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Electrochemical transformation of trichloroethylene in groundwater by Ni-containing cathodes

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Abstract

In this study, we evaluate the use of different stainless steel (SS) materials as cost-effective cathode materials for electrochemical transformation of trichloroethylene (TCE) in contaminated groundwater. Ni, which is present in certain SS, has low hydrogen overpotential that promotes fast formation of atomic hydrogen and, therefore, its content can enhance hydrodechlorination (HDC). We use a flow-through electrochemical reactor with a SS cathode followed by an anode. The performance of Ni containing foam cathodes (Fe/Ni and Ni foam) was also evaluated for electrochemical transformation of TCE in groundwater. SS type 316 (12% Ni) removed 61.7% of TCE compared to 52.6% removed by SS 304 (9.25% Ni) and 37.5% removed by SS 430 (0.75% Ni). Ni foam cathode produced the highest TCE removal rate (68.4%) compared with other cathodes. The slightly lower performance of SS type 316 mesh is balanced by the reduction in treatment costs for larger-scale systems. The results prove that Ni content in SS highly influences TCE removal rate.

Keywords

electrochemical; nickel; stainless steel; cathode material; trichloroethylene

1. Introduction

Trichloroethylene (TCE) is a halogenated aliphatic organic compound which, due to its unique properties and solvent effects, has been widely used as an ingredient in industrial cleaning solutions and as a “universal” degreasing agent. Once released to the environment, TCE and other chlorinated solvents cause or contribute to widespread groundwater contamination. TCE is among 29 of the chemicals, metals, and other substances most commonly found at US EPA Superfund sites [1]. Because of potential health effects, the US EPA has set Maximum Contaminant Levels (MCLs) for TCE in drinking water at very low

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concentrations ($5 \mu\text{g L}^{-1}$). Development of efficient methods for the dechlorination of TCE dissolved in groundwater without producing harmful byproducts or generating hazardous secondary waste streams is of interest.

A promising method for removal of chlorinated organic compounds (COCs) from groundwater is reductive dechlorination via zero-valent iron [2–5] and its bimetallic/trimetallic forms [6–19]. Due to the fast and effective processes, electrocatalytic reduction of COCs in groundwater has gained interest [20–27]. Transformation at the cathode surface may occur through both direct and indirect mechanisms. Indirect reduction (hydrodechlorination, HDC) involves the reaction of the chlorinated substance with atomic hydrogen. A typical electrochemical dechlorination reaction occurs via a series of steps [28]: transfer of dissolved molecules from the bulk solution to the electrode surface; adsorption of the molecule on the metal surface; formation of chemisorbed hydrogen from H_2O reduction (Volmer reaction); reaction between molecule and chemisorbed hydrogen (Hydrodechlorination); desorption of the hydrogenated products; and, finally, release of reduction products from the electrode surface to the bulk solution.

The rate at which adsorbed hydrogen (H_{ads}) combines to form H_2 is affected by the catalytic properties of the electrode surface [29]. For metals with high hydrogen overpotential, the discharge of the hydrated hydrogen ion is the slow step:

For many metals, this slow discharge is the controlling reaction. The discharge may also be impacted by the reduction of H_2O (Reaction 2),

And the kinetics of target compound reduction via the HDC are influenced by the rates of these processes. Clearly, the overall electrode dechlorination reaction rate is affected by mass transfer, electron transfer and chemical reactions, as well as surface reactions, such as adsorption and desorption [28].

The type of cathode material will have a major effect on HDC. Atomic hydrogen is readily formed at surfaces with the low cathodic hydrogen overpotentials (e.g. Ag, Pt, Pd, Ni, Cu) [30] which were used as working electrodes or as modified electrode materials in the literature [31–34].

Among other catalysts for hydrogen production, Ni-based alloys have a promising electrocatalytic activity for hydrogen production via water electrolysis. A relatively inexpensive material with nickel presence, such as stainless steel, was successfully employed for hydrogen production by microbial fuel cells [35–39]. Increased Ni content in stainless steel decreases the hydrogen overpotential [37–38] which favors formation of hydrogen radicals that react with protons and other reducible species. It is hypothesized that an increase of Ni content in SS cathodes will improve TCE reduction via HDC. The

performance of stainless steel types with different Ni content as cathodes was not investigated for dechlorination and for TCE removal from aqueous solutions.

The performance of stainless steel as a cost-effective cathode material for electrochemical dechlorination of TCE is evaluated in this study. A flow through column with a cathode followed by an anode is used to evaluate improved transformation of TCE using Fe/Ni and Ni foam cathodes.

2. Experimental

All chemicals used in this study were analytical grade. TCE (99.5%) and cis-dichloroethylene (cis-DCE, 97%) were purchased from Sigma-Aldrich. Calcium sulfate was purchased from JT Baker, sodium chloride and sodium bicarbonate from Fisher Scientific. Deionized (DI) water (18.2 M Ω ·cm) obtained from a Millipore Milli-Q system was used in all the experiments. Synthetic groundwater was prepared by dissolving 413 mg L⁻¹ sodium bicarbonate and 172 mg L⁻¹ calcium sulfate in DI water. The concentrations of bicarbonate ions and calcium ions are representative of groundwater from limestone aquifers, resulting in groundwater electrical conductivity of 800 to 920 μ S cm⁻¹. Excess TCE was dissolved into DI water to form a TCE saturated solution (1.07 mg mL⁻¹ at 20°C), which was used as a stock solution for preparing aqueous TCE solutions. The feedstock solution was stored in common Tedlar® bags. Headspace in the bag was minimized to limit TCE losses to the gas phase. Initial TCE concentration was 5.3 ppm, and initial pH of the synthetic groundwater was 8 $\pm$ 0.3 with an initial oxidation-reduction potential (ORP) of 210 $\pm$ 5 mV. The temperature was kept constant at 20°C. Column experiments were conducted under Darcy's velocity of 0.25 cm min⁻¹ (2.8 mL min⁻¹).

Constant flow velocity was maintained by a peristaltic pump (Cole Parmer, Masterflex C/L). Constant current was applied by an Agilent E3612A DC power supply during treatments. TCE and cis-DCE concentrations were measured by a 1200 Infinity Series HPLC (Agilent) equipped with a 1260 DAD detector and a Thermo ODS Hypersil C18 column (4.6 $\times$ 50 mm) as described in Mao et al. 2011. Analysis of chlorides was performed using an ion chromatography (IC) instrument (Dionex 5000) equipped with an AS20 analytical column. A KOH solution (35 mM) was used as a mobile phase at a flow rate of 1.0 mL min⁻¹. pH and ORP of the electrolyte were measured by pH and ORP meters, respectively, with corresponding microprobes (Microelectro, USA). The microprobes allow the measurement of these parameters using a small amount of liquid ($\approx$ 0.2 mL).

Ti/mixed metal oxide (MMO) mesh (3N International) was used as anode. The Ti/MMO electrode consists of IrO₂ and Ta₂O₅ coating on a circular titanium mesh 3.6 cm in diameter and 1.8 mm thick. Three types of stainless steel woven mesh (304, 316 and 430) were evaluated as cathodes (McMaster-Carr, IL). The main difference between these SS types is Ni content (Table 1). The cathodes were 3.6 cm diameter disks with a cross sectional area of 10.2 cm². Mesh characteristics are summarized in Table 1 in terms of mesh number (number of lines of mesh per inch, n), wire diameter, pore size (space between adjacent wires based on the given geometry of the mesh), and open area. These are calculated according to Zhang et al. 2010 [39]. Iron foam with 100 pores per inch (PPI) 90% iron and 10% nickel, was

purchased from Heze Jiaotong Group Corp., China, and was labeled Fe/Ni1. Iron foam with 45 PPI, 98% iron and 2% nickel, was purchased from Aibixi Ltd., China, and was labeled Fe/Ni2. Ni foam (100 PPI, Purity>99.99%) was purchased from MTI corporation, CA.

A vertical acrylic column was used as electrochemical flow-through reactor (Figure 1). The electrode arrangement used in this study was an upstream cathode and a downstream anode with a spacing of 2.5 cm. This arrangement minimizes the competition between TCE and oxygen (produced at the anode) for the cathodic reduction [20]. The experiments were conducted with 430, 304 and 316 SS type mesh cathodes and Fe/Ni1, Fe/Ni2 and Ni foam under a current intensity of 60 mA and a flow rate of 2.8 mL min⁻¹. The 60 mA current enables formation of gas bubbles on the electrodes confirming that all cathodes worked at a hydrogen-releasing potential, ensuring that HDC is the main TCE degradation mechanism. Samples were collected during treatment from Port 2 (Figure 2) and chemical analysis was immediately conducted. Final TCE degradation (FDE, %) was calculated by:

$$FDE = \frac{c_0 - c_t}{c_0} * 100 \quad (3)$$

where c_0 is the initial TCE concentration (mg L⁻¹) and c_t is TCE concentration at a defined time during treatment (mg L⁻¹).

3. Results and discussion

3.1 The performance of stainless steel mesh cathodes for TCE degradation

Changes in normalized TCE concentration using SS 430, 304 and 316 cathodes and their FDE correlation with Ni content in SS are shown in Figure 2. The transformation rates of TCE can be described by pseudo-first-order kinetics ($\ln C_0/C = kt$). The observed pseudo-first order coefficients are normalized by the surface area to solution volume in the reactor as $k_{sv} = k (V/A)$ where V is the volume of the electrolyte in the reactor (L) and A is the geometric surface area of the electrode (m²). TCE removal rates, pseudo-first order coefficients and the chloride recoveries achieved by the tested cathode materials are given in Table 2.

The mass transfer to the electrode surface is one of the limiting factors for the HDC mechanism in the flow-through reactors. Increasing the active electrodes' surface area improves the reaction between the reactive species via HDC. Doubling the electrode surface area from 40.4 cm² to 80.8 cm² improved TCE removal rate from 52.4 % to 61.7% using SS 316 (mesh size n = 20) cathode. This confirms that an increase in the surface area enhances hydrogen evolution rates and therefore improves the HDC mechanism for TC removal, although the increase in removal rates is not linearly related to the increase in surface area. However, there was no increase in removal rate with SS 316 (n=100), although the surface area was larger than that of SS 316_2 (Table 1). This is a consequence of the bubbles' entrapment within the small pore size of SS 316 (n=100). The influence of pore size to bubbles entrapment is consistent with *Zhang et al. 2010* [39]. The pore size of SS 316 (n=100) is 0.014 cm compared to 0.086 cm pore size of SS 316_2. The bubbles' entrapment lowers the active surface area of the electrode for the HDC reactions [40, 41]. This effect is dominant in flow-through reactors where the bubbles' accumulation at the lower side of the

cathode covers a large area of the cathode surface. Additionally, a lower flow rate contributes to the bubbles' entrapment. For further experiment, the SS mesh (n=20) with 80.8 cm² area is used as a cathode.

It is evident that Ni content in the SS cathodes influences the rate of TCE hydrodechlorination and final amount of removed TCE (Figure 2, Table 2). The results confirm that TCE FDE increases with increased Ni content in the SS cathode. A linear correlation (Figure 2b) exists between Ni content in SS cathode and removal of TCE. The HDC reduction mechanism occurs via a reaction of reducible species with atomic hydrogen. For metals with low hydrogen overpotentials, the formation of atomic hydrogen is fast and therefore the kinetic of HDC increases. An increase of Ni content in SS decreases the hydrogen overpotential [38].

The release and accumulation of chlorides during treatment further confirms TCE degradation. cis-DCE, a chlorinated degradation product, was not detected in the effluent. Within the limits of experimental precision, influent and effluent pH and ORP values were similar. Maintaining natural pH and ORP conditions after treatment is valuable since changes in their values could affect groundwater quality.

3.2 The performance of foam cathodes for TCE degradation

The use of Ni foam cathode and Fe/Ni with different Ni content and PPI (Fe/Ni1 and Fe/Ni2) was evaluated for transformation of TCE (Figure 3, Table 3). Foam materials have large active surface area which improves transformation. However, the relatively slow flow rate (2.8 mL min⁻¹) and the dense foam structure limited the movement of hydrogen gas bubbles from the cathode vicinity. The accumulation of bubbles near the cathode limited the interaction between TCE molecules and the cathode surface due to lowering of the active cathode surface area. To lower the effect of bubble accumulation, the foam cathodes were perforated (0.5 cm holes). Ni foam was used to test the influence of electrode perforation. TCE removal increased from 29.6% using Ni foam cathode without perforation to 68.4% when perforated Ni foam was used. The perforated foam cathode reduced accumulation of bubbles within the cathode vicinity.

Ni foam is an ideal cathode substrate for hydrogenation catalysts since it possesses the properties of good electrical conductivity, high specific surface area, uniform pores, high H₂ uptake capacity and good mechanical strength. Due to the Ni electrocatalytic activity towards hydrogen, the Ni foam cathode produced the highest TCE removal. The Fe/Ni2 material is more dense (100 PPI) and contains more Ni (10%) than Fe/Ni1 foam (45 PPI and 2% Ni). It would be assumed that Fe/Ni2 foam performance for TCE would significantly overcome the Fe/Ni1 activity toward TCE hydrodechlorination. However, the removal rate was significantly higher for Fe/Ni1 since the dense foam structure causes bubble entrapment.

Despite its slightly reduced performance, the cost of SS 316 mesh materials is one-fourth that of the Ni foam cathode. Balancing performance and cost, it is concluded that SS316 can be successfully used as cathode material in larger-scale flow-through reactors using a cathode followed by an anode electrode sequence.

3.3 Electrochemical process feasibility and cost

Electrochemical technologies for groundwater treatment use reactors that apply a low-level DC through electrodes into contaminated wells. This approach enables *in situ* manipulation of groundwater chemistry through electrolysis to create conditions favorable for either reduction or oxidation of the contaminants. The *in situ* implementation, limited need of additional chemicals and the control of the reactions are the main advantage of the electrochemical processes. Currently, the most comprehensive cost analysis of the electrochemical treatments is based on F.E. Warren AMB field demonstration [42]. The authors' electrochemical groundwater treatment approach to remove TCE was similar to the one applied in this study. The estimated cost of the design and construction of the demonstration-scale treatment was \$4,930 per m². With certain advances in the treatment design, we found that the cost of the electrochemical treatment was competitive with the one incurred by a zero-valent iron permeable barrier.

It was estimated that the cost of the electrodes material (Ti/MMO) could reach 18% of the total treatment cost. The Ti/MMO cost is approximately \$0.5 per cm² while SS mesh type 316 material costs \$0.04 per cm² (calculated from the same source and taking into account that 4 sheets of SS type 316 material were used as the cathode). It is evident that the overall cost of the electrochemical treatment would greatly decrease with the utilization of low-cost cathode material such as SS mesh type 316.

4. Conclusions

Stainless steel (SS) is evaluated as a potential cost-effective cathode material for degradation of TCE in aqueous solution. SS was used as cathode in a flow-through electrochemical column with an upstream cathode and a downstream anode. Stainless steel meshes with different Ni content were tested as cathodes. Increasing the Ni content positively influences TCE removal rate. Comparing SS 316_100 and SS 316_2 which differ in both pore size and surface area confirms that pore size is an important characteristic to evaluate when using mesh electrodes. It is important to optimize the cathode mesh as well as the foam pore size and surface area. The larger surface area allows more hydrogen evolution, but smaller pore size cause bubble entrapment that lowers the electrode surface area. The results demonstrate that using commonly available stainless steel can be cost-effective for electrochemical treatment of TCE in groundwater with emphasis on the Ni content as a key factor to enhance *in situ* implementation, and reduce costs.

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Highlights

- Stainless steel mesh is a cost-effective cathode material.
- Dense mesh provides large surface area but causes bubble entrapment within pores.
- Increasing the Ni content in stainless steel positively influences TCE removal.

Figure 1.
An electrochemical flow-through reactor

Figure 2.

a) TCE decay by different SS cathodes during treatment, and b) correlation between Ni content and TCE FDE (conditions: 60 mA current, 2.8 mL min⁻¹ flow rate, 5.3 mg L⁻¹ TCE concentration)

Figure 3.

The influence of different cathode foam materials on TCE removal (conditions: 60 mA current intensity, 2.8 mL min⁻¹ flow rate, 5.3 mg L⁻¹ TCE concentration)

Table 1

SS woven mesh characteristics

SS type	Ni (%)	Mesh number (n)	Number of disks	Wire diameter (cm)	Pore size (cm)	Open area (%)	Calculated surface area (cm ²)
430	0.75	20	4	0.041	0.086	46	80.8
304	9.25	20	4	0.041	0.086	46	80.8
316	12	20	4	0.041	0.086	46	80.8
316_2	12	20	2	0.041	0.086	46	40.4
316_100	12	100	4	0.011	0.014	30	98.4

Table 2

FDE, the pseudo-first order coefficients and the chloride recoveries after TCE electrochemical degradation with SS cathodes

Cathode material	FDE (%)	Pseudo-first-order rate, k (min^{-1})	Normalized pseudo-first-order rate, k_N ($\text{L m}^{-2} \text{min}^{-1}$)	Chloride (mg L^{-1})	Chloride recovery (%)
SS 430	37.5	0.0034	0.148	1.21	94.5
SS 304	52.6	0.0044	0.192	1.78	89.4
SS 316	61.7	0.0059	0.257	2.42	88.9
SS 316_2	52.4	0.0053	0.231	1.87	97.6
SS 316_100	59.9	0.0060	0.262	2.05	97.3

Table 3

FDE, the pseudo-first order coefficients and the chloride recoveries after TCE electrochemical degradation with foam cathodes

Cathode material	FDE (%)	Pseudo-first-order rate, k (min^{-1})	Normalized pseudo-first-order rate, k_{sv} ($\text{L m}^{-2} \text{min}^{-1}$)	Chloride (mg L^{-1})	Chloride recovery (%)
Fe/Ni1	43.5	0.0057	0.249	1.97	93.5
Fe/Ni2	31.9	0.0037	0.161	1.63	89.7
Ni	68.4	0.0072	0.314	2.92	95.2