Persulfate regeneration of trichloroethylene spent activated carbon

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ABSTRACT

The objective of this study was to demonstrate the regeneration of trichloroethylene (TCE) spent activated carbon using persulfate oxidation and iron activated persulfate (IAP) oxidation. Both processes resulted in decreases in the adsorbability of regenerated activated carbons. IAP was shown to rapidly degrade the aqueous TCE and causes a significant mineralization of the TCE. The release of chloride ions provided evidence of this. Persulfate oxidation mainly resulted in desorption of TCE from the activated carbon and only partial mineralization of the TCE through a carbon activated persulfate reaction mechanism. Concerning destruction of the TCE, in the regeneration test using persulfate, 30% of the original TCE was present in the solution and 9% remained on the activated carbon after the first regeneration cycle. In contrast, in the test that used IAP, it was observed that no TCE was present in the solution and only approximately 5% of the original TCE remained on the activated carbon after the first regeneration. Following the regeneration cycles, elemental analysis was carried out on the samples. BET surface area and EDS analysis showed some effects on the physico-chemical properties of the activated carbon such as a slight decrease in the surface area and the presence of iron precipitates on the carbon.
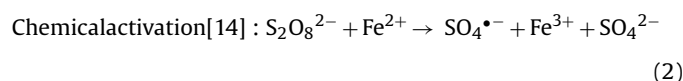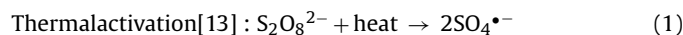
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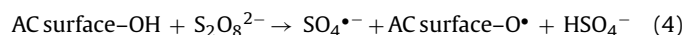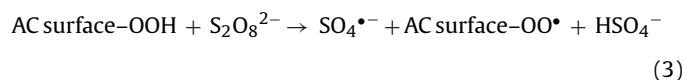
1. Introduction

Trichloroethylene (TCE), a nonflammable, colorless liquid with a sweet odor and burning taste, is widely used as a solvent to remove grease from metal machine parts. TCE has been found in at least 852 of the 1430 National Priorities List sites compiled by the United States Environmental Protection Agency [1]. Activated carbon (AC) adsorption is mainly used for the treatment of above-ground contaminant. However, other sorption control methods, such as the usage of permeable reactive barrier (PRB) zones [2], can also be implemented to limit the migration of contaminants, such as TCE, from groundwater systems. In order to allow the continuous use of the AC and reduce the cost of replacing AC that has been exhausted; regeneration or in situ regeneration for PRB application has to be considered. Adsorption is a heat-sensitive process. Therefore, the spent AC is commonly regenerated by thermal means, such as a rotary kiln and multiple hearth furnaces. These processes present some clear disadvantages, for example: high energy requirement (a temperature above 800 °C is required); gaseous emissions containing concentrated volatiles stripped from the AC; carbon monoxide formed as a result of incomplete combustion [3].

An alternative technique is chemical regeneration. The Fenton-driven mechanism (i.e., the oxidation of ferrous ion (Fe²⁺) by hydrogen peroxide for the production of hydroxyl radicals) for

regenerating spent AC has been successfully demonstrated to be an effective chemical process [4–6]. Persulfate ($S_2O_8^{2-}$), a strong oxidant $E° = 2.01$ V and fairly stable in the subsurface, is the newest form of oxidant currently being used for in situ chemical oxidation remediation of subsurface contamination [7,8]. Both thermal and chemical (e.g., by Fe²⁺) persulfate activation can generate sulfate radicals ($SO_4^{•-}$), a more powerful oxidant $E° = 2.4$ V [9] which has been demonstrated to effectively degrade TCE [10–12]. The thermal and chemical generation of sulfate radicals is shown in Eqs. (1) and (2), respectively.

$$\text{Thermal activation [13]}: S_2O_8^{2-} + \text{heat} \rightarrow 2SO_4^{•-} \qquad (1)$$

$$\text{Chemical activation [14]}: S_2O_8^{2-} + Fe^{2+} \rightarrow SO_4^{•-} + Fe^{3+} + SO_4^{2-} \qquad (2)$$

It has also been suggested that AC containing oxygen functional groups may act as an activator of the electron-transfer mediator [15]. Thus, the AC may facilitate the decomposition of persulfate with releases of organic radicals and sulfate radicals during radical propagation processes [15]. The following reaction mechanisms are proposed based on the findings of Kimura and Miyamoto [15] and Georgi and Kopinke [16]:

$$\text{AC surface–OOH} + S_2O_8^{2-} \rightarrow SO_4^{•-} + \text{AC surface–OO}^{•} + HSO_4^{-} \qquad (3)$$

$$\text{AC surface–OH} + S_2O_8^{2-} \rightarrow SO_4^{•-} + \text{AC surface–O}^{•} + HSO_4^{-} \qquad (4)$$

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In light of these findings, the purpose of this study was to investigate the regeneration of spent AC by persulfate, and sulfate radicals produced using ferrous ion activated persulfate.

2. Materials and methods

2.1. Chemicals

Water used for the preparation of the TCE contaminated solutions was purified using a Millipore reverse osmosis (RO) purification system. The chemicals used were purchased from the following sources: TCE (>99.5%) and sulfuric acid (>99.8%) were purchased from Fluka; n-pentane (min. 99.9%) was purchased from Tedia; sodium thiosulfate pentahydrate (min. 99.5%) and sodium chloride (min. 99.8%) were purchased from Riedel-deHaën; sodium persulfate (min. 99.0%), and hydrochloric acid (min. 37%) were purchased from Merck. A commercial AC, Calgon Filtrasorb 400 (F400), which is bituminous-coal-based carbon, was purchased from the Calgon Carbon Corporation. The AC was purified using an acid washing procedure (5% HCl) to remove impurities present and then rinsed with RO water in accordance with the procedure outlined by Cao et al. [17]. Thereafter, the AC was dried at 105 °C for 1 h prior to storage in a desiccator. Note that the notation of AC was used to represent the original activated carbon which had already been acid-washed.

2.2. Experimental methodology

2.2.1. Regeneration of spent AC by persulfate

The TCE solution (100 mg/L, pH 3 adjusted by H_2SO_4) was prepared by adding the required amount of pure TCE and stirring for 12 h in a 1.3 L heavy-walled plain pressure reaction flask (IWAKI 7740 glass) in which a zero head space was maintained. The flask was placed in a temperature-controlled chamber at 20 °C and the top of the flask was covered and clamp-sealed with a flat Teflon reaction head containing Teflon-lined septum ports. One gram of AC was added to the TCE solution and allowed to reach sorption equilibrium by magnetic-stirring for 24 h. Note that a preliminary adsorption kinetic experiment showed equilibrium was reached after around 6 h (data not shown) and therefore, to ensure that complete equilibrium was achieved, a 24 h period was used. A sodium persulfate concentration of 5 g/L was prepared in a single dose to initiate the regeneration process. TCE, persulfate and chloride concentrations were measured during the course of the 3 h reaction. At each sampling interval, 10 mL of solution was removed using a gas-tight syringe (SGE gas-tight syringe, fitted with a push-button luer lock valve) and filtered using a PTFE filter (0.2 μm, Toyo stainless syringe holder). A total of 90 mL was taken, which accounts for only a 7% variation in volume and therefore, adjustment for the measured concentration was not carried out. Thereafter, the solution was decanted and a newly prepared TCE solution was added into the reaction flask to initiate another equilibrium adsorption run. This sequence of procedure is hereafter defined as one regeneration cycle. These regeneration steps were repeated 5 times. A control test to observe TCE degradation by persulfate oxidation in the absence of AC was also conducted.

2.2.2. Regeneration of spent AC by iron activated persulfate (IAP)

In the experiments where the IAP process was used to produce the sulfate radicals for regeneration of the spent AC, the procedure used was similar to those described in previous persulfate regeneration process. However, at the beginning of the experiments using the IAP process, 100 mL of solution was removed from the reaction flask to allow ferrous ion activator stock solution (0.2 M, pH 3) to be pumped (Cole-Parmer Masterflex C/L variable-speed peristaltic pump) into the flask at a rate of 0.5 mL/min for 3 h (i.e., a total of

90 mL injection). A control test to observe TCE degradation by IAP oxidation in the absence of AC was also conducted.

2.2.3. Comparison of AC regeneration by persulfate and IAP

In order to confirm the distribution of TCE during the regeneration process, the first regeneration step was carried out again for two oxidation processes and TCE quantities in the aqueous and sorbed phases were analyzed. The experimental procedures were in accordance with those described in Sections 2.2.1 and 2.2.2 for persulfate and IAP oxidation regeneration processes, respectively.

2.3. Analytical method

2.3.1. Aqueous phase analysis

For TCE analysis, a 2.5-mL aliquot was placed in a 5 mL brown bottle and extracted along with 1.5 mL of pentane. The extraction vials were shaken for 5 min on a vortex shaker (Thermolyne Type 65800) and then equilibrated for 5 min. The TCE extract was measured using gas chromatography/flame ionization detector (Agilent 6890N) in accordance with operational conditions outlined by Liang et al. [18]. Chloride ions (i.e., the endproduct of TCE mineralization) were analyzed using a Metrohm 790 ion chromatograph coupled with a conductivity detector using a Metrosep A Supp 5 column (method detection limit: 0.056 mg/L). pH was monitored using a ROSS pH combination electrode and a pH/Ion meter (Thermo Orion 720A+). The concentration of persulfate was monitored by iodometric titration of 2 mL aliquots [19]. All analysis was performed in duplicate and averaged data has been presented. Control tests in the absence of persulfate were carried out in parallel.

2.3.2. Residual AC analysis

The residual AC was filtered and approximately 0.2 g of AC was weighed and immediately placed in 5 mL brown bottles containing 2 mL of pentane for a subsequent 2 min extraction at 1400 rpm on a vortex shaker. BET surface area and pore volumes were determined using N_2 adsorption at 77 K (Micrometrics ASAP 2020, a high surface area and porosimetry analyzer). The surface morphology and chemical composition of the AC was analyzed using a JEOL JSM-6700F scanning electron microscope (SEM) equipped with an Oxford Energy 400 X-ray energy dispersive spectrometer (EDS). The AC, P-AC, and IAP-AC samples were digested using nitric acid in accordance with the procedure described by Huiling et al. [5]. Extracts were analyzed for iron using a PerkinElmer AAnalyst 100 flame atomic absorption spectrophotometer.

3. Results and discussion

3.1. Regeneration of spent AC by persulfate

Fig. 1 shows the equilibrium adsorption in every generation step. TCE, chloride and persulfate variations during the course of the whole generation reaction are also shown. The amount of TCE remaining in the aqueous phase after adsorption equilibrium for every regeneration cycle gradually increased and hence the observed adsorbability (i.e., q_e) decreased. This could be due to the following reasons: (1) incomplete removal of TCE from AC by persulfate; (2) following persulfate oxidation, the number of surface acidic functional groups increased [20,21] and they initiated the process of water molecule adsorption via hydrogen bonding to reduce TCE adsorption [22]; (3) the interaction of persulfate and AC may involve the exchange of an oxonium-hydroxyl group (e.g., AC surface) $C–OH_2^+OH^-$) with persulfate anions [15,23] and weaken persulfate strength in accordance with Eqs. (5) and (6); (4) oxidation may destroy AC structure and decrease AC surface area [24].
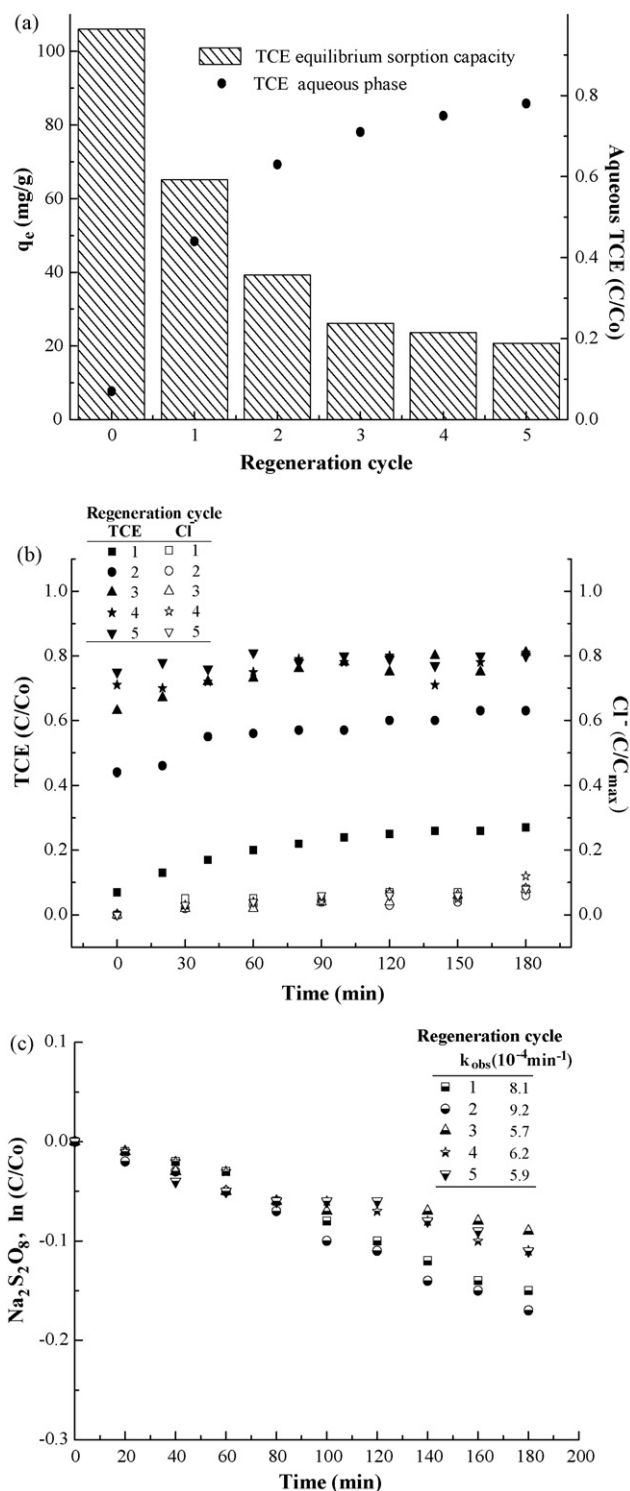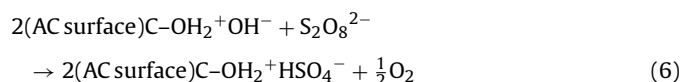
$$S_2O_8^{2-} + H_2O(\text{self-decomposition}) \rightarrow 2HSO_4^- + \tfrac{1}{2}O_2 \qquad (5)$$
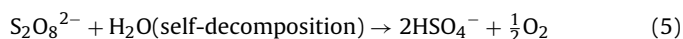
Fig. 1. Persulfate regeneration of TCE spent activated carbon. (a) Adsorption capability as a function of regeneration cycle; (b) Variation of TCE and chloride concentrations and (c) persulfate decomposition during the course of regeneration. $[Na_2S_2O_8] = 5\,g/L$; $[AC] = 1\,g/L$; $[TCE] = 100\,mg/L$; pH 3. *Note*: $[Cl^-]_{liberated}$ $\% = [Cl^-]_{measured}/[Cl^-]_{max}$, $[Cl^-]_{max} = 3[TCE]_o$.

$$2(AC\,surface)C\text{–}OH_2{}^+OH^- + S_2O_8{}^{2-}$$

$$\rightarrow 2(AC\,surface)C\text{–}OH_2{}^+HSO_4{}^- + \tfrac{1}{2}O_2 \qquad (6)$$

TCE desorption by persulfate can be seen in Fig. 1b. After comparing the initial and final TCE concentrations, the results indicate that, during the course of the 3 h persulfate oxidation, the earlier regeneration cycles (i.e., 1st and 2nd cycles) resulted in approximately 20–30% removal of TCE from the AC while the later cycles resulted in removal of <10%. After five regeneration cycles, sorption was reduced by around 70% (see Fig. 1a). It should be noted that this percentage decrease in adsorbability is not equivalent to an actual decrease in the quantity of equilibrium adsorption due to the reason (1) explained above. Also, it was observed that (Fig. 1b), for all five regeneration tests around 10% of the chloride ions were liberated after 3 h of oxidation.

The AC may enable the decomposition of persulfate with releases of sulfate radicals in accordance with Eqs. (3) and (4). Therefore, during the course of persulfate regeneration, destruction of TCE by persulfate or sulfate radicals could be expected and the release of chloride is evidence of TCE mineralization. The decrease in the adsorbability and extent of TCE removal from AC could also be related to the persulfate decomposition rate. It can be seen in Fig. 1c that the observed persulfate pseudo-first-order decomposition rate constants were higher for the 1st and 2nd regeneration cycles and thereafter slowed down (see insert table in Fig. 1c). The pH of the solution dropped slightly from 3.0 to 2.8 in each regeneration cycle.

3.2. Regeneration of spent AC by IAP

Results obtained from the IAP regeneration are illustrated in Fig. 2. Adsorbability losses of approximately 60% (see Fig. 2a) are similar to those observed in the persulfate regeneration process. However, it can be seen that the remaining aqueous TCE was rapidly and completely degraded by the IAP process and that chloride concentrations were significantly increased (Fig. 2b). Furthermore, the percentage of chloride liberated was higher than that liberated upon mineralization of TCE in the aqueous phase. This indicates that TCE sorbed by AC can also be degraded to release chloride as a net result of direct oxidation on sorbed TCE and desorption following indirect oxidation in the aqueous phase. For example, during the 2nd regeneration cycle, the concentration of aqueous TCE was ~50% upon sorption equilibrium and the concentration of chloride ions liberated was ~70% after 3 h of IAP oxidation. That is approximately 20% higher than that which was predicted to be liberated from mineralization of the aqueous TCE. However, there are still some quantities of TCE or chloride ions that were not accounted for. The reasons could possibly be due to the difficulty in removing TCE from the AC or the formation of precipitates (e.g., ferric chloride) deposited on the AC surface.

The persulfate decomposition rate constants were similar in each regeneration cycle (Fig. 2c) and also comparable to the rate constant of $3.6 \times 10^{-3}\,min^{-1}$ determined after the control test which was carried out without the presence of AC. At the end of experiment (3 h), the total amount of Fe^{2+} that had been injected into the reaction flask was 18.0 mmol and the average persulfate concentration following consumption was 48% of the initial amount (10.1 mmol equivalent). The molar ratio of Fe^{2+} and $S_2O_8{}^{2-}$ used was 1.8, which is analogous to the theoretical stoichiometric molar ratio of 2 in accordance with Eq. (7). Therefore, it is evident that persulfate consumption by AC is insignificant when compared to consumption by Fe^{2+} activation. These results suggest that, in terms of the destruction and mineralization of TCE, the IAP process can regenerate TCE spent AC more effectively than using persulfate alone. The pH of the solution decreased from 3.0 to 2.5 in each regeneration cycle.

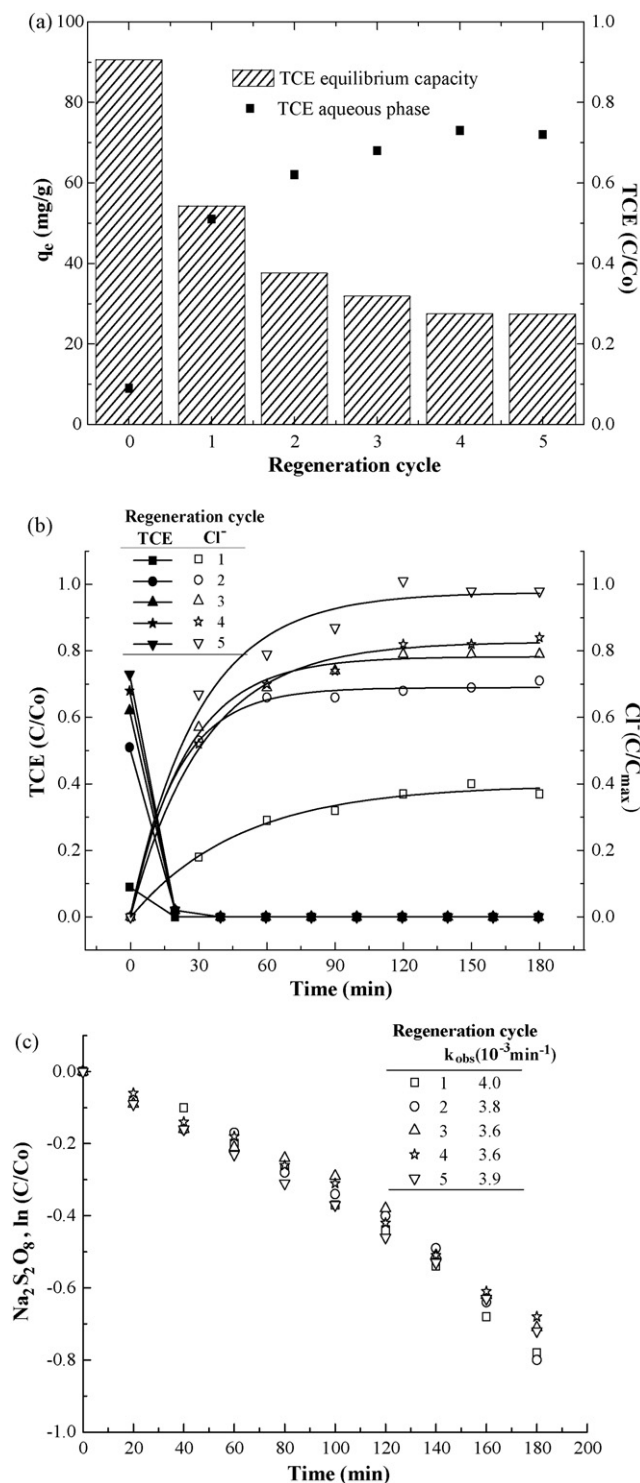$$S_2O_8{}^{2-} + 2Fe^{2+} \rightarrow 2Fe^{3+} + 2SO_4{}^{2-} \qquad (7)$$
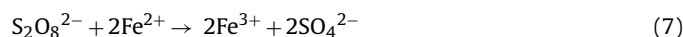
Fig. 2. Iron activated persulfate regeneration of TCE spent activated carbon. (a) Adsorption capability as a function of regeneration cycle; (b) Variation of TCE and chloride concentrations and (c) persulfate decomposition during the course of regeneration. $[Na_2S_2O_8] = 5$ g/L; $[AC] = 1$ g/L; $[TCE] = 100$ mg/L; $[Fe]$ stock $= 0.2$ M and pumping rate $= 0.5$ mL/min, pH 3. *Note*: $[Cl^-]_{liberated}$ % $= [Cl]_{measured}/[Cl]_{max}$, $[Cl]_{max} = 3[TCE]_o$.

3.3. Comparison of two different regeneration processes

In order to confirm whether TCE was being removed, destroyed, or mineralized, an additional test, i.e., only one regeneration cycle, was conducted and aqueous and sorbed phases of TCE were ana-
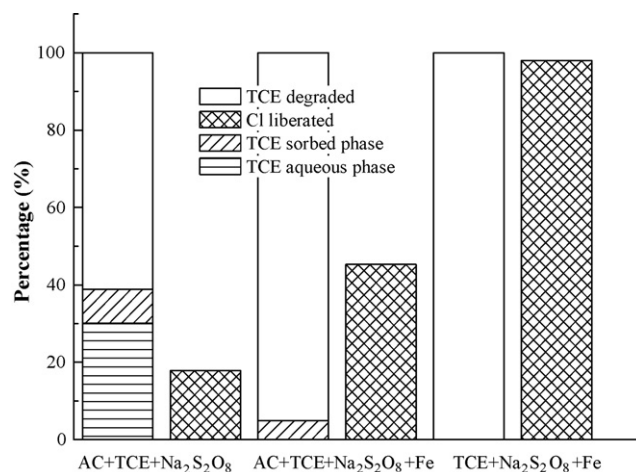

Fig. 3. A comparison of different regeneration processes. *Note*: $[Cl^-]_{liberated}$ % $= [Cl]_{measured}/[Cl]_{max}$, $[Cl]_{max} = 3[TCE]_o$.

lyzed. Activated persulfate oxidation of TCE by gradually adding Fe^{2+} in the absence of AC was also conducted for the purpose of comparison. Results are shown in Fig. 3. The percentages of TCE that were destroyed, sorbed or that remained in the aqueous phase at the end of the persulfate regeneration process (3 h) are 41%, 9%, and 30%, respectively. Therefore, it can be seen that a significant percentage of TCE was destroyed by AC activated persulfate oxidation and the results demonstrate the validability of the reaction mechanisms of Eqs. (3) and (4). Note that a control test on persulfate oxidation of TCE showed no reduction in the concentration of TCE during a 3 h reaction. As speculated, TCE cannot be completely removed from AC by persulfate alone. In the control test (i.e., IAP oxidation of TCE in the absence of AC), in contrast with the IAP process in the presence of AC, no TCE remained in the solution. However, there was still residual TCE present on the AC. Nevertheless, it is clear that the IAP process is a very aggressive oxidation mechanism that can be used for destroying TCE and can achieve more complete mineralization than that of oxidation by persulfate alone.

BET surface areas and iron precipitate contents on the AC are presented in Table 1. Oxidation resulted in decreases in the surface area for persulfate and IAP regenerated AC of approximately 15% and 23%, respectively. This decrease in surface area could be a factor responsible for the decrease in adsorbability. However, the differences in variation of surface area and adsorbability induced between persulfate and IAP oxidation are minor. Furthermore, it is speculated that an additional decrease (i.e., 7%) in surface area by IAP oxidation may be related to iron precipitates. SEM micrographs and EDS elemental analysis of the original and oxidized AC are presented in Figs. 4 and 5, respectively. The existence of iron and an increased Cl weight percentage (see inserted table in Fig. 5) for the IAP oxidized AC sample may also explain the aforementioned iron precipitates (5.5 mg Fe g^{-1}) (see Table 1). Some external differences

Table 1
Surface characteristics of the original and regenerated activated carbons.

Adsorbent	BET surface area (m²/g)	AC iron concentration (mg/g)
AC	930	0
P-AC ($n = 5$)	786	0
IAP-AC ($n = 5$)	719	5.5

Note: P-AC: persulfate oxidized carbon; IAP-AC: iron activated persulfate oxidized carbon; n: number of regeneration cycle. The P-AC and IAP-AC samples were washed using RO water and oven-dried at 105 °C before BET surface area analysis was carried out. For iron content analysis, both oxidized ACs were only oven-dried at 105 °C.
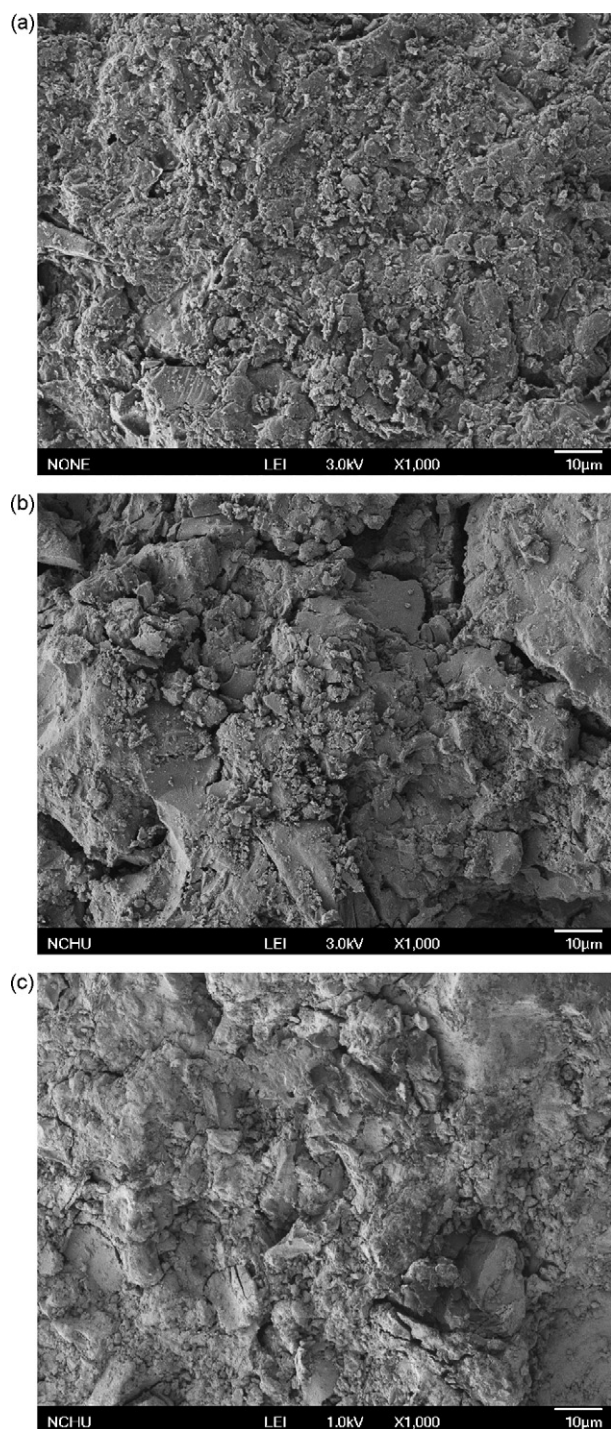
Fig. 4. SEM images of (a) AC; (b) persulfate oxidized AC; and (c) IAP oxidized AC.

Fig. 5. The results of EDS elemental analysis on the surface for (a) AC; (b) persulfate oxidized AC; and (c) IAP oxidized AC.

4. Conclusions

In recent years, persulfate has been used for in situ chemical oxidation remediation of TCE. Persulfate is a relatively stable material that can be activated to form a reactive sulfate radical, which can be used as a strong oxidant. Persulfate has been demonstrated to effectively degrade a variety of organics including TCE. However, the purpose of this study was to use persulfate to regenerate TCE spent AC. The iron activated persulfate process used in this study was seen to oxidize aqueous and sorbed phases of TCE more aggressively than persulfate oxidation alone. Moreover, TCE was not only degraded by IAP oxidation but was also mineralized to form chloride ions. However, both persulfate and IAP oxidation caused a loss in the adsorbability of regenerated AC. Nevertheless, these results indicate that regeneration of TCE spent AC using the IAP process could be an effective method for treating above-ground contaminants. This method could also produce less secondary pollutants than thermal methods. It is known that persulfate oxidation does not result in significant heat or gas generation that could prevent possible volatilization during AC regeneration. Therefore, it might be more applicable for in situ regeneration, e.g., regeneration of AC used in the PRB. However, it needs to be considered that both iron activator and persulfate must be delivered together in contact with the TCE spent AC. In that event, this study could serve as a reference for future application.

in the surface morphology of the samples are apparent. For example, persulfate oxidized AC revealed less small particles and a smoother surface than that of the original sample and this could be linked to the decrease in AC surface area caused by oxidation. When the surface morphology for persulfate oxidized AC was compared to that of IAP oxidized AC, the surface appeared dusty and, to some extent, with a reduced number of pores [20]. Furthermore, many issues remain unresolved such as the variation in surface acidic functional groups, the identities of TCE byproducts, adsorption isotherm behavior before and after oxidative treatment, and the determination of optimum oxidative regeneration conditions.
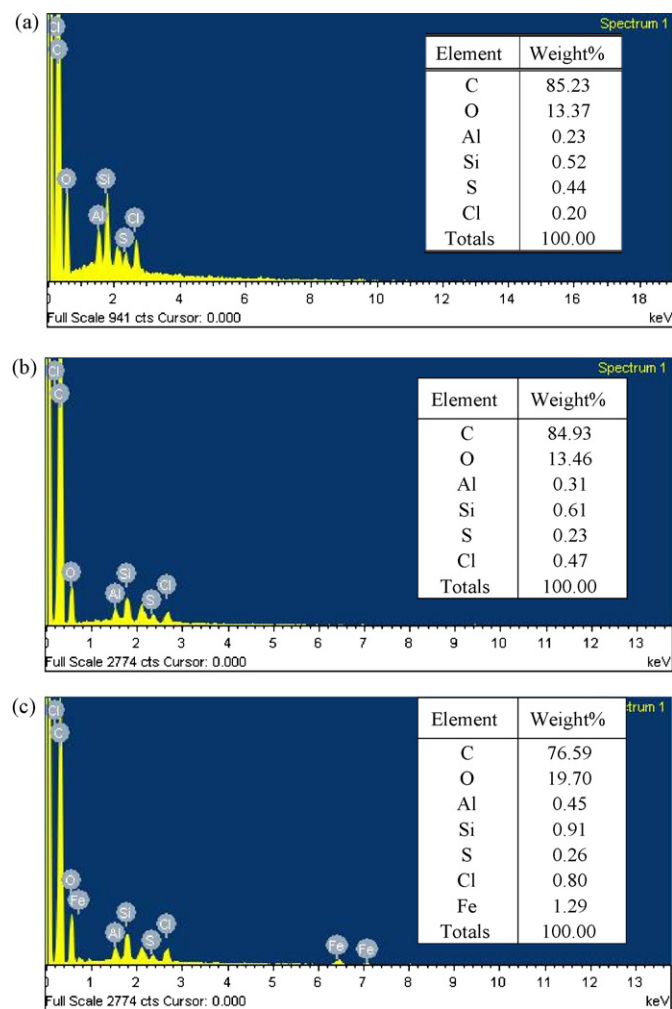
Acknowledgement

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