



# Selecting enhancing solutions for electrokinetic remediation of dredged sediments polluted with fuel



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## ABSTRACT

In this paper a procedure for selecting the enhancing solutions in electrokinetic remediation experiments is proposed. For this purpose, dredged marine sediment was contaminated with fuel, and a total of 22 different experimental conditions were tested, analysing the influence of different enhancing solutions by using three commercial non-ionic surfactants, one bio-surfactant, one chelating agent, and one weak acid. Characterisation, microelectrophoretic and electrokinetic remediation trials were carried out. The results are explained on the basis of the interactions between the fuel, the enhancing electrolytes and the matrix. For one specific system, the electrophoretic zeta potential, ( $\zeta$ ), of the contaminated matrix in the solution was found to be related to the electroosmotic averaged  $\zeta$  in the experiment and not to the efficiency in the extraction. This later was correlated to a parameter accounting for both contributions, the contaminant and the enhancing solution, calculated on the basis of differences in the electrophoretic  $\zeta$  in different conditions which has allowed to propose a methodology for selection of enhancing solutions.

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## 1. Introduction

Electrokinetic remediation has been widely studied in the remediation of heavy metal contaminated soils and sediments, demonstrating the feasibility of this technology (Peters, 1999; Nystroem et al., 2005, 2006; Al-Hamdan and Reddy, 2008; Castellote et al., 2010; Rozas and Castellote, 2012). It relies on the direct application of an electric field to the contaminated sample. The contaminants are removed by several combined mechanisms, mainly electroosmosis and electromigration (Yeung and Gu, 2011; Alshawabkeh and Bricka, 2000). However, its applications in the remediation of organic contaminants have been limited due to low solubility's of organics in water. In addition, most organic contaminants are non-ionic, and therefore they are not mobile under electric fields. The efficiency of the process must be enhanced to increase the solubility of the organic compounds in water. Synthetic and natural surfactants can be used as solubility-enhancing additives in the remediation of organic-contaminated soils and sediments (Mulligan et al., 2001; Chu and Kwan, 2003; Mulligan and Eftekhari, 2003; Fabbri et al., 2009; Mulligan, 2009). Surfactants are compounds containing both hydrophilic and hydrophobic groups. The solubility can be increased by lowering the interfacial

tension as well as by accumulating the hydrophobic material in molecular aggregates called micelles (Haigh, 1996; West and Harwell, 1992).

Since most organics do not have a net negative or positive charge, their transport can be affected by the electroosmotic flow (EOF); in this context,  $\zeta$  is one of the most important parameters because this potential controls the direction and rate of the EOF; therefore, ionic surfactants seem to be the most appropriate, as they introduce charged species that can be moved by electromigration. However, non-ionic surfactants are often used because of their lower critical micelle concentration compared to ionic surfactants, higher degree of surface-tension reduction, and relatively constant properties in the presence of salt, which result in better performance and lower concentration requirements (Shen, 2000; Yang and Lee, 2009). Maybe for this reason, as well as because of the complex matrix/surfactant/contaminant interactions, the results found in the literature concerning the use of surfactants in the electrokinetic remediation of organic compounds are sometimes contradictory (Haigh, 1996; Saichek and Reddy, 2005; Cameselle and Reddy, 2012; Pazos et al., 2013; Maturi and Reddy, 2006).

An understanding of the processes that take place at the sediment–surfactant–contaminant interface, together with those which could affect  $\zeta$ , is essential to achieve good results in the removal of organic compounds using an electrokinetic method. In this context, the objectives of this study are to deepen the knowledge of the interactions between the different agents involved and

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their influence on the efficiency of the process and to propose a method to select the most appropriate enhancing solutions based on accounting for the contributions of interactions between the contaminant, the solution and the matrix.

## 2. Experimental procedure

### 2.1. Materials

In this paper, electrokinetic remediation experiments with dredged material from a Mediterranean Spanish harbour have been carried out. After dredging, the material was stored under controlled conditions. Because of its high organic matter content, freezing was chosen as the best option to preserve it. Storage conditions are described in detail in Rozas and Castellote (2012).

### 2.2. Procedures

#### 2.2.1. Characterization and contamination of the dredged material

The chemical and mineralogical composition as well as the size distribution of the material was characterized. Later the dredged sediment was polluted by the direct addition of commercial automotive fuel, with constant stirring, and allowed to stand for several hours. The final concentration of automotive fuel in the material was about 18,000 ppm. A higher concentration of fuel (30,000 ppm) was used in the assessment of the charge density passed in the remediation process.

The content of fuel in the samples was determined exactly in all the cases before and after the experiments by calcination in an oxidizing environment following the standard UNE EN 1744-1 (UNE EN 1744-1:99). Previously, a calibration curve was found for this material: samples of around 50 g of dredged material containing different amounts of automotive fuel were dried in an oven in two steps; at 100 °C the amount of free water was known. Carbon dioxide coming from decomposition of the calcite, organic matter in the sediment, and the fuel was removed by heating at  $975 \pm 25$  °C.

The enhancing solutions used in this investigation were also characterized by Infrared spectroscopy (IR). The IR spectra of the samples were recorded with an ATR-FTIR spectrophotometer model BX equipped with a diamond-ZnSe ATR single reflection

crystal (Pike Tech.). The samples were analysed in the 4000–600  $\text{cm}^{-1}$  spectral range.

Micro-electrophoretic measurements were carried out using the commercial equipment Zeta Meter 3.0 + (Zeta Meter, New York). The zeta potential of the dredged material in distilled water with and without pollution as a function of pH, using HCl and NaOH, respectively, to decrease and increase the pH, was also measured. The zeta potential of the dredged material in the presence of the enhancing electrolytes with and without contamination was also determined. In all cases, to prepare the suspensions for measurement, 0.1 g of dredged material was suspended in 100 mL of the solution, in which the proportion of the enhancing electrolytes, when present, was 0.1 vol%.

#### 2.2.2. Remediation experiments

Decontamination experiments were carried out by using the Electroosmotic Cell Device (ECD) described in Castellote et al. (2006). A total of 22 different experimental conditions were tested, analysing the influence of different enhancing solutions by using three commercial non-ionic surfactants, one bio-surfactant, one chelating agent, and one weak acid. Table 1 shows the details of the experiments conducted for this study. The commercial name of these products is not given for the sake of confidentiality of the results for the producers. All of the enhancing agents were used in a proportion of 0.1 vol%. Some tests were preceded of pretreatments in which the samples were kept in contact with an excess of the treating solution for 3 days and then dried under vacuum filtration. Electrokinetic tests were conducted with and without pretreatment, with and without enhancing electrolytes and also tests without electrical current as a reference were carried out. The influence of the time and electric charge passed in the remediation process was tested by measuring the decrease in the concentration of automotive fuel in a highly polluted sample of dredged material (test no. 13).

## 3. Results and discussion

### 3.1. Characterization of the dredged material and enhancing electrolytes

The dredged sediment was mainly composed of sand,  $\text{SiO}_2$  (71%), and calcite (26%). From the size point of view, the dredged

**Table 1**  
Testing program and decontamination percentages at the end of the trials.

Test	$\Delta V$ (V)	Anolyte	Catholyte	Pretreatment	% Remediation
1	–	–	–	Bio-surfactant	55
2	–	–	–	Surfactant C	16
3	–	–	–	Surfactant D	16
4	–	–	–	Surfactant D	25
5	–	–	–	Chelating agent	73
6	30	Distilled water	Bio-surfactant	–	8
7	30	Distilled water	Surfactant B	–	31
8	30	Distilled water	Surfactant C	–	91
9	30	Distilled water	Surfactant D	–	34
10	30	Distilled water	Chelating agent	–	5
11	30	Distilled water	Distilled water	–	6
12	–	Distilled water	Distilled water	–	–
13	30	Distilled water	Surfactant C	–	92
14	30	Distilled water	Bio-surfactant	Bio-surfactant	–
15	30	Distilled water	Surfactant C	Surfactant C	40
16	30	Distilled water	Surfactant D	Surfactant D	38
17	30	Distilled water	Surfactant D	Surfactant D	26
18	30	Distilled water	Chelating agent	Chelating agent	–
19	30	Distilled water	Acetic acid 0.5 M	–	29
20	30	Distilled water	Acetic acid 0.5 M	Surfactant C	31
21	30	Distilled water	Surfactant C/acetic acid 0.5 M	–	34
22	30	Surfactant C	Acetic acid 0.5 M	–	26

The initial concentration of fuel was 18,000 ppm in all cases except for test no. 13, in which it was 30,000 ppm.

% Remediation =  $100 \cdot \frac{[\text{initial}] - [\text{final}]}{[\text{initial}]}$ .

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