



## Are electrochemical fences effective in the retention of pollution?

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### ABSTRACT

In this work, a three-month test is used to evaluate the role of electrokinetic fences in the prevention of the diffusion of herbicide in case of accidental spills of large amounts of highly concentrated products in clay soil. To do this, a mock-up ( $0.95 \times 2.00 \times 0.50 \text{ m}^3$ ) is used and an electrokinetic fence is placed far away from the hydraulic plume in order to mitigate the effect of the diffuse pollution caused by the spill of an important amount of 2,4-dichlorofenoxyacetic acid (2,4-D) and oxyfluorfen. The results confirm that diffusion of pollutants is important and that the presence of an electrokinetic fence can contain micellar pollutants and help to minimize the trespassing of soluble pollutants, although they cannot fully prevent this trespass. The results obtained in this work are compared to those obtained in previous works, in which fence technology with surrounding electrode placement was evaluated and important differences were pointed out; this has generated valuable data for the design of this technology.

### 1. Introduction

Despite being a reference technology for the remediation of soil polluted with metal ions, the application of electrokinetic processes for the remediation of soil polluted with organics can be considered as a relatively recent topic, which has attracted more and more attention in recent years because of the interesting results obtained by different research groups all around the world [1–3]. Electrode arrangement is one of the key factors in electrokinetic technology, because it fixes the fluxes of the different species (water, pollutants) in the electro-remediated zone [4,5]. According to the literature, there are other arrangements that can be used to flush the pollutants from soil, replacing the well-known pump and treat processes in soils with low permeability by others that prevent dispersion by fencing the pollution in a restricted narrow zone.

Regardless of the type of treatment, two parameters seem to have a great influence on the application of electrokinetic soil remediation processes. One is the solubility of the pollutant in water, related to the necessity of adding surfactants in the flushing fluid formulation, in order to mobilize the pollutant. The other is the vapor pressure, which is related to the relevance of the volatilization during the treatment that, in turn, is known to be promoted in electrokinetic processes because of the rise in the temperature caused by the ohmic heating

[6–19]. Two good models of pollutants for testing are oxyfluorfen and 2,4-dichlorofenoxyacetic acid (2,4-D), because they have a very different solubility but a similar vapor pressure. Thus, oxyfluorfen has low water solubility ( $0.1 \text{ mg dm}^{-3}$  at  $22^\circ\text{C}$ ) and a vapor pressure of  $0.026 \text{ mPa}$  at  $25^\circ\text{C}$ , while 2,4-D has a great solubility in water ( $900 \text{ mg dm}^{-3}$ ) and a very similar vapor pressure of  $0.020 \text{ mPa}$  at  $25^\circ\text{C}$  [20].

In previous works, we have evaluated in 180 L-mock-ups the remediation of soils polluted with oxyfluorfen and 2,4-D with different EK based technologies, including EKSF (electrokinetic soil flushing) with linear rows of faced electrodes [21,22], EKSF with surrounding electrodes configurations 1C6A (one cathode surrounded by six anodes) and 1A6C (one anode surrounded by six cathodes) [23,24], and electrokinetic fence (EKF) technologies with surrounding configurations of alternating electrodes [25]. From these works, it was concluded that:

- dispersion of the pollutant was very rapid once an accidental spill was simulated and
- the technology applied (in particular the electrodes distribution) was very relevant to the results attained, because it determined the total efficiency of the removal of pollutants from soil.

For the removal of oxyfluorfen, the EKF – the process in which the

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pollution point is surrounded by a circular configuration of alternating electrodes – was found to be the most efficient technology, being the dragging of micelles to the cathode, the primary electrokinetic mechanism, although the electrophoresis of micelles towards the anode was also important in order to explain the experimental observations. This was not an expected outcome, because this configuration was proposed as an electrokinetic fence to prevent pollution dispersion and not as a remediation technology. However, it was found inefficient in the prevention target but very efficient in the removal of the micellar herbicide [25,26].

On the other hand, for the removal of 2,4-D, which is known to be a herbicide with high solubility in water, the application of EKSF with a 1C6A configuration allowed us to reach the best removals. In this case, the primary mechanism was the efficient transport of the 2,4-D to the anodes by electromigration, which is promoted in this specific technology by the high number of anodes used.

In addition to the electrokinetic transport mechanisms, another key outcome made in our previous studies at bench-scale was the relevance of the volatilization mechanisms, which explain the very high transport of pollutants to the atmosphere, and that obliges in a real treatment to include polluted gas treatment technologies in the complete remediation scheme. This volatilization also develops in non-remediated soils, although the increase in the temperature associated with the ohmic heating caused by the application of an electric field during electrokinetic remediation technologies strongly promotes it.

Notwithstanding this, all these technologies were not found to be efficient in retaining the pollution in the soil, which means that it is still important to study this issue further. Thus, despite the large size of the bench-scale plants used (especially if compared to most studies found in the literature), the rapid transport of the pollutant through all the set-up after the simulated accidental spill does not allow us to reach conclusions about the possibility of containing the pollution in real cases. For this reason, in this work we are simulating an accidental spill of a mixture of 2,4-D and oxyfluorfen, in a mock-up with a dedicated hydraulic flow pattern. In this case study, we have placed the EK fence at a relatively large distance, in order to confirm if there is a way to avoid pollution dispersion in places far away from hydraulic plumes with EK processes. It is important to take into account that in real situations the hydraulic plume can flush pollution (by combination with a water treatment technology) and that a key problem can be the dispersion, by diffusion, of the pollution to places far away from this plume.

## 2. Materials and methods

### 2.1. Experimental set-up

A scheme of the process aimed to be studied is displayed in Fig. 1A. The clay soil used in this study was from a quarry located in Toledo (Spain), and its characterization and preparation for the experiments is described elsewhere [27]. The bench scale plant was a methacrylate prism with a soil capacity of 950 dm<sup>3</sup> (LWH: 200 × 95 × 50 cm<sup>3</sup>), in which 1 × 1 × 10 cm<sup>3</sup> graphite rods were connected to a power supply (400 SM-8-AR ELEKTRONIKA DELTA BV) and were used as positive and negative electrodes, being positioned in semi-permeable electrolyte wells. Fig. 1B presents the instrumentation of the plant including feed-wells and electrode-wells (anodic and cathodic wells), tensiometers and thermocouples. A constant voltage gradient of 1.0 VDC cm<sup>-1</sup> was applied between each consecutive pair of electrodes. The reactor was designed to separate and collect the fluids through an outlet situated on one of the sidewalls of the reactor, which was used to simulate the hydraulic plume. To monitor the flux of water and the temperature evolution during the experiment, tensiometers and thermocouples were inserted into the soil.

### 2.2. Experimental procedure

Once the plant was fully instrumented, the experimental procedure began with the pollution of the soil (simulating an accidental spill over 71 days). Thus, in one side of the mock-up, where the feed wells were, an accidental leak of 11.6 g of 2,4-D and 11.6 g of Fluoxil 24 EC (oxyfluorfen 24%) was simulated over 71 days (0.166 mg of each herbicide d<sup>-1</sup>). In addition, a hydraulic flux of 0.648 dm<sup>3</sup> d<sup>-1</sup> was also induced (water, pH 7.64 and 0.391 mS cm<sup>-1</sup> of conductivity). This hydraulic flux was added in the feed wells and collected in a special outlet made at the bottom part of one of the walls of the mock-up.

The test started when the power source, a 400 SM-8-AR ELEKTRONIKA DELTA BV, was turned on and applied a constant voltage gradient of 1.0 V cm<sup>-1</sup>. During the experiment, the electrical current, temperature, pH, conductivity, 2,4-D concentration and oxyfluorfen concentration of the hydraulic fluid were monitored. The hydraulic plume outlet was sampled daily. The anodic and cathodic wells could not be sampled regularly because it was a complicated process to keep the wells filled. At the end of the experiment, an in-depth sectioned analysis of the complete soil section was conducted (post-mortem analysis) by dividing the set-up into 87 pieces in order to obtain 2-D and 3-D maps of different parameters such as pH, conductivity, moisture, 2,4-D concentration and oxyfluorfen concentration.

### 2.3. Analyses

The oxyfluorfen and 2,4-D concentration were determined by high performance liquid chromatography (HPLC), using an Agilent 1100 (Agilent Technologies, Palo Alto, California, EEUU) with a UV detector (220 nm) and a 150 × 3.0 mm Phenomenex Gemini 5 μm C18 column. The flow rates used were 0.6 cm<sup>3</sup> min<sup>-1</sup> of acetonitrile (70%)/water (30%) for oxyfluorfen and 0.6 cm<sup>3</sup> min<sup>-1</sup> of acetonitrile (40%)/water, with 0.1% phosphoric acid (60%) for 2,4-D. To quantify the amount of oxyfluorfen in the liquid samples, an L-L extraction process was carried out in Eppendorf tubes (15 cm<sup>3</sup>), using acetonitrile as the solvent (ratio = 1 w/w). Both phases were vigorously stirred in a vortex mixer (VV3 VWR multi-tube) for five min. before injection into the HPLC. To quantify the amount of oxyfluorfen in the soil, an L-S extraction process was performed in Eppendorf tubes (15 cm<sup>3</sup>), using acetonitrile as the solvent (ratio of polluted soil/solvent = 0.4 w/w). Both phases were vigorously stirred in a vortex mixer (VV3 VWR multi-tube) for five min., and subsequent phase separation was accelerated using a centrifuge rotor angular (CENCOM II P-elite) for 20 min. at 4000 rpm, before injection into the HPLC. To quantify the amount of 2,4-D in the soil, the same L-S extraction process was used but using water as solvent. Measurements of pH and electric conductivity were completed using an InoLab WTW pH-meter and a GLP 31 Crison conductivity meter respectively. The electric current was measured with a KEITHLEY 2000 Digital Multimeter. The temperature measurements were performed with PT-100 thermocouples.

## 3. Results and discussion

Fig. 2 shows the current intensity produced by the electric field of 1.0 V cm<sup>-1</sup> between each consecutive anode and cathode. As can be seen, for every day there is an abrupt decrease from an initial value, which shows an increasing trend with time, down to zero; this corresponds to the depletion of water from soil in the electrolyte wells. Water was added once a day, and after this addition, the intensity increased quickly, up to the maximum daily value. This means that this configuration of electrodes dries the soil in the immediacy of the electrode zone and requires the permanent addition of water into the wells in order to get a suitable performance. This is an important difference with respect to what was observed in the case of EKf with a circular configuration of electrodes, for which the water content of the soil did not generate any operational problem during the tests [28,29]. In fact, with

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