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Combination of Electrokinetic Separation and Electrochemical Oxidation for Acid Dye Removal from Soil

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Abstract: The remediation of kaolin soil contaminated with Acid Blue 25 was performed by a combination of electrokinetic separation and electrochemical degradation. The anionic dye was removed from the soil mainly by electroosmosis towards the cathode, with up to 89% removal being achieved at 30 mA for 7 days. The dye solution was completely mineralized in a separate electrochemical oxidation process using a boron-doped diamond anode. A NaCl solution enhanced the oxidation rate of the dye through indirect oxidation mediated by active chlorine, as well as direct oxidation. The results demonstrate that complete removal of dye from soil can be achieved by electrokinetic separation followed by electrochemical oxidation of effluent with a chloride-containing electrolyte.

Keywords: Acid dye, boron-doped diamond electrode, electrochemical oxidation, electrokinetic soil remediation, electrolyte

INTRODUCTION

The development of textile industries has resulted in large volumes of wastewater containing dyes being discarded annually, creating a major environmental contamination problem. The dyes are often highly toxic due to the high content of aromatic components in the molecules. The

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complexity and variety of dyes makes the problem difficult to resolve (1). Advanced electrochemical oxidation processes have recently been attracting attention for their application in the removal of recalcitrant pollutants from wastewater. Many studies have demonstrated high efficiency mineralization of various dyes in wastewater using high oxygen over-voltage anodes such as SnO_2 , PbO_2 and boron-doped diamond (BDD) electrodes (2). However, there have been few reports addressing the removal of dyes from contaminated soil.

Electrokinetic (EK) soil remediation is an emerging technology which can be used to extract heavy metals and organic compounds from soils, especially low-permeability ones, by applying a direct current between electrodes placed in the soil. Pazos and co-workers showed that an azo dye, Reactive Black 5, could be effectively removed from kaolinite by EK separation, and when concentrated in the electrode chambers, could be destroyed by an additional electrochemical degradation process involving a high concentration of potassium sulfate (3).

For successful application to dye-contaminated soils it is necessary to determine the appropriate EK operational conditions for various dyes, and the posttreatment of dyes in the EK effluent also needs to be considered. Electrolyte solutions are typically used in the EK process to increase the electrical conductivity of the soil. When the pollutant in the EK effluent is decomposed, the rate of electrochemical oxidation may vary with electrolyte type.

In the present study, we investigated the remediation of soil contaminated with an acid dye, using a combination of EK transport and electrochemical oxidation for the complete elimination of the contaminant. In particular, the effect of electrolyte type on EK separation and electrochemical degradation was assessed.

BACKGROUND

Principles of Electrokinetics

Soil remediation by EKs relies on a low-intensity direct current, on the order of mA/cm^2 , via electrodes. Subsurface contaminants in the electric field are transported by several EK phenomena, mainly electromigration and electroosmosis. Electromigration describes the transport of ions and ion complexes towards the electrode of opposite charge, which is largely responsible for generating the electrical current. The rate of electromigration is affected by ionic mobility and the electrical gradient. Pore fluid in the soil medium moves towards the cathode (rarely towards the anode) by electroosmosis. The average velocity of electroosmotic flow is often

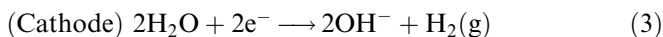
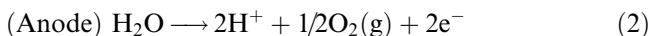
estimated by the Helmholtz-Smoluchowski (H-S) equation (1).

$$v_{eo} = -\frac{D\epsilon_0\zeta}{\eta}E_x \quad (1)$$

According to this equation, the flow velocity is proportional to the electrical gradient (E_x), the zeta potential (ζ) and the dielectric constant (D), and inversely proportional to the viscosity of the solution (η). In equation 1, ϵ_0 is the permittivity of a vacuum. These transport mechanisms allow pollutants to be separated from a soil even if it has low hydraulic conductivity or matrix heterogeneity (4,5).

The mobilization of charged species is influenced simultaneously by electromigration and electroosmosis. While the electromigration of anionic species towards the anode is reduced because of electroosmosis in the opposite direction, cation migration is enhanced as both mechanisms drive cations towards the cathode (6,7).

During the EK process, electrolysis reactions take place at the electrodes, producing protons and hydroxyl ions at the anode (equation 2) and cathode (equation 3), respectively.

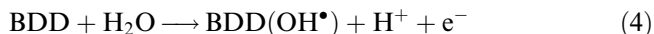


These ions move towards the cathode or anode by electromigration, creating acid and base fronts in the soil, respectively. The change of soil pH may affect sorption, precipitation, and dissociation reactions as well as the zeta potential of the soil surface, which is closely associated with the rate of electroosmosis (8,9).

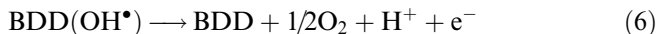
Electrochemical Oxidation using BDD Electrodes

Electrochemical oxidation of pollutants can be achieved by indirect and direct oxidation. Direct (or anodic) oxidation of organic compounds is mediated by electrochemically-generated hydroxyl radicals using only high oxygen overvoltage anodes (10).

BDD electrodes are receiving increasing attention because of their extremely wide potential window ($-1.3 \sim 2.8$ V), inert surface and remarkable corrosion stability, making these the most promising electrodes for anodic oxidation (11). Organic compounds are oxidized by electrochemically-generated hydroxyl radicals (equations 4 and 5) which are weakly adsorbed on BDD electrodes (12).



The reaction in equation 5 competes with the side reaction of hydroxyl radical discharge to O_2 (equation 6).



However, the BDD anode has low activity for O_2 evolution, so it is possible to oxidize toxic and biologically refractory pollutants in wastewater with high current efficiency. In particular, for high organic concentrations and low current densities, the chemical oxygen demand (COD) decreases linearly with charge loading, and at this time the instantaneous current efficiency (ICE) is about 100% (11). The ICE for the anodic oxidation is calculated from the values of COD using equation 7 (13),

$$\text{ICE} = 4FV \frac{[(\text{COD})_t - (\text{COD})_{t+\Delta t}]}{I\Delta t} \quad (7)$$

where $(\text{COD})_t$ and $(\text{COD})_{t+\Delta t}$ are the CODs (in $\text{mol O}_2 \text{m}^{-3}$) at times t and $t+\Delta t$ (respectively), I is the current (A), F is the Faraday constant (C mol^{-1}), and V is the volume of electrolyte (m^3).

Recent studies on electrochemical oxidation using BDD electrodes have shown high efficiency in mineralizing dyes including Orange II, Alizarin Red S and methylene blue (2,11,14,15).

MATERIALS AND METHODS

Soil Preparation

Soil used in this study was kaolin collected from Sancheong-gun in the province of Gyeongsangnam-do, Korea. The air-dried soil was sieved to 1.0 mm (US Standard Screen No. 18) to remove coarse particles. The properties of the model soil are given in Table 1. The anionic dye Acid Blue 25 (AB25; Sigma-Aldrich, USA) was selected as a target pollutant (Fig. 1) representing anthraquinone dyes, which are the second most important class of commercial dyes after azo-compounds. The soil was artificially contaminated by addition of an AB25–electrolyte solution and left for several days to allow sorption of the AB25 to soil. Deionized water was then added to the contaminated soil to achieve an appropriate moisture content for the EK process. The initial AB25

Table 1. Properties of soil used in the study

Property	Value
Texture	Loam
Particle size analysis, % (ASTM D422)	
<2 μm	10.1
2–50 μm	43.4
50–1000 μm	46.5
pH (ASTM D4972)	7.44 (H_2O) 6.43 (CaCl_2)
Moisture content, %	19.9
AB25 concentration, mg/kg soil	105

concentration and moisture content of the soil loaded into the EK cell were about 105 ppm (mg/kg dry soil) and 19.9%, respectively.

Electrokinetic Experiments

A schematic diagram of the EK reactor is shown in Fig. 2a. The rectangular acrylic resin reactor ($4\text{ cm} \times 4\text{ cm} \times 20\text{ cm}$) consisted of a 10-cm-long soil cell with two electrode chambers (60 mL) placed at each end. Dimensionally-stable anode (DSA[®]; $4\text{ cm} \times 4\text{ cm}$; DOES Co. Ltd, Korea) electrodes were used for both the anode and cathode. A purging solution containing electrolyte was continuously supplied into the anode reservoir, and a constant current was applied by a DC power supply.

Table 2 shows the operating conditions of the EK reactors. The applied current varied from 20 to 50 mA and two electrolytes of the same ionic strength (0.05 M Na_2SO_4 and 0.15 M NaCl) were used. In Reactor 3, the effluent that overflowed from the cathode reservoir was reintroduced

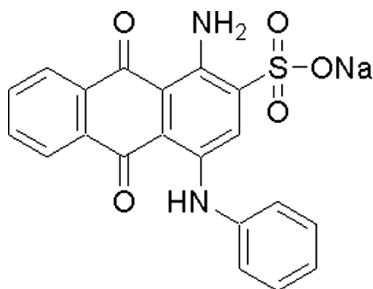


Figure 1. Molecular structure of Acid Blue 25.

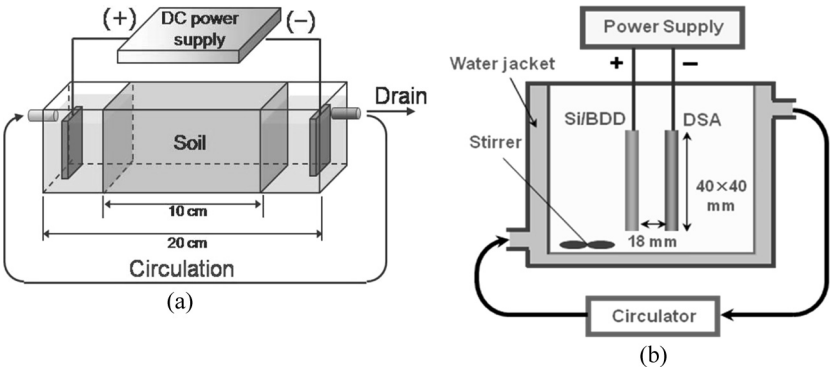


Figure 2. Schematic diagrams of the EK test reactor (a) and the electrochemical oxidation test cell (b).

into the anode reservoir to investigate electrochemical degradation at the anode in the EK system.

During the EK operation the variation of electrical potential through the soil cell, and the amount of electroosmotic flow, were periodically measured. In addition, the change in concentration of AB25 in both electrode reservoirs was also determined. At the end of the experiment the soil was separated into 11 sections which were analyzed for residual AB25 and moisture content.

Electrochemical Oxidation Tests

To assess the effectiveness of treatment of EK effluent containing dyes, electrochemical oxidation tests were performed in a cylindrical batch reactor (11 cm × 15 cm, diameter × length; Fig. 2b). The electrochemical oxidation cell employed a Si/BDD anode (Fraunhofer IST, Germany) and a DSA[®] cathode (4 cm × 4 cm).

An AB25 solution (100 ppm) was used as a synthetic EK effluent and the effect of electrolyte was investigated using three electrolytes; 0.01 M

Table 2. EK test conditions

	Current	Electrolyte	Duration	Effluent
Reactor 1	30 mA	0.05 M Na ₂ SO ₄	7 days	Drain
Reactor 2	20 mA	0.05 M Na ₂ SO ₄	7 days	Drain
Reactor 3	50 mA	0.15 M NaCl	4 days	Circulation

Na_2SO_4 (test 1), 0.01 M Na_2HPO_4 (test 2) and 0.03 M NaCl (test 3). The volume of solution was 500 mL and all tests were carried out under galvanostatic conditions (15 mA/cm^2) at 25°C . During the electrolysis, samples of the solution (5 mL) were periodically removed for the analysis of AB25 concentration and COD.

Analysis

For measurement of the AB25 content in soil samples, the dye was extracted from soil by mixing 2~3 g of soil with 10 mL of deionized water for 18 h in a rotary shaker (40 rpm, 25°C). The extraction procedure was repeated three times to ensure complete recovery of the dye. After the final extraction the overall recovery of the dye was more than 98%.

AB25 was analyzed by high performance liquid chromatography (HPLC) with a Symmetry C18 column (Waters, USA) using acetonitrile/water (50/50, v/v) as the mobile phase. AB25 was detected at 600 nm with a photodiode array detector (Waters 996, USA). The COD was measured by the dichromate method using a COD kit (Humas, Korea) and a DR/2010 spectrophotometer (Hach Company, USA).

RESULTS AND DISCUSSION

Electrokinetic Separation of Acid Blue 25

Electrical Potential Gradient

Figure 3 shows the profile of electrokinetic potential gradient through the EK cell. In all EK tests, the gradient tended to increase gradually and then stabilize. The initial rise in the gradient may have been due to increased soil resistance as the ion concentration within the soil matrix was reduced by ion migration and the neutralization of hydrogen and hydroxyl ions (8).

The rate of increase in the electrical potential gradient was affected by current intensity; the higher the current applied, the more rapid was the increase. In Reactor 3, operating at 50 mA, the electrical potential gradient increased more rapidly than in the other three reactors. However, it stabilized at a lower value (20 V/cm) than in Reactor 1 (operating at 30 mA) due to the higher ion concentration of NaCl than Na_2SO_4 . The total power consumption during the treatment in Reactor 1, 2 and 3 was 1037, 323, and 733 Wh, respectively.

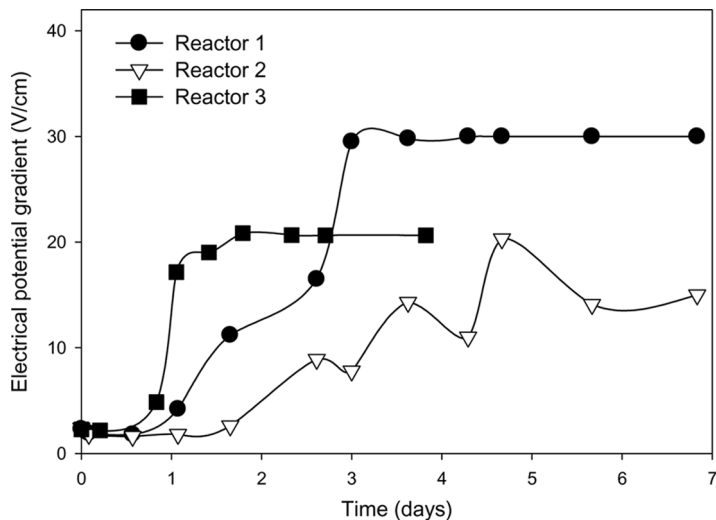


Figure 3. Variation of electrical potential gradient during the EK tests.

Electroosmotic Flow and AB25 Transport

Generally, electroosmotic flow moves towards the cathode because most fine-grained soils are negatively charged, and excess cations in the diffuse double layer (especially hydrogen ions) contribute to the movement of pore fluid (5). In this study, electroosmotic flow followed this generalized pattern, and the accumulated amount of overflow from the cathode reservoir is shown in Fig. 4. The behavior of the electroosmotic flow was similar to that of the electrical gradient. This is explained by the H-S equation, whereby the velocity of electroosmosis is proportional to the electrical gradient. Thus, Reactor 1 (30 mA) produced 223 mL of electroosmotic flow over 7 days, while Reactor 2 (20 mA) produced only 137 mL during the same period. In Reactor 3, where the highest current was applied, 172 mL of electroosmotic flow was generated over 4 days.

The AB25 was mainly transported towards the cathode by electroosmosis in the EK experiments. This was readily observed by the color change in the EK reactor and was confirmed by analysis of the AB25 concentration in the electrode reservoirs (Fig. 5). In reactors 1 and 2, AB25 was only detected in the cathode reservoir and not in the anode chamber. Due to electrolyte circulation, some dye was present in the anolyte in Reactor 3 but the concentration was much lower than that in the catholyte. This indicates that electroosmosis was more dominant than electromigration in causing the migration of AB25 under the test

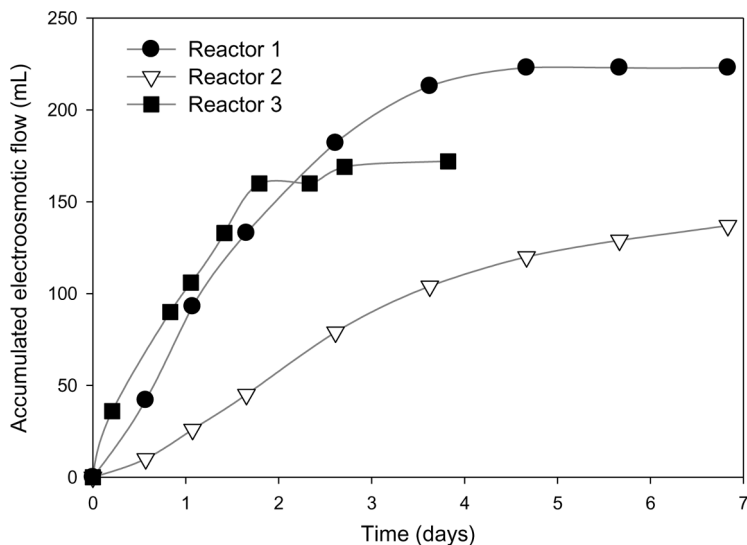


Figure 4. Accumulated electroosmotic flow during the EK tests.

conditions, even though AB25 is an anionic pollutant. However, relative to the initial mass of AB25 in the soil (about 25 mg), only a small amount (<0.2 mg) was detected in effluent samples in all tests.

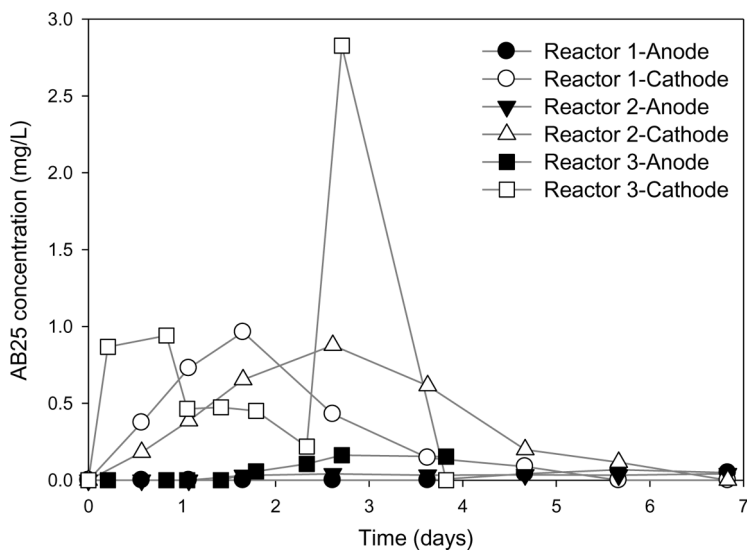


Figure 5. Concentration of AB25 in the electrode reservoirs of the EK reactors.

Efficiency of AB25 Removal

Figure 6 shows the ratio of residual AB25 to its initial mass in the EK cell, after the EK procedure. The dye was comparatively well removed from all sections of the soil cell, except in the middle, and the removal efficiency ranged from 47% to 89%. The removal efficiency was much higher than that estimated from the dye content in the effluent. This was probably because there was a significant loss of dye due to adsorption in compartments of the EK reactors, silicone tubing, and sampling bottles.

Reactor 1 exhibited greater removal (89%) than Reactor 2 (80%), as a consequence of the greater electroosmotic flow. However, the dye accumulated around the middle of the soil cell and near the cathode. As mentioned above, the anionic AB25 is mobilized simultaneously by electromigration towards the anode and electroosmosis towards the cathode, both of which are related to the electrical gradient. However, the velocity of electroosmosis in the EK cell varies with distance from the electrode because the zeta potential depends on the solution pH (H-S equation). Therefore, the velocity of AB25 migration may have varied in different sections of the soil, and this could have caused the dye to accumulate in the middle section of the EK reactor. This phenomenon appeared to be worse with the higher current in Reactor 1

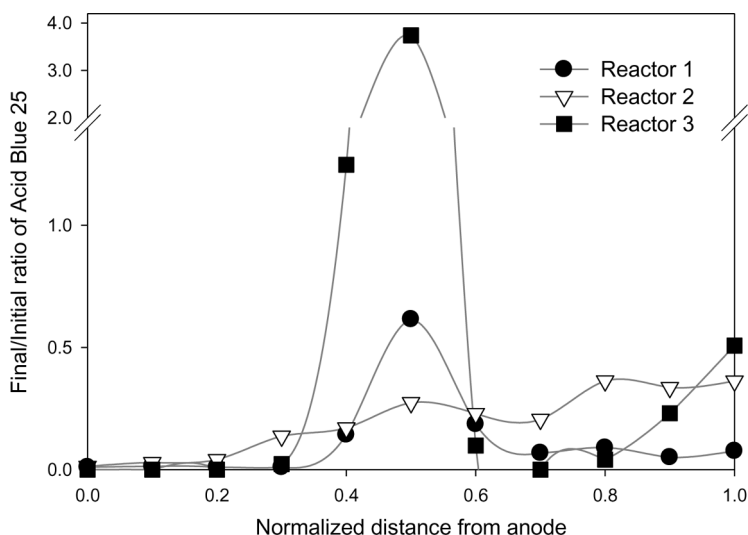


Figure 6. Distribution of residual Acid Blue 25 in the EK cell.

(30 mA) than in Reactor 2 (20 mA). Accordingly, to prevent the accumulation of dye it is necessary to maintain a uniform electroosmotic flow throughout the soil sample by, for example, pH adjustment. In Reactor 3, the AB25 was highly concentrated in the center of the soil sample, where there was approximately a four-fold increase relative to the initial concentration. This may have been caused by the higher current as well as recirculation of the dye. Nevertheless, 47% of the initial dye content was removed during the EK treatment, suggesting that degradation of the AB25 occurred by induction of anodic oxidation coupled with EK separation. A detailed discussion of the anodic oxidation of the dye is provided in the next section.

Electrochemical Oxidation of AB25

The electrochemical oxidation of AB25 in the liquid phase was studied to assess the complete degradation of dye extracted from the soil. Because the dye concentration in effluent samples from the EK tests was too low (<3 mg/L) for assessment of the practical application of the process, an AB25 solution of 100 ppm in electrolyte was used as a synthetic EK effluent. The effect of the electrolyte on the degradation rate was investigated using three electrolytes of the same ionic strength: 0.01 M Na_2SO_4 , 0.01 M Na_2HPO_4 and 0.03 M NaCl . The electrolyte concentration was lower than that used in the EK tests because of the need to maintain the solution resistance above the limit value of the power supply.

Figure 7 shows the variation of AB25 concentration and COD removal in the electrochemical oxidation tests using the BDD anode. The dye concentration declined rapidly and the dye was completely destroyed during the electrolysis in all tests. The COD of the solution also decreased exponentially, and the ICE was below 100% (Fig. 8), which indicates that the oxidation was under mass-transport control (11,13).

The rates of COD (and AB25) removal and the ICE decrease were influenced by electrolyte type. Among the tested electrolytes, the NaCl solution gave the best results. Many studies have reported that organic pollutants can be removed electrochemically by indirect oxidation in the presence of chloride ions (2,16,17). In direct oxidation, organic pollutants are oxidized on the anode surface by adsorbed hydroxyl radicals that are electrogenerated by water discharge (reactions 4 and 5), and it takes place on anodes with high oxygen evolution overpotential, such as BDD electrodes. Indirect oxidation in bulk liquid is mediated by oxidants that are formed electrochemically. The main oxidants include

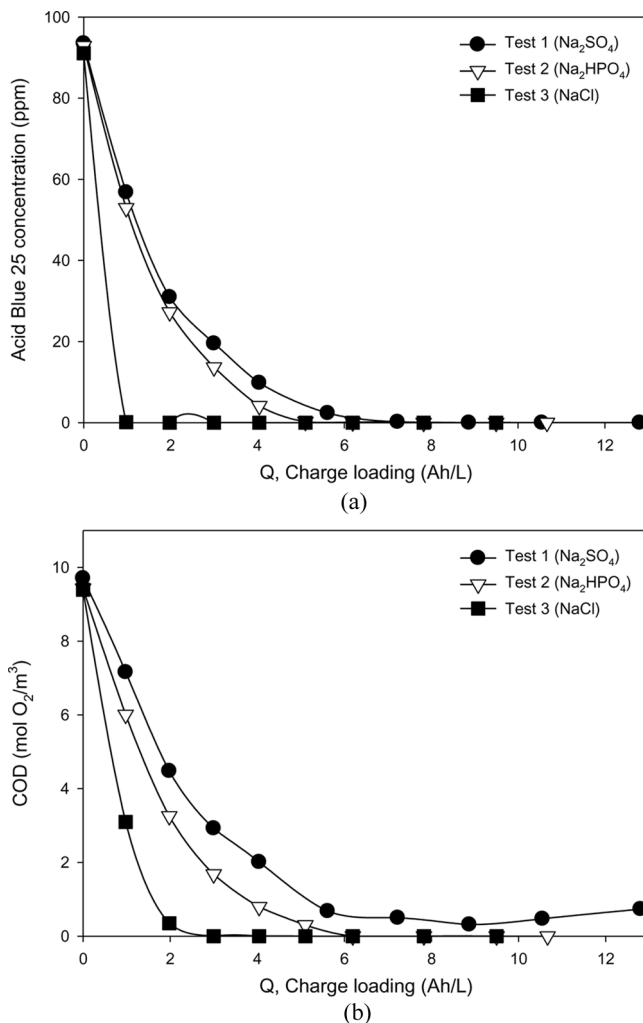
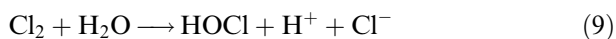


Figure 7. Variation of Acid Blue 25 concentration (a) and COD (b) during electrochemical oxidation tests.

active chlorine, hypochlorous acid or hypochlorite ions, which are anodically produced (equations 8–10) from chlorides in solution (2).



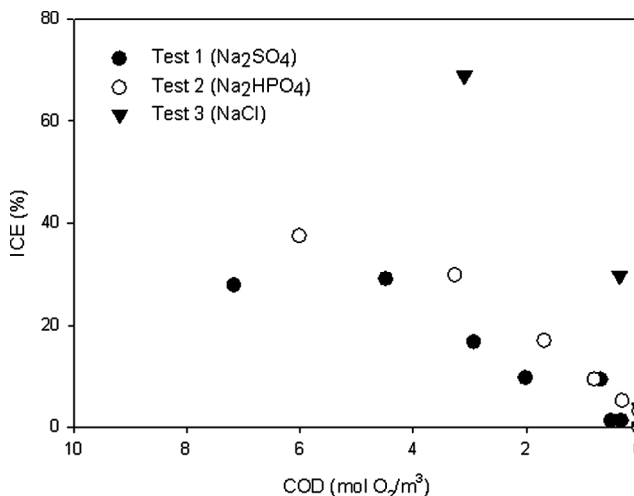
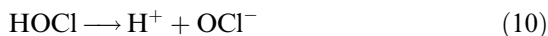


Figure 8. Variation of ICE during electrochemical oxidation tests.



Therefore, the mineralization of AB25 could occur by both direct electrochemical oxidation and indirect oxidation in the presence of chloride ions on a BDD electrode under mass-transport control. These factors contribute to the higher degradation rate of organic pollutants in the presence of NaCl relative to other electrolytes (17).

The degradation efficiency of the dye and rate of COD removal were lower in the tests with Na₂SO₄ and Na₂HPO₄, which may have been a consequence of the absence of chloride. However, it indicated that the electrochemical oxidation of AB25 occurred primarily by direct oxidation on the BDD electrode. The results for the Na₂HPO₄ electrolyte were better than for the Na₂SO₄ electrolyte. Peroxodisulphates are powerful oxidants and can be formed at high temperature in solutions containing sulfates during electrolysis with BDD electrodes (13). However, it seemed that SO₂, a moderate reductant, was formed under the test conditions (16).

Several studies have demonstrated that in the presence of chloride ions, dyes including methylene blue (2) and Reactive Blue 19 (18), as well as real textile waste (19), can also be removed electrochemically by indirect electrolysis using active electrodes such as metal oxide or DSA[®]. This implies that indirect oxidation of AB25 may have occurred during EK treatment on the DSA[®] of Reactor 3 when NaCl was used as the electrolyte, and suggests that the use of NaCl could improve in situ oxidation at the anode in EK processes.

CONCLUSIONS

The following conclusions can be drawn from this study:

- EK separation of the anionic dye AB25 from soil can be accomplished mainly by electroosmosis towards the cathode. The removal efficiency increases with increasing applied current, and the rate of electroosmosis needs to be controlled to prevent accumulation of the dye within the soil.
- The concentrated AB25 solution in the outflow of the EK system can be completely treated in an electrochemical oxidation process using a BDD electrode. Depending on the electrolyte used, direct oxidation of the dye can occur via reaction with electrogenerated hydroxyl radicals, and indirect oxidation may also take place in the presence of chloride ions.
- The complete remediation of soils contaminated with acid dyes can be achieved by a combination of EK separation and electrochemical oxidation using a BDD electrode. The presence of chloride ions in the purging solution can enhance the oxidation rate of the extracted dye in the EK effluent and, moreover, may cause *in situ* oxidation of pollutants migrated to the anode of the EK system.

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