



Application of electrokinetic soil flushing to four herbicides: A comparison



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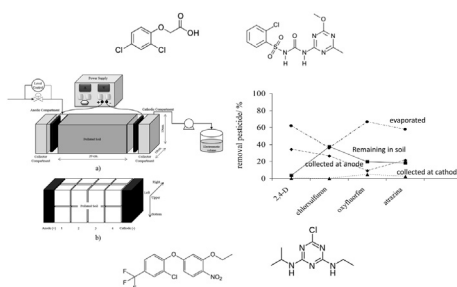
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HIGHLIGHTS

- EKSF can remediate soils polluted with ionic and non-ionic herbicides.
- Herbicides can be mobilized towards the electrodes by electromigration.
- Herbicides can be transported towards the cathode by electroosmotic dragging.
- Volatilization has a great importance and should be accounted in large processes.
- After 15 d long treatment, more than 80% of pesticide is removed.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, four bench-scale plants containing soil spiked with four herbicides (2,4-Dichlorophenoxyacetic acid (2,4-D), oxyfluorfen, chlorsulfuron and atrazine) undergo treatment consisting of an electrokinetic soil flushing (EKSF). Results clearly demonstrate that efficiency of EKSF depends on the chemical characteristic of the pesticide used. The amount of pesticide collected in the anode well is more significant than that collected in the cathode wells, indicating that the electromigration is much more important than drainage by electro-osmotic flux for this application. After 15 d of treatment, the 2,4-D is the pesticide most efficiently removed (95% of removal), while chlorsulfuron is the pesticide more resilient to the treatment. Additionally, volatilization was found to be a process of the major significance in the application of electrokinetic techniques to soil polluted with herbicides and because of that it should always be taken into account in the future design of full-scale processes.

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1. Introduction

Soil pollution is an important problem in modern society. The

diffuse pollution and particularly the effects of this pollution on water reservoirs is becoming an issue of the major significance. Consequently, soil remediation is becoming a very promising area of study within environmental engineering and the development of novel and efficient treatment technologies (such as the electrochemically assisted technologies) is becoming more and more important (Hamdan et al., 2014).

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Among these technologies, the electrokinetic soil flushing (EKSF) is gaining attention in the recent years. When polluted soil undergoes the effect of an electric field between anodes and cathodes, electroosmosis, electromigration and electrophoresis mobilize the pollutants contained in the soil. Under suitable conditions, these processes can be used to remediate the soil, transferring the pollution to a flushing fluid that can be then treated.

EKSF process has been studied for the remediation of soils polluted with different contaminants (Trellu et al., 2016) such as pesticides (Ribeiro et al., 2011; Gomes et al., 2012; Guedes et al., 2014; Ribeiro et al., 2005), hydrocarbons (Gomez et al., 2009; Alcantara et al., 2010, 2012; Mendez et al., 2012; Lopez-Vizcaino et al., 2014a,b; Mousset et al., 2014; Bocos et al., 2015) and heavy metals (Maturi and Reddy, 2006, 2008). In fact, the removal of pesticides using EKSF has become a hot topic (Jackman et al., 2001; Ribeiro et al., 2005, 2011; Harbottle et al., 2009; Gomes et al., 2012; Rodrigo et al., 2014), and even recent works were focussed not only on the efficiency of EKSF but also on the integration of this technique with other remediation technologies such as bioremediation and permeable reactive barriers (Trellu et al., 2016). In general, removal efficiencies reported with spiked kaolinite (as model of low permeable soil) show the influence of electrode arrangements, flushing fluid and electric field. However, the loss of the pollutants by filtration, dragging of superficial water or evaporation have not been clearly stated and these are important points to be capable to control the diffusion of pesticides into the soil (Jackman et al., 2001; Ribeiro et al., 2005, 2011; Harbottle et al., 2009).

Regarding the pesticides, it is important to point out the high risk associated with the hazardousness of these pollutants and their wide range of chemical properties. Therefore, it makes necessary to limit their presence in the environment and to develop efficient technologies of soil and water remediation (Dombek et al., 2004; Castelo-Grande et al., 2005; Malpass et al., 2006; Cao et al., 2015). Among the chemical properties, water solubility and volatilization of pesticides influence significantly the efficiency of the remediation processes and thus determine the feasibility of the treatment.

Taking into account this background, this work aims to compare in the same experimental conditions the remediation of four pesticides (2,4-Dichlorophenoxyacetic acid (2,4-D), Chorosulfuron, Atrazine and Oxyfluorfen) by EKSF. These herbicides have different characteristics, in terms of solubility and volatility, and they were selected in order to understand the mechanisms accomplished in the soil when this type of technologies are applied. To do this, 15 d long tests in bench scale soil columns are used. The scale is large enough to account the important processes happening in the soil in more realistic conditions. Synthetic soil has been used in order to prevent the interaction of the processes studied with other processes that can mask their influence.

2. Materials and methods

2.1. Chemicals

Kaolinite, provided by Manuel Riesgo Chemical Products (Madrid, Spain), was used as a model of clay soil. Properties of this synthetic clay soil were provided by the commercial supplier, and are detailed in the Table SM-1 of the Supplementary Material (SM). All chemicals, including anhydrous sodium sulfate, sodium phosphate (Fluka, Spain), 2,4-D, Chorosulfuron, Atrazine and Oxyfluorfen (Sigma-Aldrich) were analytical grade and used as received. Acetonitrile HPLC grade (Sigma-Aldrich, Spain) was used for the mobile phase. Double deionized water (Millipore Milli-Q system, resistivity = 18.2 M Ω cm at 25 °C) was used to prepare all solutions. Sodium Dodecyl Sulfate (Fluka, Spain) used as

solubilizing agent while hexane and ethyl acetate (Sigma-Aldrich, Spain) were used as solvent for extraction of liquid and solid samples.

2.2. Electrokinetic cell

Bench setup used in this work is shown in the SM (Fig. SM-1). It was made of transparent methacrylate and divided into five compartments. The central compartment with length of 20 cm was loaded with herbicide-polluted soil, which was compacted manually and separated of the electrode compartments by a 0.5 mm nylon mesh. One of these compartments served as anode and the other as cathode. Graphite electrodes (10.0 cm \times 10.0 cm \times 1.0 cm) provided by Carbosystem (Madrid, Spain) were used as electroodic material. Each electrode compartment was connected to an additional compartments to collect the liquid overflowing from the wells that is transported by the electrokinetic processes. The experiments were performed in a potentiostatic mode, i.e., setting a voltage at 1 V cm⁻¹. The duration of the experiment was two weeks.

2.3. Preparation of the soil

The model soil was spiked with 960 mL of herbicide aqueous solutions (100 mg L⁻¹) until attaining an initial pollutant concentration of 30 mg per kg of soil. In the case of atrazine and oxyfluorfen solutions, sodium dodecyl sulfate was used as solubilizing agent (1000 mg L⁻¹). The initial target moisture level of the soil was 30%. Polluted soil was compacted manually in the central compartment of the cell (3 kg). This compaction was carefully done to avoid the formation of heterogeneities zones in the soil, which can result in preferential paths for the fluid transport. In all cases, tap water was used as flushing fluid. The level of the anode wells were maintained by means of a level regulation loop. On contrary, from cathode well water was daily pumped. Evaporation was estimated by mass balance.

2.4. Experimental procedure

During the experiments, the electrical current, the temperature on different portions of the soil, the electroosmotic volume removed from the cathodic collector, pH, conductivity, pesticide and TOC concentration of the electroosmotic fluid were monitored. At the end of the experiment, a *post mortem* analysis was carried out to evaluate the final state of the treated soil (post-study characterization): pH, conductivity, humidity, herbicide and TOC concentrations were measured. For this analysis, the soil compartment was divided into sixteen portions (Fig. SM-1b of SM). Sampling procedure of each soil-portion consists of taking it out carefully from the set up and manually homogenizing it. After that, representative samples were taken for carrying out each analysis. Profiles of concentrations were obtained by sampling solutions at predetermined time intervals during the electrolysis process (robustness of the results was evaluated by four independent analyses).

2.5. Analysis procedures

Measurement of the moisture was carried out by drying the soil samples in an oven for 24 h at 105 °C. Moisture was estimated taking into account the weight difference in the samples before and after drying. For the determination of the pH and conductivity in the soil samples, the standard method (E.P.A.-9045C, 1995) of the saturated paste was used. This method consists of the mixture of 10 g of dry soil with 25 mL of distilled water which was magnetically agitated for 20 min. After sedimentation, the aqueous

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