



## Removal of oxyfluorfen from spiked soils using electrokinetic soil flushing with linear rows of electrodes



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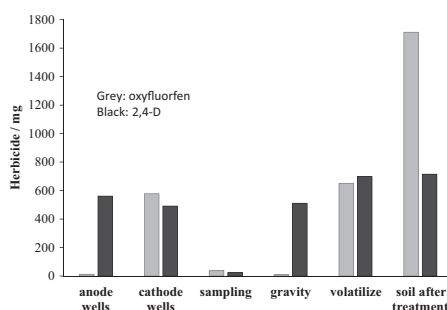
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### HIGHLIGHTS

- The herbicide oxyfluorfen can be transported by electrokinetic processes in EKSF.
- The dragging of oxyfluorfen to cathode wells is the main EK mechanism in EKSF.
- EKSF attains a 26.8% improvement in the removal of oxyfluorfen after 34 days.
- EKSF yields a soil pH gradient as a consequence of the acidic and basic fronts.
- The removal of a given herbicide by EKSF strongly depends on its solubility in water.

### GRAPHICAL ABSTRACT



### ARTICLE INFO

#### Article history:

Received 12 January 2016

Received in revised form 24 February 2016

Accepted 26 February 2016

Available online 3 March 2016

#### Keywords:

Oxyfluorfen

Herbicide

Electroremediation

Natural soil

Electrokinetic soil flushing

Pilot plant

### ABSTRACT

This study focuses on the evaluation of the electrokinetic soil flushing (EKSF) strategy to remediate soil following a simulated spill of the herbicide oxyfluorfen. EKSF is attained by placing (in the soil mockup) two rows of electrodes of different polarity facing each other. The results are compared with those obtained in a reference experiment in which the same spill was simulated and no remediation actions were taken. In addition to the daily monitoring of the most important parameters in the flows, after the remediation test, a post-mortem analysis was performed to obtain a 3-D map of the pollutant distribution. Those results demonstrate that despite the hydrophobic character of oxyfluorfen, it can be efficiently transported by EKSF. Hence, after 34 days of treatment, a 26.8% improvement in the removal of oxyfluorfen was achieved (explained in terms of the effect of the electric field on the pollutant) compared with the reference experiment in which only volatilization can explain the removal of the herbicide. Comparison of the removal of oxyfluorfen by EKSF with that of 2,4-D (studied in a previous study) demonstrates that comparable dragging to the cathode and volatilization are obtained. However, the lower efficiency of the transport of oxyfluorfen by gravity fluxes and electromigration (explained because it is contained as micelles) yielded worse performance of EKSF for this water-insoluble pesticide and hence less efficient remediation. This contradictory result reveals the importance of tests at large-scale facilities such as that used in this work to predict the performance of real systems in future full-scale applications.

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## 1. Introduction

Pollution of soils is currently an environmental issue of major significance. Therefore, the improvement of soil remediation processes is at the cutting edge of environmental technology. In the development of such processes, a positive impact on the environment is sought [1,2], associated with both

- (1) avoiding the rapid diffusion of pollution after an accidental discharge event, and
- (2) removing pollutants from soils with an in situ technology that does not require excavation and hence a substantial modification in geotechnical soil properties.

To meet this objective, a great variety of processes have been developed to remediate soils, including physical (e.g., heating and evaporation), chemical (e.g., oxidation) or biological (e.g., bio-oxidation) processes. One of the most interesting technologies is based on the effect of electric fields induced in the soil using electrodes placed in different positions. These electric fields transport water, pollutants and other species through the soil, increase the temperature (by resistance heating) and cause chemical changes, including precipitation and ion exchange (associated with the acidic and basic fronts produced, respectively, on the surfaces of the anodes and cathodes). The transport processes are typically known as electrokinetic processes. The arrangement of electrodes in the soil and the operation conditions applied allows multi-decadal, full-scale promotion of different types of electrochemically assisted soil remediation processes [3–8], in particular, for the removal of metal ions [9,10].

Electrokinetic soil flushing (EKSF) is one of the most promising technologies [3,11–14], consisting of adding a flushing fluid near the anodes placed in the soil and collecting near the cathodes. The flushing fluid removes pollution without significantly affecting the physical properties of the soil; the fluid may consist of water, when a highly soluble species is to be removed, or a surfactant solution, for low water-solubility species. This technology is for use in soils with low hydraulic-conductivity, and its efficiency has been demonstrated with inorganic species, soluble organics and non-soluble organics [15–19]. Recently, EKSF has been studied for the removal of metals [9,18,20–26], pesticides [1,27–29], polycyclic aromatic hydrocarbons [5,30–34,20,35–38], and PCBs [15,39–41]. The transport of hydrophobic organic compounds (HOC) through the soil should be evaluated because these pollutants cannot be mobilized by electro-osmosis (low water solubility) or electromigration (non-polar molecules), and thus the use of special flushing-fluids is necessary [37,42]. These flushing-fluids usually consist of surfactant solutions that are able to improve the mass transfer of HOCs from the soil into the aqueous phase by breaking up HOCs into the hydrophobic cores of surfactant micelles [39,43–59].

The use of herbicides in agriculture has many economic advantages but also serious environmental drawbacks. Currently, great effort is being exerted in the development of more environmentally friendly pesticides, but the development of processes that can rapidly remove them from soils when accidental discharges occur remains necessary, and because of its characteristics, EKSF could be quite useful for this purpose. Several of the most commonly applied herbicides have low solubility, and the development of technology to remove them from soils is a topic of major interest. Among these herbicides, oxyfluorfen [2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl)benzene] is a good model of a non-soluble herbicide commonly used in agriculture to control broadleaf and grassy weeds. The compound belongs to the diphenyl ether chemical group and has low water solubility (0.116 mg/

L at 20 °C), low vapor pressure (0.026 mPa at 25 °C), high Koc (log Koc = 3.46–4.13) and high Kow (log Kow = 4.86). This herbicide is not a good candidate for phytoremediation or bioremediation, and even its transport in clay soils is not favored [60–62].

The objective of this study is to evaluate the use of EKSF to remediate soil in which an accidental discharge of commercial oxyfluorfen reagent (FLUOXIL 24 EC) was simulated. It contains oxyfluorfen and a surfactant cleansing agent (calcium dodecylbenzenesulfonate) that allow the breaking up of oxyfluorfen into surfactant micelles. The tests were performed at a pilot plant to evaluate the interactions between the different processes at the proper scale; this scale is one of the primary innovations of this study. The electrode arrangement was a linear row of anodes facing a linear row of cathodes. This is the most typical configuration for EKSF processes. The results are compared with those obtained in a reference experiment in which the diffusion of the herbicide following an accidental discharge is not controlled but simply monitored. In addition, the results are also compared to those obtained in a previous study [27] in which the same technology was used for a different model of herbicide, 2,4-D, with greater solubility in water. Due to the very different properties of both pollutants, comparison will give very valuable conclusions about EKSF technology.

## 2. Materials and methods

### 2.1. Preparation of the polluted soil

Field soil from Toledo (Spain) was used in this study. This soil is characterized by its inertness, low hydraulic conductivity and lack of organic content. The mineralogical composition and parameters used to classify this soil by the Unified Soil Classification System (USCS) are provided elsewhere [27,36]. In addition, particle size distribution of the soil was determined using a laser diffraction particle size analyser (model LS 13320, Beckman Coulter) with an aqueous module. The results of this analysis show that the soil are composed on these fractions: 4.9% clay, 68.2% silt and 26.9% sand, and it can be classified as a silty loam according with the texture classification of the United States Department of Agriculture (USDA). Oxyfluorfen 24% (with calcium dodecylbenzenesulfonate as the surfactant cleansing agent) was used as a model of a non-polar, hydrophobic herbicide. The commercial herbicide used was “Fluoxil 24 EC” (CHEMINOVA AGRO, S.A., Madrid, Spain). The soil preparation process is important because of the complexity of natural soil. The process was divided into four different stages: (1) installation of three layers of gravel with different particle sizes for mechanical and drain support; (2) moistening the soil to 11% (natural water content); (3) compacting layers of the soil of a fixed thickness (5 cm) in the electrokinetic reactor until the natural density of the soil (approximately  $1.4 \text{ g cm}^{-3}$ ) was achieved; (4) drilling the electrolyte wells and the instrumentation of the plant.

### 2.2. Experimental setup

The electrokinetic experiments were conducted in an electrokinetic remediation plant consisting of an electrokinetic reactor, a power source and tanks of electrolyte. The reactor was a methacrylate prism with a soil capacity of  $175 \times 10^3 \text{ cm}^3$  (LWH:  $70 \times 50 \times 50 \text{ cm}^3$ ). The electrodes used as both the anodes and the cathodes were  $1 \times 1 \times 10 \text{ cm}^3$  graphite rods, positioned in semipermeable electrolyte wells, with an electrode configuration of rows facing the electrolyte wells, as described elsewhere [63,64]. The cathodic wells are connected to  $100 \text{ cm}^3$  sewers to accumulate the fluid transported through the soil and to facilitate

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