab fine-B”) and in pilot runs with soil A („Pilot soil-A” - refers to energy used since suspension was acidified to pH around 2) and fine B with NaOH addition („Pilot Fine-B+NaOH”) and without any chemicals addition to the suspension („Pilot Fine-B”), where only As was aimed for removal

Conclusions

Laboratory studies proved that it is possible to apply electrodialytic remediation for separation of arsenic and copper from soil materials. Chromium compounds were removed in a minor degree. The amount of treated material can be reduced with help of soil washing that enables to separate the most contaminated soil fraction, which was found to be the finest fraction (<0.063 mm).

However different approaches have to be applied to remove cations and anions, especially when scaling up the process. To remove cations it is necessary to mobilize them at low pH, but for anions removal, especially arsenic the high pH of the suspension has to be used to have sufficient anions mobilization. The factors influencing the ED pilot scale, beside pH of treated suspension, are the stirring routine to maintain material suspension, solution used for electrolyte and agents added during soil washing, which can be eliminated when performing the washing on-site. Basing on experiences from this and previous ED-based remediation processes an upscaling of the process has been proposed to treat contaminated soil according to following on-site procedure:

1. Soil excavation.
2. Separation and soil fractionation in a soil washing facility on-site.
3. Performing on-site EDI remediation on the fine fraction slurry
4. Soil regeneration.

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Appendix



Picture 1 Pilot plant with 5 1000L tanks for soil fraction slurry

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