Removal of TPHs from Soil Media using Persulfate Oxidant in the Presence of Mineral Siderite

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ABSTRACT

The objective of the present study was to evaluate the potential of persulfate (PS) oxidant in the presence of mineral siderite (SI) for remediation of fuel oil contaminated soils. Clay and sandy soils were selected as representative soils which were spiked with 5000 mg fuel oil per kilogram of dry soil. The effects of controlling factors such as different persulfate concentrations (100-500 mM), siderite concentrations (0.1-0.5 g), pH (3-9), and temperature (20-60°C) were also investigated. The results indicated that in clay and sandy soil samples, the highest total petroleum hydrocarbon (TPHs) degradation was observed in the following condition: pH= 3, soils temperature =60°C and PS/Fe (II) molar ratio = 400 mM/0.4 g and 300 mM/0.3 g in clay and sandy soil, respectively. Based on our findings, using persulfate oxidation in the presence of siderite as an activator is a promising technique to remediate soil contaminated by petroleum hydrocarbons.

Keywords: Contaminated soil, persulfate, remediation, siderite, TPHs

INTRODUCTION

Many developing countries around the world are facing problems of soil contamination resulting from human activity (Kaimi *et al.*, 2006). Common causes of soil pollution are the release of petroleum compounds from pipelines and storage tanks (Ferguson *et al.*, 2005). Petroleum hydrocarbon pollution, because of its high sorption capacity, lower mobility, low instability, low solubility and high hydrophobicity, is a major environmental problem (Do *et al*., 2009; Tsai and Kao, 2009). Soil contaminated with petroleum hydrocarbons is a possible pollution source for groundwater used for drinking and irrigation purposes; contamination of agricultural products is a common consequence (Chien, 2012). In order to prevent groundwater pollution, remediation of petroleum contaminated soil should be a vital course of action worldwide.

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There are several methods of remediation for petroleum contaminated soils; some examples include on-site remediation using microwave energy (Chien; 2012); bioremediation (Namkoong *et al*., 2002; Wang *et al.*, 2008; Wang *et al*., 2011); chemical oxidation using hydrogen peroxide (Tsai and Kao, 2009); and persulfate (Huling and Pivetz, 2006). Chemical methods using various oxidants are being developed for the remediation of contaminated soil and water resources. Persulfate is one of the chemical materials used for *in situ* chemical oxidation (ISCO) to remove petroleum compounds (Huling and Pivetz, 2006). Costanza *et al*., (2010) studied the oxidation of tetracholoroethylene (TCE) by thermal activation of sodium persulfate in aqueous solution, while Huang *et al*. (2005) studied the degradation of volatile organic compounds (VOC) with heat-activated persulfate. As a strong oxidant, persulfate ($S_2O_8^2$, E0 = 2.01 V) is popular because of its stability which allows for its transportation over a longer distance in the subsurface. Furthermore, under certain reactions with Fe^{2+}, persulfate can generate sulfate radicals ($SO_4^{\cdot-}$, E^0 = 2.6 V) (see Eq. (1)), which have higher oxidative capacity than common oxidants and are effective in the removal of petroleum compounds in contaminated soil (Fanaroff *et al*., 1994; Huling and Pivetz, 2006; Do *et al.,* 2010). Persulfate activated by Fe^{2+} has been used to degrade various contaminants such as BTEX (Liang *et al*., 2008) in the aqueous phase.

$$S_2O_8^{-2} + Fe^{+2} \rightarrow SO_4^{+2} + SO_4^{-2} + Fe^{+3} \qquad (1)$$
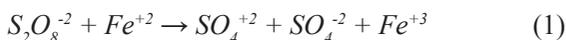
The role of soil minerals on the persulfate activation is proven; however the type of minerals that makes such activation possible is not very clear. Teel *et al*. (2011) surveyed the role of 13 common soil minerals and their effects on persulfate activation; they found that cobaltite, ilmenite, pyrite, and siderite increase persulfate oxidation potential, significantly. Siderite has been used as a catalyst in combination with persulfate and hydrogen peroxide to remove TCE, resulting in 100% removal of TCE in 24 h in aqueous phase (Yan *et al*., 2013). This study aimed to investigate the removal of TPHs from soil media using persulfate oxidant in the presence of mineral siderite as an activator. The TPHs degradation factors of persulfate concentration, siderite dosage, temperature, and pH were investigated in detail in this study.

MATERIALS AND METHODS

Materials

The fuel oil used in this study was purchased from Iran Petroleum Corp. Sodium persulfate ($Na_2S_2O_8$, reagent grade (>99%), Merck Co, Germany) was used as an oxidant. Siderite ($FeCO_3$), purchased from Zamin Tavana Tajhiz Co (Tehran, Iran), was used for ferrous ions supplement to activate persulfate. Contaminated soils were prepared in the laboratory and the soil samples were mechanically homogenised and sieved (10-mesh sieve) to assure uniformity. The soil was spiked with fuel oil dispersed in 1 L of a 1:1 (v/v) n-hexane/acetone solution, and then further homogenised. The solvents were allowed to vaporise from the soils

by placing the container of spiked soil in a fume hood, therefore leaving behind the petroleum hydrocarbon in the soil at a theoretical initial total petroleum hydrocarbon (TPHs) concentration of approximately 5,000 mg kg^{-1} of soil. Properties of both the soil samples used in this study are presented in Table 1. All analyses were performed according to ASTM (1998). The soils and siderite elements were determined by X-ray fluorescence (XRF) (Table 2)

Batch Degradation Kinetic Experiments
Serum bottles (125 mL) were used as reactors for the batch experiments. Each reactor was filled with 2.5 g of the contaminated soil, and various concentrations of persulfate (100, 200, 300, 400, and 500 mM) and siderite (0.1, 0.2, 0.3, 0.4, and 0.5 g) were added to the sample. The experiments were conducted in duplicate. The prepared batch reactors were maintained at different temperatures (20-60°C) until analysis. In order to simulate static subsurface conditions, the reactors were stirred and then placed in the refrigerator. Pseudo-first order reaction model was applied to study the kinetics of diesel removal based on the results reported in previous studies (Huang *et al.*, 2002; Xie *et al.*, 2012)

TABLE 1
Physical and chemical characteristics of the soils used in this study

Parameter	Clay soil	Sandy soil
pH	7.8	8.3
Soil organic matter (%)	2.5	0.116
Electrical Conductivity (dS m^{-1})	2040	229
Total organic carbon (%)	0.82	0.39

TABLE 2
Characteristics of elemental soil and siderite

Sample	Composition	S	P$_2$O$_5$	MnO	TiO$_2$	MgO	K$_2$O	Na$_2$O	CaO	Fe$_2$O$_3$	Al$_2$O$_3$	SiO$_2$	L.O.I
1	Siderite	0.008	0.005	3.021	0.068	0.48	0.03	0.02	0.28	73.16	0.31	13.59	8.76
2	Clay	0.096	0.112	0.124	0.563	5.24	1.87	0.26	13.82	5.86	9.98	43.56	18.26
3	Sand	0.008	0.071	0.087	0.259	3.86	0.89	0.34	24.91	2.6	5.24	38.52	22.73

Analytical Methods

Texas Natural Resource Conservation Commission (TNRCC) Method 1005 was used for quantification of the contaminants; this method is one of the more practical ways of determining hydrocarbons in soil samples using a chromatographic procedure (TNRCC, 2001). Slurry samples in the serum bottle were accurately??? shaken in an incubator (Innova 4048, USA) for about 2 h at 170 rpm to allow the soil to mix well with the persulfate. Prior to commencing the analyses, TPHs were extracted by 10 mL of N-pentane solution. Following this, 0.6µL of the extracted liquid sample was injected carefully into a gas chromatography (CP-3800, Varian: Holland) equipped with a flame ionisation detector and capillary column (CP- SIL 8 CB column model, 30 m x 0.32 mm, 0.25 µm film thickness, VARIAN, Holland). The injector and detector temperatures were maintained at 280°C and 300°C, respectively. The initial temperature of the column was 35°C (held for 8 min) and increased by 9°C/minute to 100°C (held for 8 min).

RESULTS AND DISCUSSION

Effect of Persulfate Concentration on Fuel Oil Removal

In order to determine PS optimum concentration in the range of 100, 200, 300, 400, and 500 mM for TPHs removal, PS was applied with SI fixed at 0.2 g as an activator. When PS was used solely, TPHs degradation rate was not significant at different PS doses (100, 200, 300, 400, and 500 mM), and the maximum removal rate was about 6% (Figure 1). Using SI together with PS led to a higher degradation rate for TPHs which might be due to greater activation of PS because of the presence of SI (Teel *et al*., 2011). Figure 1 shows a clear trend of TPHs degradation rate versus persulfate concentration. It is apparent from Figure 1 that persulfate in concentrations of between 100 and 500 mM led to 8% to 18% and 7% to 20% TPHs removal in sand and clay soil, respectively. As reported by Achugasim et al. (2011), diesel degradation rate using 10% and 20% persulfate concentration were 43% and 55%, respectively. Yen *et al*. (2011) found that TPHs (light crude) removal rate using persulfate activated by Fe(II) for sandy soils was more than 96% under different pH conditions, while Do *et al*. (2009) have reported approximately 35% efficiency for degradation in diesel-contaminated soils. What is interesting in Figure 1 is that the best dosage of persulfate concentration, in the range used, were 400 and 300 mM in clay and sandy soils with efficiency rates of 20% and 18%, respectively. Moreover, the fuel oil degradation rate was not positively related to PS concentration in the range of 100–500 mM, because further increases in PS concentrations, that is, 300 mM for sand and 400 mM for clay soil, resulted in a decrease in fuel oil removal rate. The possible explanation is PS consumption by sulfate radicals which could be more remarkable than the removal of the target (Liang *et al*. 2004). Do *et al*. (2010) suggested that the maximum diesel removal in soil was achieved at PS/Fe (II) ratio of 100:1.
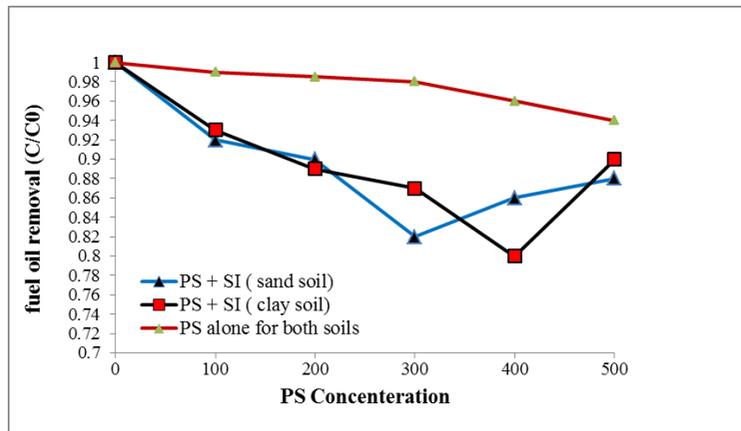
Figure 1: Fuel oil removal efficiency at different PS concentrations plus 0.2g SI for clay and sandy soil at 20±1∘C

Effect of Siderite Dosage on the Persulfate System

Fuel oil removal rates with initial SI concentration varying from 0.1 to 0.5 g SI with a constant PS concentration (400 mM as the best concentration for clay and 300 mM for sand) were studied under laboratory conditions (20 °C), and the results are depicted in Figure 2. The results show that maximum degradation rate for clay soil was achieved at PS/SI ratio of 400 mM/0.4 g, while in the case of sandy soil, the best removal rate was achieved at 300 mM/0.3 g which led to 27% and 24% removal efficiency, respectively. Satapanajaru *et al.* (2014) studied reactive black 5 dye (RB5) removal from water using PS and Fe (II), and concluded that the optimum conditions for 0.01 mM reactive black 5 dye (RB5) treatment were 445 mM PS and 4.91 mM Fe(II). Do *et al*. (2009) used peroxymonosulfate activated with $FeCl_2$ in order to degrade diesel-contaminated soil, and found that the maximum removal rate was 19%. According to Rastogi *et al.* (2009), the optimum condition for 2-chloroethylbiphenyl removal from an aqueous system was achieved when PS/Fe^{2+} mole ratio was 1:1 and the process was conducted at pH= 3. Moreover, lower fuel oil degradation might be explained because of sulfate radical consumption by Fe^{2+} (eq (2)). In these circumstances, the scavenging of the sulfate radicals (SO_4^-•) can be more important than the degradation of the fuel oil. Similar results have been confirmed in other studies (Do *et al*., 2010; Rodriguez *et al*., 2014; Satapanajaru *et al.*, 2014).

$$Fe^{2+} + SO_4^- \rightarrow SO_4^{2-} + Fe^{3+} \qquad (2)$$
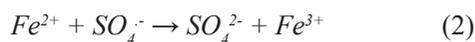
Interestingly, in our study, it was observed that the removal rates were reduced when siderite exceeded 0.4 g in clay and 0.3 g in sand soil, respectively. Hence, in order to minimise the adverse effects of Fe^{2+} on sulfate radical production, the Fe^{2+} concentrations should be controlled. It can be inferred from Figure 2 that in
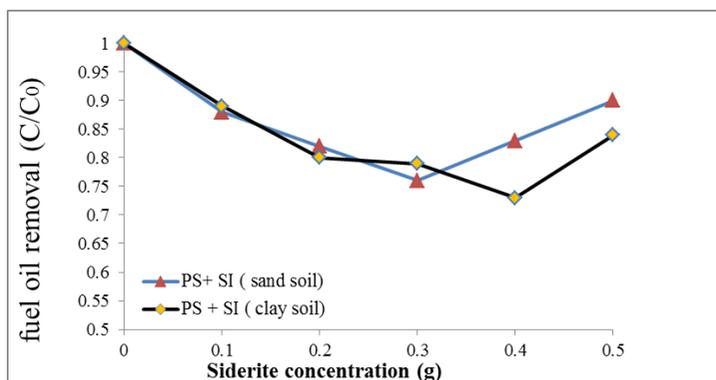
Figure 2: Fuel oil removal efficiency at different SI concentrations the clay soil received 400 mM PS and the sandy soil received 300 mM PS

the persulfate system, the best siderite dose is 0.4 g and 0.3 g for clay and sand soil, respectively. Based on the results, removal efficiency for clay was higher than in sandy soil, which is possibly due to the involvement of metal oxides in the clay soil matrix (mineral such as iron and insoluble organic matter), that have a synergistic effect on the persulfate oxidation potential and persulfate activation (Do *et al.*, 2009; Do *et al*., 2010).

Effect of pH on the Persulfate System
Persulfate is recognised to be highly reactive under both acidic and basic conditions (Block *et al*., 2004). We evaluated the impact of metal oxides on PS combined with Fe (II) presented in siderite, in order to show fuel oil degradation at pH 3, 6 and 9. As expected, the removal rate for PS without siderite was lower than in a system with siderite. Figure 3 demonstrates TPHs removal in the soil sample at different pH levels. It is apparent that in both soils, fuel oil degradation rate was higher with decreasing pH. The finding is similar to other studies (Peyton 1993; Huang *et al.* 2002; Do *et al.*, 2010). However, they suggest that under highly acidic conditions (i.e. pH < 2), persulfate might decompose without generating sulfate radicals, and can result in reduced reactivity with the target contaminants. As demonstrated in Figure 3, maximum removal rate at pH=3 were 36% and 32% in the clay and sandy soil, respectively. Some researchers claim that higher pH values cause the precipitation of Fe^{+2} and result in less sulfate radical production (Rastogi *et al.*, 200; Satapanajaru *et al*. 2014). The investigation of Rastogi *et al.* (2009) showed that degradation of 2-chlorobiphenol in aqueous solutions increased with increasing acidic conditions. Also, Liang *et al*. (2007) reported that maximum TCE removal by persulfate in aqueous solutions occurred at neutral pH, and TCE degradation rate was higher at acidic than basic pH. Sulfate radicals are the main radicals generated in a persulfate system where the pH is between 3 to 7 (House, 1962).

$$S_2O_8^{-2} + H^+ \rightarrow HS_2O_8^- \qquad\qquad (3)$$

$$HS_2O_8^- \rightarrow SO_4^{-2} + SO_4^{\cdot-} + H^+ \qquad\qquad (4)$$
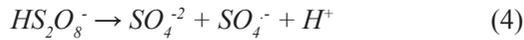
Sulfate radicals can also react with water to produce hydroxyl radicals (eq. 5) (Hayon *et al*., 1972). However this reaction is slow when organic carbon is more than 1 mg L^{-1} (Peyton,1993).

$$SO_4^{\cdot-} + H_2O \rightarrow H^+ + SO_4^{-2} + OH^{\cdot} \qquad\qquad (5)$$

The basic condition (pH > 8.5) is the most widely used activator of persulfate which can produce hydroxyl radicals (OH$^{\cdot}$) according to eq (6).

$$SO_4^{\cdot-} + H_2O \rightarrow HSO_4^- + OH^{\cdot} \qquad\qquad (6)$$
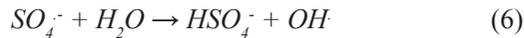
Both hydroxyl radicals (OH$^{\cdot}$) and sulfate radicals (SO$_4^{\cdot-}$) are formed at basic condition (i.e. pH 9). The degradation rate observed in pH=9 might be due to formation of both factors i.e. hydroxyl radicals (OH$^{\cdot}$) and sulfate radicals (SO$_4^{\cdot-}$); however, at this pH value degradation mainly occurs because of generation of sulfate radicals (Dogliotti & Hayon, 1967; Norman *et al.* 1970; Liang & Su, 2009). The lower degradation of contaminants was expected because SO$_4^{\cdot-}$ and OH$^{\cdot}$ radicals degenerate rapidly because of the reaction with hydroxyl ions (Huang *et al.*, 2002).
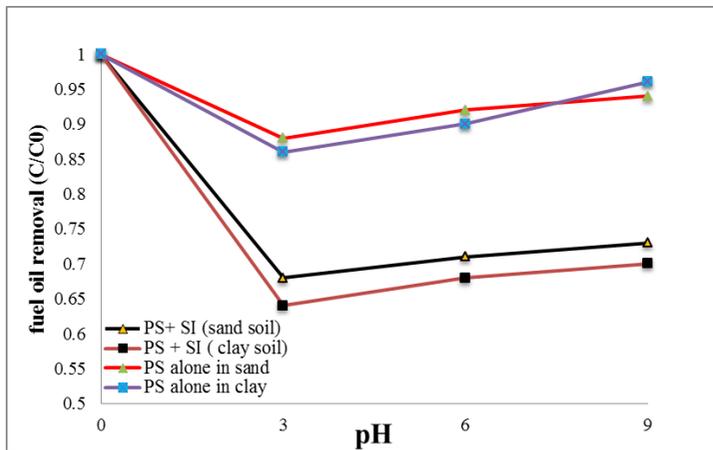

Figure 3: Fuel oil removal rate at different pH for sandy and clay soils with different treatments

Effect of Temperature on the Persulfate System

High temperature is another important factor which determines the efficiency of persulfate activation (Liang & Su 2009). According to Yukselen-Aksoy *et al*. (2010), persulfate activation was at pH >12 when the PCBs degradation was examined in soil. Although, some activation occurred at temperatures as low as 20°C, most heat activated persulfate in situ chemical oxidation (ISCO) systems utilise temperatures higher than 40°C. Thermal activation changes with temperature and is one of the ways to control the generation of sulfate free radicals as defined in eq. 8 (House 1962; Zhang *et al.*, 2010)

$$S_2O_8^{-2} + heat\ or\ UV \rightarrow 2SO_4^{-} \qquad (8)$$

In this study, temperature action was studied between 20°C and 60°C. Removal rate of fuel oil at different temperatures is shown in Figure 4. Temperatures more than 50°C were considered because Johnson *et al*. (2008) reported that the relatively short lifetime of the persulfate at elevated temperatures (e.g., > 50°C) will limit delivery time in contaminated soils. Nevertheless, as can be seen in Figure 4, the degradation rate at 60°C is only slightly higher than at other temperatures (i.e. 20 and 40°C), which indicates that the chemical activation rate increases with increasing temperature. Hence, it is apparent from Figure 4, that fuel oil is rapidly degraded with persulfate under experimental conditions. For example, at 60°C and pH=3, fuel oil removal efficiency for clay soil was about 41%, and at 20°C, removal was 32%. Our findings indicated that PS=500 without SI at pH=3 and at 60°C have nearly 17 and 20 % removal rates for clay and sandy soil, respectively.
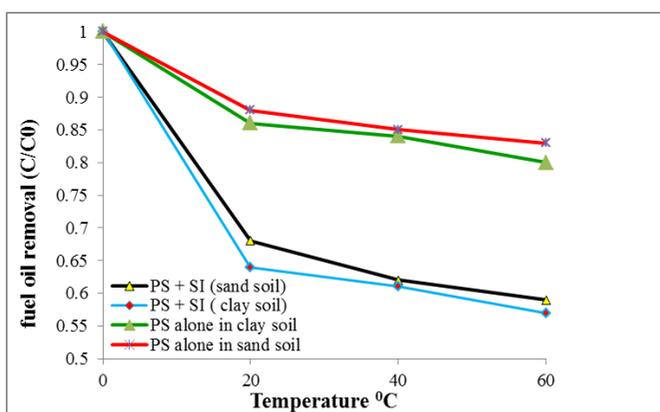

Figure 4: Fuel oil removal rate at different temperature, for sandy and clay soils with different treatments.

CONCLUSION

The highest TPHs degradation in each system containing either clay or sand soil was observed under the following conditions: pH =3 temperature, 60°C, PS/Fe (II) molar ratio = 400 mM/0.4 g, and 300 mM/0.3 g for clay and sandy soil, respectively. The effect of siderite mineral indicated that this metal oxide can activate PS. Moreover, pH effects on clay were more remarkable than in sandy soil and maximum persulfate concentration needed for clay was higher than for sandy soil. Results of this study will be helpful to determine the optimum amount of persulfate and siderite needed for the remediation of soil at petroleum hydrocarbon contaminated sites.

ACKNOWLEDGEMENTS:

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