



Electrokinetic treatment of polluted soil at pilot level coupled to an advanced oxidation process of its wastewater



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ABSTRACT

Soil contaminated with hydrocarbons is a current problem of great importance. These contaminants may be toxic, can retain water and block gas exchange with the atmosphere, which produces a poor-quality soil unsuitable for ecological health. Electroremediation is among the treatments for the removal of such contaminants. In this research, a pilot-level electroremediation test was applied using a circular arrangement of electrodes with a Ti cathode at the middle of the cell surrounded by six IrO₂–Ta₂O₅ | Ti anodes. The presence of an NaOH electrolyte helps to develop the electromigration and electro-osmosis of gasoline molecules (at 1126 mg kg⁻¹) surrounded by Na⁺ ions. The hydrocarbons are directed towards the cathode and subsequently removed in an aqueous Na⁺ – hydrocarbon solution, and the –OH migrates to the anode. During electrokinetic treatment, the physicochemical characteristics of the soil close to either the cathode or anode and at the half-cell were evaluated during the three weeks of treatment. During that time, more than 80% of hydrocarbons were removed. Hydrocarbons removed by the electrokinetic treatment of gasoline-polluted soil were collected in a central wastewater compartment and subsequently treated with a Fenton-type advanced oxidation process. This achieved more than 70% mineralization of the hydrocarbons to CO₂ and H₂O within 1.5 h; its low toxicity status was verified using the Deltatox[®] kit test. With this approach, the residual water complied with the permissible limits of COD, pH, and electrical conductivity for being discharged into water bodies, according to Mexican norm NOM-001-SEMARNAT-1996.

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1. Introduction

Pollutants in soil can be classified into two types, endogenous or exogenous, depending on their origin. An endogenous pollutant is derived from within soil, whereas an exogenous pollutant is derived from an external source. Of particular concern is exogenous contamination, such as when contamination is due to waste discharges, particularly if they are not quickly assimilated or neutralized by the soil, leading to an accumulation of toxins. One type of exogenous contamination is caused by discharges of organic pollutants, such as an accidental spillage of hydrocarbons or combustible petroleum distillates such as gasoline, kerosene, jet

fuel, diesel and fuel oil (Hernández-Soriano, 2014; Bustos, 2012; Alcántara et al., 2012). The contamination of soils and aquifers with hydrocarbons has diverse environmental implications, such as toxicity, surface water retention, and low productivity, and in some cases renders the use of the soil with ecological health (Hernández-Soriano, 2014; Bustos, 2012; Gómez et al., 2010).

Soil remediation technology implies applying any unit operation, or a series of unit operations, that will act to alter the composition of either a contaminant or any dangerous substance through chemical, physical or biological actions to reduce toxicity, mobility, or contaminated material volume (Yeung and Gu, 2011; Lombi and Hamon, 2004). Such remedial technologies are classified according to three principles. The first classification is based on the strategy for remediation, which is described in the function of its basic action, which can be destruction or modification in composition, extraction or separation and the isolation or

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immobilization of the targeted pollutant. The second classification is based on how the soil will be handled during the remediation process. In this classification, the approach can be *in-situ* when soil remediation is carried out without digging at the site where the pollutant is present. An alternative approach is the *ex-situ* type, when the remediation process requires the excavation and removal of contaminated soil for further treatment, either in the same place (*on site*) or away from it (*off site*). The third classification is the treatment approach, which can be biological, thermal or physico-chemical; the electroremediation method is included in this last classification (Bustos, 2012, 2014).

Electroremediation, or electrokinetic treatment (EKT), is a useful technology for restoring contaminated soils. This is a process based on the generation of an electric field by applying either a direct current or a potential difference to obtain such a current; either way, it requires the use of electrodes (anodes and cathodes), which are placed in pits dug into the ground. Usually, these pits are moisturized with an electrolyte to improve the conductivity for the electrical field (Alcántara et al., 2012; Gómez et al., 2010; Yeung and Gu, 2011; Lombi and Hamon, 2004; Bustos, 2014).

The effectiveness of an EKT process depends on variables such as pH, the content of water, electrical conductivity, the physico-chemical properties of the soil and contaminants, the spatial distribution and electrode materials; it is therefore necessary to evaluate these factors before carrying out *in situ* electroremediation (Alcántara et al., 2012) in a pilot plant (López-Vizcaíno et al., 2014; Zhou et al., 2006; Acar et al., 1993). Once contaminants are removed from soil and solubilized in an aqueous medium, they require further treatment via biological, chemical and/or physicochemical methods. Processes such as absorption and extraction technologies simply transport the contaminant to other media without actually solving the problem itself. The latter category includes advanced oxidation processes (AOP), which involve the generation of highly oxidizing species by rapid reactions and can easily degrade organic substances. One of the most oxidizing species generated within these reactions is the hydroxyl radical ($\cdot\text{OH}$), which has an oxidizing strength second only to that of fluorine. One advantage of these oxidative processes is that hydroxyl radicals directly attack the contaminant by altering its chemical structure. These radicals can oxidize organic pollutants by removing hydrogen or by adding it to the double bonds, forming free radicals, which can react with oxygen molecules to form peroxyradicals; these initiate a series of oxidizing reactions that lead to a complete mineralization of the organic pollutant (Bañuelos et al., 2014).

The AOP are divided in two groups: photochemical and non-photochemical. The photochemical reaction group includes the photo-Fenton process, heterogeneous photocatalysis, UV/ H_2O_2 and O_3/UV . The non-photochemical group includes alkaline ozonation, $\text{O}_3/\text{H}_2\text{O}_2$, Fenton reaction, electrochemical oxidation, hydro-pneumatic cavitation/ultrasonic and water sub/super critical. The destructive processes that have been widely include some Fenton process variations (Fenton, photo-Fenton, electro-Fenton, and photo-electro-Fenton) and electrochemical oxidation technologies (Bañuelos et al., 2014; Yap et al., 2011; Bokare and Choi, 2014; Garrido-Ramírez et al., 2010; Panizza and Cerisola, 2001; Park et al., 1999). Other reactive species, such as Fe (IV) or Fe (V) (as FeO^{3+} and Ferril complexes), are the active agents of the process. In the presence of an excess of H_2O_2 , the concentration of Fe^{2+} is small in relation to Fe^{3+} , reaction (6) being slower than reaction (2). Both radicals ($\cdot\text{OH}$ and the $\text{HO}_2\cdot$) react indiscriminately with organic matter, but the latter is less reactive. The high value of the rate constant for the reaction of ferrous ion with H_2O_2 means that Fe (II) is oxidized to Fe (III) in seconds or minutes, when there is an excess of H_2O_2 . The destruction of gasoline by a Fenton process is due to the catalytic effect of the pair Fe (III) – H_2O_2 (Yap et al., 2011; Bokare

and Choi, 2014; Garrido-Ramírez et al., 2010; Panizza and Cerisola, 2001; Park et al., 1999; Rosales et al., 2012).

The use of the Fenton reaction has been reported when combined with an electrokinetic treatment of polluted soil *in situ* in the same process (Yap et al., 2011; Bokare and Choi, 2014; Garrido-Ramírez et al., 2010). In this study, the degradation of contaminants such as diesel reached good efficiencies after 30 days; its removal was enhanced with the surfactant Tween 80 (Pazos et al., 2011). In addition, different bioassays were used to test the progress of toxicity during an AOP to eliminate pesticides. Bioassays are a useful tool to verify the reduction in toxicity due to the mineralization of these types of pollutants (Oleszczuk et al., 2014; Mena et al., 2014; Fernández Rodríguez et al., 2014); examples of species used are *Vibrio fischeri*, *Daphnia magna* and *Selenastrum capricornutum*. Additional information was obtained with total organic carbon (TOC) determination (Fernández-Alba et al., 2002).

In the present research, an EKT was implemented at a pilot level, with the purpose of treating 3.3 m^3 of soil polluted with 1126 mg kg^{-1} of gasoline. The experimental setup considered a 2D circular arrangement of electrodes comprising a Ti cathode surrounded by $\text{IrO}_2\text{-Ta}_2\text{O}_5 \mid \text{Ti}$ anodes (Pérez-Corona et al., 2013a, 2013b; Oszust et al., 2013), applying 20 V and producing a constant current of 9 A intermittently for 20 days ($4\frac{1}{2}$ h/day). The potential and current were measured directly from the power supply. A daily addition of 60 L of $0.7 \mu\text{M}$ NaOH was applied by aspersion, with the purpose of recovering wastewater with Na^+ – hydrocarbon and maintaining the soil saturated with the electrolyte support. The presence of an NaOH electrolyte helps to develop the electromigration and electro-osmosis of gasoline molecules surrounded by Na^+ ions, as mentioned in the literature (Pérez-Corona et al., 2013a, 2013b; Oszust et al., 2013; Alba et al., 2013; Pérez-Corona et al., 2013c; Méndez et al., 2012; Ramírez et al., 2011). The excess of NaOH – hydrocarbon was removed from the EKT and treated by AOP in a Fenton reactor to mineralize the aqueous phase of hydrocarbon; the coupling of EKT and AOP in a Fenton reactor has not been previously reported. Finally, this research delivered soil and water free of hydrocarbons (HCs). Bean germination in the clean soil was possible, as previously reported (Alba et al., 2013). Furthermore, it was possible to reuse the clean water, which was shown by the Deltatox[®] kit test in the EKT to have minimal toxicity.

2. Materials and methods

2.1. Preparation of the cell for electrokinetic treatment of gasoline-polluted soil

EKT at the pilot level was developed using 3.3 m^3 of real Vertisol pellic-type from Querétaro place already polluted with 1126 mg kg^{-1} of gasoline (Fig. 1A – a), which was completely mixed and placed in an acrylic electrochemical cell (Fig. 1A – b). Vertisol pellic soil has a neutral pH of 7.0, an electrical conductivity close to 0.100 dS m^{-1} , and a cationic exchangeable capacity of $10.21 \text{ Cmol Kg}^{-1}$ with at least 30% of clay, as previously reported by our research group (Pérez-Corona et al., 2013b; Alba et al., 2013). The EKT system was built with a 2D circular arrangement of electrodes, applying a constant voltage drop of 20 V, which generated a constant current of 9 A and was used intermittently for 20 days ($4\frac{1}{2}$ h/day). Each day, 60 L of $0.7 \mu\text{M}$ NaOH solution was applied in all pilot cells by irrigation to remove the HCs and collect them as NaOH–HCs in the wastewater compartment (Fig. 1A – c). This arrangement for the treatment had a Ti cathode (–) in the centre surrounded by six $\text{IrO}_2\text{-Ta}_2\text{O}_5 \mid \text{Ti}$ anodes (+). Each electrode had a diameter of 24 cm and was 0.6 m in length, buried only 0.5 m into the ground, and the distance between the electrodes was 117 cm

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