

A LABORATORY STUDY ON THE DEGRADATION OF GASOLINE CONTAMINATION USING FENTON'S REAGENT

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ABSTRACT

In situ chemical oxidation is currently being developed as a technique for the remediation of hydrocarbon contaminated soils. The end products of this chemical oxidation are usually carbon dioxide and water. In particular, Fenton's reagent has been used as an oxidizing agent since it can produce strongly oxidizing hydroxyl radicals from injected hydrogen peroxide solution with iron as a catalyst.

The successful application of Fenton's reagent to gasoline contaminated soils *in situ* is affected by several factors such as the concentration of hydrogen peroxide, the concentration of the iron catalyst, the level of hydrocarbon concentration, the pH of the reaction environment, soil composition, and the expected thermodynamics of the reaction. This process may be further modified by the rate of chemical application and flow characteristics of the aquifer.

In this study, the effective oxidation of unleaded gasoline by Fenton's reagent under various concentrations of iron, hydrogen peroxide and buffering solution was studied using both aqueous solutions and sand columns for reactions up to 2 hours in duration. Results indicate that (i) pH buffering may not be necessary, (ii) 17.5% hydrogen peroxide and 200 mg/L iron solutions provide the best range for hydrocarbon reduction, (iii) temperatures higher than 100 °C can be produced when 35% peroxide solutions are used.

RÉSUMÉ

Une technique d'oxydation chimique *in situ* est en développement pour remédier à la contamination des sols par les hydrocarbures. Les substances résiduelles de ce processus chimique d'oxydation sont habituellement du dioxyde de carbone et de l'eau. En particulier, l'agent de réaction de Fenton a été utilisé comme agent d'oxydation parce qu'il produit des radicaux hydroxyle fortement oxydés à partir de la solution de peroxyde d'hydrogène en utilisant le fer comme catalyseur.

Le succès de l'application de l'agent de réaction de Fenton *in situ* dans les sols contaminés par la gasoline est affecté par plusieurs facteurs tels la concentration du peroxyde d'hydrogène, la concentration de fer catalyseur, le niveau de concentration d'hydrocarbure, le pH de l'environnement immédiat de la réaction, la composition du sol, et la thermodynamique de la réaction. Ce processus peut de plus être affecté par le taux d'application chimique et le débit caractéristique de l'aquifer.

Dans cette étude, l'efficacité de l'oxydation de gasoline sans plomb par l'agent de réaction de Fenton sous différentes concentrations de fer, de peroxyde d'hydrogène et de solution tampon a été étudiée en utilisant des solutions aqueuses d'une part, et des colonnes de sable d'autre part pour des périodes allant jusqu'à 2 heures. Les résultats indiquent que (i) le pH tampon n'est peut-être pas nécessaire, (ii) des solutions de 17.5% de peroxyde d'hydrogène et de 200 mg/L de fer donnent les meilleures gammes pour la réduction des hydrocarbures, (iii) des températures supérieures à 100°C peuvent être produites quand des solutions de 35% de peroxyde sont utilisées.

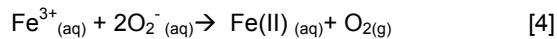
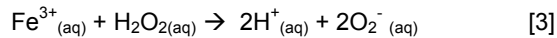
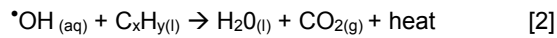
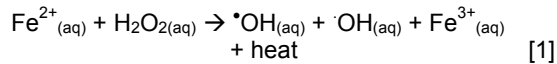
1. INTRODUCTION

In situ chemical oxidation is currently being developed as a technique for the remediation of hydrocarbon contaminated soils. The end products of this chemical oxidation are usually carbon dioxide and water. For the past few decades, Fenton's reagent has been established as an effective oxidizing agent for treating industrial wastewater. However, it is only in the past ten years that it has been considered as a viable option for the *in situ* remediation of hydrocarbon-contaminated soils (Bryant and Wilson, 1999).

Fenton's reagent is made up of hydrogen peroxide (H₂O₂) and an iron (II) catalyst. It oxidizes hydrocarbons by forming hydroxyl radicals (*OH) derived from H₂O₂ in the presence of the iron (II) catalyst (Huling *et al*, 2000). Hydroxyl radicals are powerful oxidizing species, which non-selectively react with organic substrates via bond addition and hydrogen abstraction under suitable conditions (Nyer and Vance, 1999; Bryant and Wilson, 1999). The reaction is exothermic and leads to the complete mineralization of organic substrates as illustrated by Equations [1] and [2]. The iron (II) catalyst



is then regenerated by the reaction of iron (III) with peroxide according to Equations [3] and [4].



The terms (aq), (l), and (g) denote aqueous, liquid and gaseous phase respectively. As described in Figure 1, the degradation of organic contaminant species takes place within a larger framework of reactions that involve H_2O_2 and allows for the regeneration of ferrous ions, Fe(II), from ferric ions, Fe(III). Consequently, for successful applications of Fenton's reagent, sufficient peroxide must be supplied to serve not only as a source of hydroxyl radicals, but also as a reactant to satisfy catalyst renewal and other competing reactions, such as those with manganese or enzymes from soil microbes (Huling *et al.*, 2000).

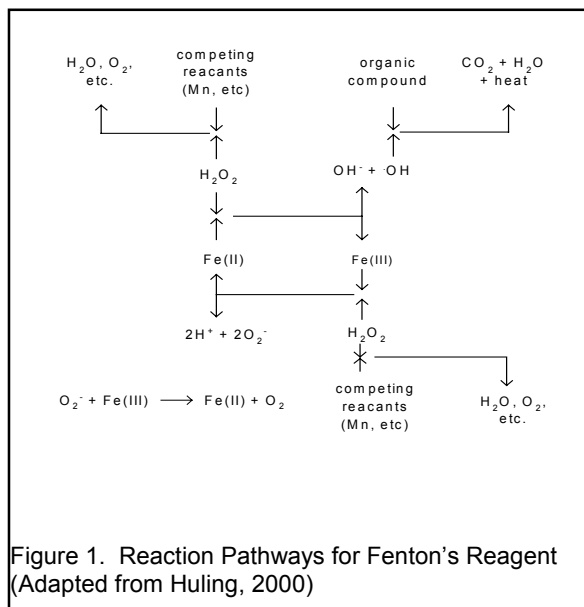


Figure 1. Reaction Pathways for Fenton's Reagent (Adapted from Huling, 2000)

In this paper, the oxidation of organic compounds brought about by Fenton's reagent is generically termed Fenton's reaction. In the following sections, factors affecting the rate of Fenton's reaction are identified. Some of these factors are then studied using laboratory experiments in order to establish guidelines for the successful application of Fenton's reagent for the *in situ* remediation of hydrocarbon-contaminated soils.

2. FACTORS AFFECTING FENTON'S REACTION

From the literature cited previously, key factors affecting the effectiveness of Fenton's reagent as an oxidizing agent are summarized in the following.

2.1 Hydrogen Ion Concentration (pH)

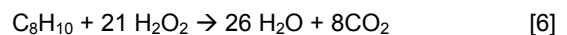
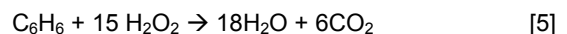
The formation of hydroxyl radicals from Fenton's reagent is known to occur most favourably in neutral to acidic environments. In alkaline environments, the decomposition of peroxide to oxygen and water predominates thus reducing the effectiveness of the reagent. A strong acid (e.g. sulphuric) is often used to ensure initiation of the reaction, while occasionally a strong base has also been used to maintain a pH level for the reaction to be efficient (Watts *et al.*, 1994). During a Fenton's reaction, organic acid intermediates created by the degradation of organic material can cause a drop in pH. In this case, the use of a true chemical buffering system (i.e. weak acid/acid salt mixture) may be beneficial in providing process control.

2.2 Hydrogen Peroxide Requirements

The quantity of hydrogen peroxide required for a Fenton's reaction is influenced by:

- the mass and concentration of target contaminants.
- the amount and concentration of available Fe(II),
- the rate of catalyst regeneration,
- the organic matter content in the soil and groundwater, and,
- the amount of available interfering agents (e.g. manganese and copper) or microbial enzymes present

Using diesel fuel, Chen *et al.* (1998) found that doubling the amount of peroxide required, as calculated from stoichiometric ratios of an empirical equation, was sufficient in natural soils with significant total organic carbon (TOC) levels (1800 to 2400 mg/kg) and total iron content (7900 to 12000 mg/kg). The calculation used pentane as a species representative of the diesel fuel components. Likewise, using benzene and xylene as representative of gasoline mixtures, H₂O₂:benzene and H₂O₂:xylene molar ratios of 15:1 and 21:1 are obtained from the following equations.



From these molar ratios, the minimum ratio (by weight) of H₂O₂:gasoline required for gasoline degradation is estimated to be around 6-7:1. Other peroxide to gasoline ratios (wt/wt) reported include 1-5:1 for water treatment, 5-50:1 for soil treatment (US Peroxide, 1999), and 12:1 (Lou and Lee, 1995).

2.3 Iron Requirement

Iron (Fe) required for Fenton's reaction can be provided by adding aqueous Fe(III) or Fe(II) solutions, or by capitalizing Fe contents already existing in soil minerals and groundwater. Watts and Dilly (1996) reported that Fe(III) species derived from iron perchlorate and iron nitrate provided higher diesel fuel reduction than Fe(II) catalysts (90% vs. 75%) because Fe(II) catalysts tended to consume peroxide too quickly, resulting in less

peroxide available for catalyst regeneration. Also noteworthy was the extent to which the anionic species of the compound “quenched” hydroxyl radicals. They also found that limited desorption of diesel from soil particles occurred, indicating that Fenton’s reaction was carried out on the surface of particles. In addition to being more economical, utilizing *in situ* iron minerals can be advantageous especially in regions where iron solution injection is not desirable for logistical, economical or regulatory reasons.

2.4 Temperature

Fenton’s reaction is exothermic and is most effective at temperatures between 10 °C and 20 °C (US Peroxide, 1999). Typical of exothermic reactions, its reaction rate will reduce with higher temperatures.

2.5 Gas Production

Fenton’s reaction can produce significant amounts of gaseous by-products (CO₂, O₂ and H₂O). In addition, the heat and gas produced may volatilize some hydrocarbon contaminants. The gases generated may result in dangerous pressure build-up under floor slabs (Nyer and Vance, 1999) or inside injection wells. Consequently, control of gas and temperature produced in a Fenton’s reaction are not only significant from a process efficiency perspective, but also from one of health and safety.

3. LABORATORY TESTING PROGRAM

In order to gain insight into Fenton’s reaction, a laboratory testing program was carried out to:

- profile the heat and gas production during reactions in an aqueous environment,
- evaluate the effective destruction of gasoline solution over a range of Fe:H₂O₂ ratios with and without pH buffering, and,
- establish the amount of H₂O₂ required to treat a gasoline-contaminated sand column.

3.1 Temperature and pH Profiles

250mL solutions of soluble ferric nitrate nonahydrate (Fe(NO₃)₃•9H₂O) were mixed in a 500 mL flask with 3.5%, 17.5% and 35% H₂O₂ solutions respectively. Temperature and pH were measured periodically over 60 minutes. Experimentation with acid buffering was also performed, adding 15mL glacial acetic acid (pKa = 3.75) to the iron/water gasoline solution, followed by the addition of solid potassium acetate until a pH of 3.75 was achieved. A trial using 3.5% H₂O₂ and 500 mg/L Fe was also conducted. This trial solution was spiked with 10 mL regular unleaded gasoline.

3.2 Fenton’s Treatment to Aqueous Gasoline Solution

400 mL of an iron solution ranging from 0 to 1000 mg/L in concentration were spiked with 0.35 mL of regular unleaded gasoline. The reaction vessel was then capped and inverted 20 times to ensure adequate mixing. Hydrogen peroxide was added in H₂O₂:gasoline weight

ratios of 5:1, 10:1, 20:1 and 50:1 respectively. 25mL aliquot samples were then pipetted from the reaction vessels into 40 mL amber sample vials prepared with 5 mL of 98% H₂SO₄ to quench the Fenton’s reaction (Watts *et al*, 1994). Vessels were uncapped during the reaction time.

Sample vials were agitated for 2 minutes and allowed to equilibrate for 15 minutes prior to analysis using a HNU Model 311 Portable GC-PID (gas chromatograph photoionization detector). Tests were run in triplicate to determine the average percent of gasoline reduced over the time period. The percentage reduction is given by:

$$\% \text{ Reduction} = [(1 - (A_t/A_o))] \times 100\% \quad [5]$$

where A_o is the total chromatogram peak area at time = 0 and A_t was the total chromatogram peak area at time t, allowing for relative comparison between trials. Trials were compared against non-treated controls (i.e. no H₂O₂ added), whose % reduction was considered to represent evaporative loss to the atmosphere during an experiment.

3.3 Fenton’s Treatment to Sand Columns

Sand columns were prepared using Sil4 Silica Sand inside a cylindrical clear PVC cell measuring 49 mm in diameter and 200 mm in length equipped with stainless steel end-caps. At each end cap there were two ¼” valved openings covered with GeoSock™ to avoid loss of sand through the fittings. Sand was compacted to have

an average dry bulk density of approximately 1650 kg/m³. Its hydraulic conductivity was in the order of 10⁻³ m/s.

Each sand column was first weighed. Then Gasoline was introduced into the sample to about 50 mm from the base of the column through the bottom port (see Figure 2). At the same time, a small suction was applied to the column through the top valve using a hand pump. To



Figure 2. Adding Gasoline to a Sand Column

promote further mixing, the column was inverted twice at 30-minute intervals. After letting the column stand for another 10 minutes, the column was weighed to obtain the mass of gasoline added. To determine the initial contaminant concentration, a series of contaminated columns were sampled, weighed, and analyzed to produce a standard contamination curve. Sampling occurred by opening each end of the column and pushing

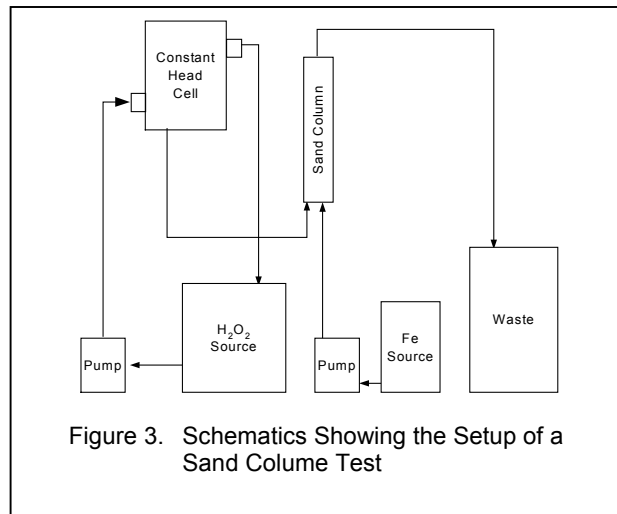


Figure 3. Schematics Showing the Setup of a Sand Column Test

the sand into 2x125mL glass sampling jars. Samples were kept cool and transported to ETL Laboratories in Calgary for BTEX, TVH and TEH analysis.

The treatment of a contaminated soil column using Fenton's reagent was achieved by passing peroxide solution upwards through a vertical column using a constant head flow system shown schematically in Figure 3. Ferric nitrate nonahydrate solution was also introduced upwards through the column by feeding concentrated an aqueous solution into the peroxide stream just before the bottom port. The flow rate of iron solution into the column was controlled by a variable speed peristaltic pump.

Fenton's treatment was continued until a desired mass of hydrogen peroxide had been introduced into the column based on the initial concentration of the reagents and the flow rate through the column.

Iron solutions introduced had stock concentrations of 0, 250, 1000 and 2500 mg/L total Fe at a 1:5 volume ratio with peroxide solutions such that final Fe concentrations within the cell were 0, 50, 200 and 500 mg/L respectively. Peroxide treatment was continued until ratios (wt/wt) of H₂O₂:gasoline content in the column were 5:1, 10:1 and 20:1. This was achieved by applying approximately 600 mL of 3.5%, 17.5% and 35% peroxide solutions respectively. To account for losses due to soil column flushing and sorption onto GeoSock™ materials, trial blanks using only water were conducted so that relative comparisons could be performed.

Due to the potential for pressure and temperature build-up within the closed sand columns, experiments were conducted in a 762 mm wide x 914 mm deep drum lined with washed sand. This precaution provided for greater

containment of any spills and protection of personnel should pressure or temperature build-up result in column failure.

4. TEST RESULTS

4.1 Temperature and pH Profiles

Iron solutions were observed to be orange and acidic. The pH of 50, 200 and 500 mg/L solutions of Fe(NO₃)₃·9H₂O were 2.9, 2.5, and 2.2 respectively. H₂O₂ solutions were observed to be clear and less acidic than iron solutions. Solutions of 3.5%, 17.5% and 35% H₂O₂ were found to have pH levels of 7.8, 6.2 and 4.0 respectively.

When the reactants were first mixed, the solution became dark amber to black in colour. This colour change became more intense as the concentration of H₂O₂ and Fe were increased. With time, the mixture returned to its initial orange colour.

As shown in Figure 4, temperature was observed to peak at about 100 °C in trials with H₂O₂ concentration higher than 3.5% and Fe concentration higher than 50 mg/L. The peak was reached earlier as the concentrations of the reagents were increased. All 50mg/L Fe trials were observed to remain at ambient temperatures for all concentrations of peroxide. Bubbling was observed in all trials and a rapid boil was observed for tests using higher reagent concentrations. For trials using 500mg/L Fe and H₂O₂ concentrations greater than 17.5%, solutions were observed to boil over the walls of the containing flask. Generally, the amount of gas production was observed to have a positive correlation with temperature.

Figure 5 shows the variations of pH when aqueous solutions of Fenton's reagents were mixed. Initially, the pH was acidic, then it increased slightly before it decreased to a minimum. The pH minima occurred sooner and at lower levels as the concentration of reagents was increased.

The additional trial using 500 mg/L Fe and 3.5% H₂O₂ spiked with 10 mL of unleaded regular gasoline was also conducted. Compared to the non-spiked trials, slightly higher temperatures and slightly lower pH's were recorded.

4.2 Fenton's Treatment to Aqueous Gasoline Solution

When Fenton's reagents were applied to aqueous solutions of regular unleaded gasoline at about 0.08% concentration, only minimal gas production and temperature change were observed. Colour changes associated with the reaction are similar to those reported in the previous section.

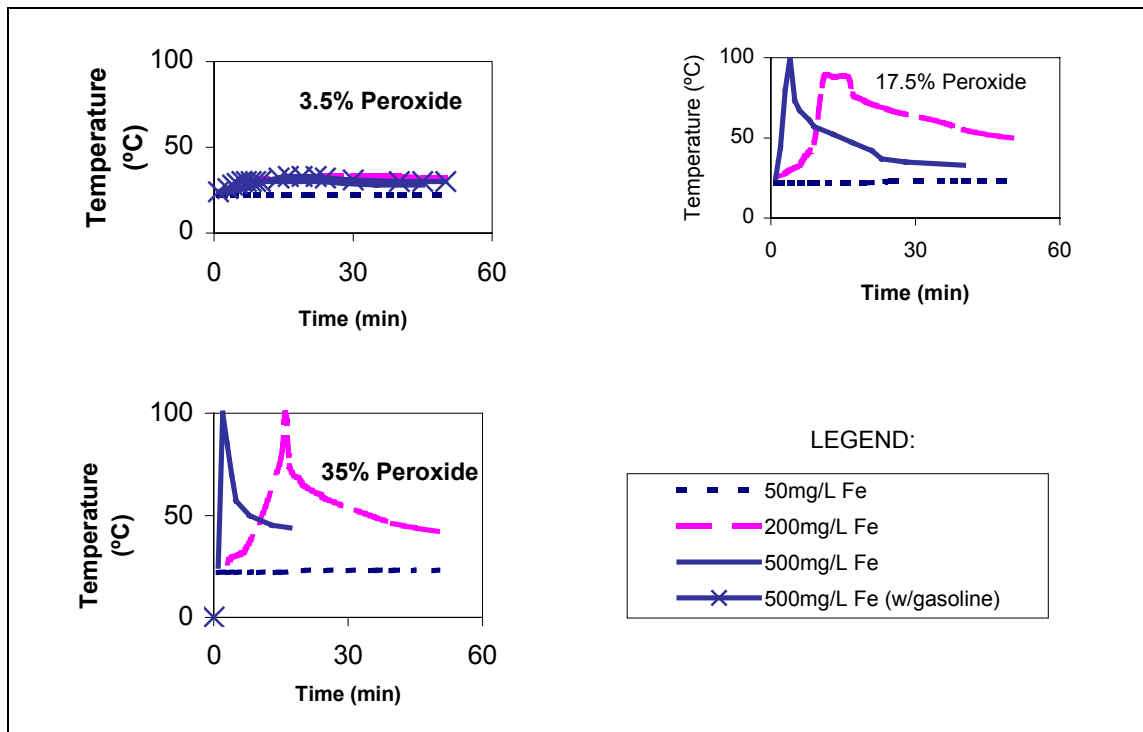


Figure 4. Temperature Variations in Aqueous Solutions of Fenton's Reagent

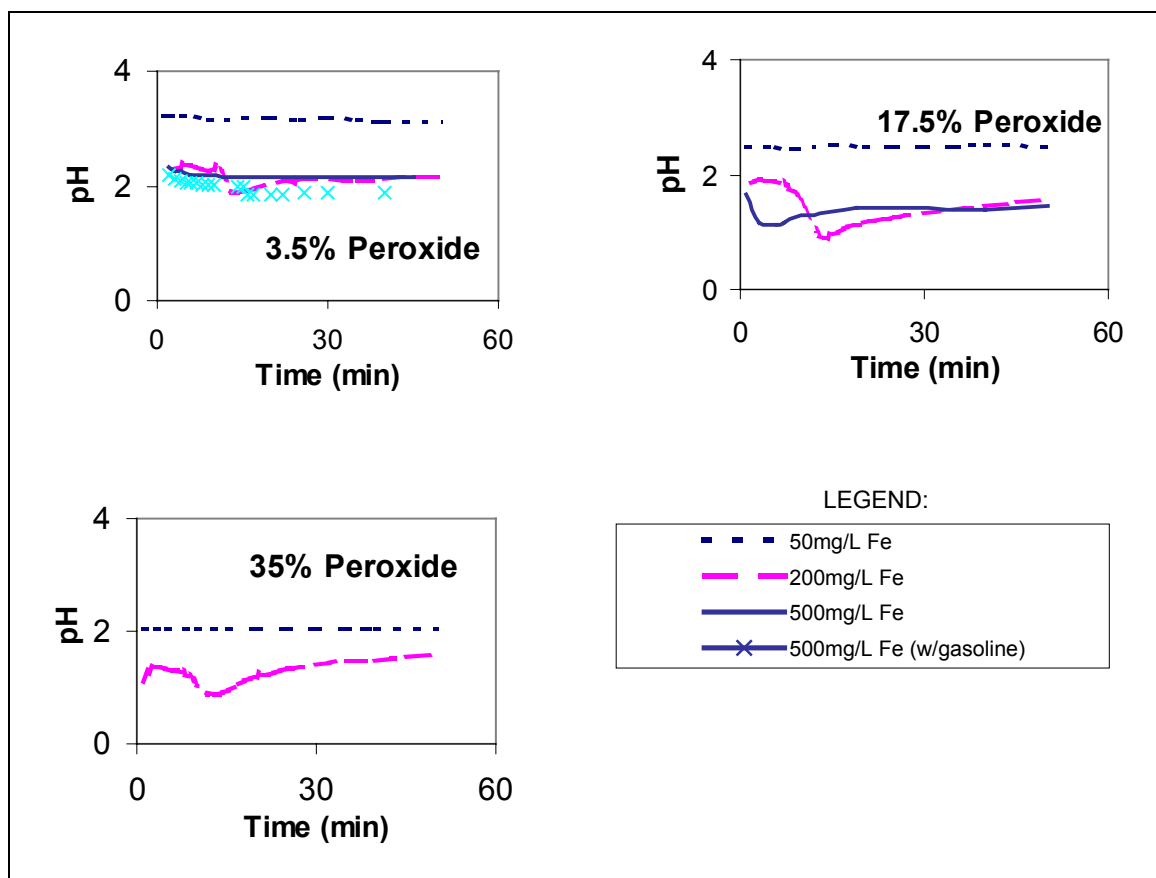


Figure 5. pH Variations in Aqueous Solutions of Fenton's Reagent

Gexhibit high variation and large standard errors. Despite of this, it was generally found that hydrocarbon contamination destruction was significantly greater for trials using 200 mg/L Fe catalyst. When [Fe] was higher than 50 mg/L, $[H_2O_2]$ appeared to have little effect on the test results.

4.3 Fenton's Treatment to Soil Columns

Temperature increases and gas production (frothing) were observed in the column experiments with higher reagent concentrations. In one case ($[Fe] = 200\text{mg/L}$, $[H_2O_2] = 35\%$), the trial had to be stopped periodically for the column to cool down when temperatures over 65°C were reached. Due to the potential violent nature of the reaction when higher concentrations of peroxide and Fe were used, upper limits were set for $[H_2O_2]$ and $[Fe]$ at 17.5% and 500 mg/L respectively. Effluent from the column was observed to be dark amber to black in colour at higher concentrations. Although gases were produced in all tests, gas production was more intense when higher reagent concentrations were used.

Chemical analysis for total petroleum hydrocarbons (TPH) in the post-treatment sand indicated that soil column flushing, by either water or peroxide without the addition of iron, accounted for 90% ($\pm 2\%$) of gasoline level reduction alone. As shown in Figure 9, trials using 200mg/L Fe with 17.5% H_2O_2 as well as 50mg/L Fe with 35% H_2O_2 were the most effective treatments, reducing gasoline levels further to approximately 1% of their original mass respectively. The former was observed to yield temperatures upwards of 60°C , being stopped in order to allow for column cooling. In contrast the latter trial yielded temperatures upwards only to 40°C . Trials using lower concentrations of the reagents were observed to be no different or less effective in reducing soil TPH

than soil washing alone. Additionally, these trials were found to have little to no temperature change.

5. DISCUSSION

5.1 Temperature and pH Characteristics

Temperature, pH, and colour appeared to follow some characteristic trend as Fenton's reagents were mixed. Initially intense colour change occurred and progressed to a lighter colour. It can be inferred that colour change, can be used as a qualitative indicator to determine the progression and intensity of the reaction.

The sharp increase in speed and intensity of temperature peaks with respect to varying reagent concentration and therefore, colour change, indicates that the formation of hydroxyl radicals and their consequent reactions produce a considerable amount of heat and gas. The violent nature of this reaction provides affirmation that concerns regarding control of the reaction in confined spaces are warranted, and suggests that combinations of peroxide over 17.5% with iron solutions greater than 200mg/L in concentration be approached with caution.

The minimal additional increase in temperature observed in the gasoline spiked trial suggests that heat from the Fenton's process can be attributed largely to hydroxyl radical formation. Heat produced from gasoline oxidation may become a factor when significant volumes of gasoline are present. Further experimentation with other natural and artificial hydrocarbon sources would likely be beneficial for application purposes.

The acidic nature of both peroxide and $Fe(NO_3)_3 \cdot 9H_2O$ solutions provides an acidic environment for the duration of reaction, thus it ensures that the Fenton's reaction was

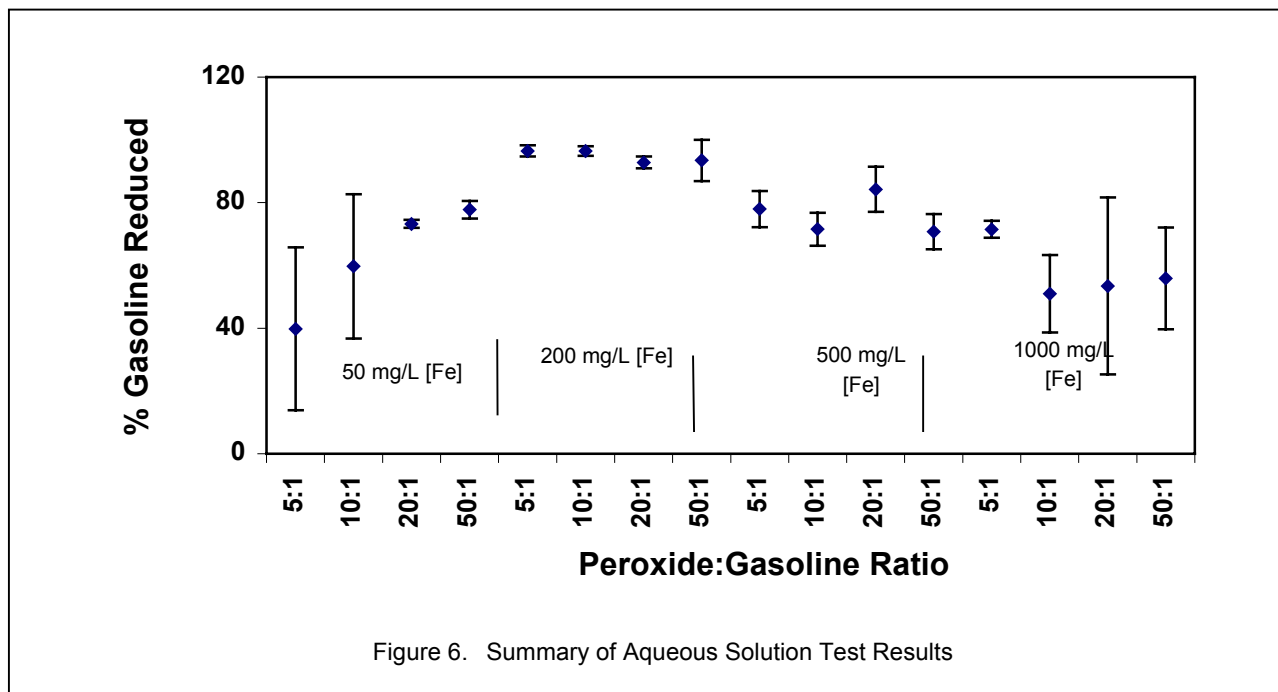
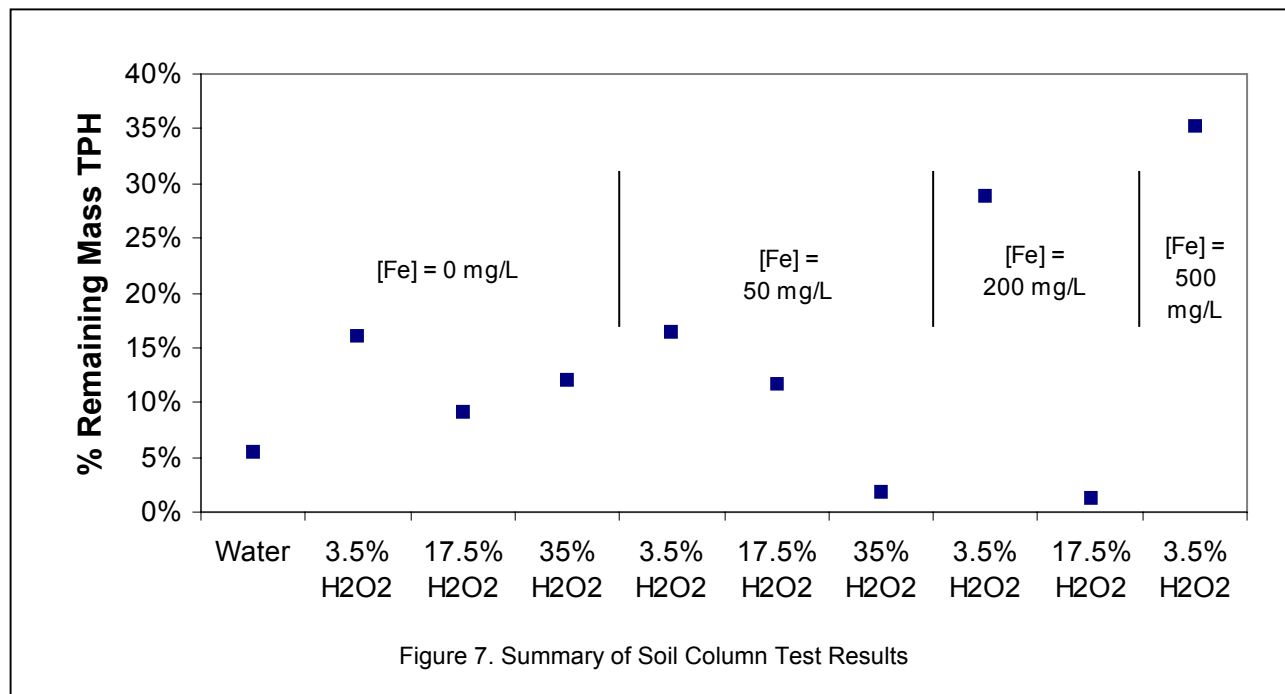


Figure 6. Summary of Aqueous Solution Test Results

favoured over other peroxide dissociation mechanisms. This observation suggests that pH buffering to ensure acidic conditions may not be necessary for certain iron amendment levels and types. The extreme pH drop below 2 observed in some trials indicates that occasional addition of alkaline buffers may still be considered to enhance the reaction.

contaminant reduction, as long as the level of iron is appropriately optimized.

Experiments using acetic acid buffer solutions proved to produce greater total peak areas and less overall hydrocarbon destruction. The observation that peak areas were biased by a large peak eluting at approximately the same time as acetic acid sample peaks



5.2 Fenton's Treatment to Aqueous Gasoline Solution

The significantly higher destruction of hydrocarbons by solutions using 200 mg/L iron catalyst suggests that higher levels of iron may result in peroxide being consumed by the reaction too quickly, reducing the exposure time for hydrocarbons to hydroxyl radicals to for them to be oxidized. In contrast, the lower destruction of hydrocarbons observed at 50 mg/L iron may indicate that too much peroxide is required for catalyst regeneration (Watts and Dilly, 1996). This conclusion is further supported by the increase in destruction of gasoline at 50 mg/L Fe with an increase in peroxide concentration, which suggests that higher peroxide: gasoline ratios at lower iron levels permit a greater production of hydroxyl radicals for gasoline oxidation. Further optimization of iron catalyst type and concentration must be explored together with site-specific factors (i.e. mineral iron content), cost (i.e. $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ = ~\$48/kg) and the effect of selected anionic constituents (i.e. nitrate, sulphate or acetate).

Despite the trends observed in iron optimization, it was found that little difference in gasoline destruction existed in the optimal iron range, despite changes in peroxide concentration. Therefore, one may conclude that H_2O_2 :gasoline ratios of 5:1 or greater are sufficient for

suggests that the acetate buffer complicated the evaluation of the reaction. As acetic acid is an organic acid (CH_3COOH), it participates in Fenton's reaction as a competing species. As prior pH characterizations found that acidic environments are produced by the iron catalyst, one may conclude that acid buffering with acetic acid only further complicates an already complex process, and lead to increased costs for questionable savings. Consequently, buffering using an organic acid appears at this time ineffective.

5.3 Fenton's Treatment to Soil Columns

Trials using peroxide solutions alone (no Fe) yielded results similar to those using column flushing using water. This indicates that hydrogen peroxide without iron is ineffective as a chemical oxidizing agent for reaction times up to 2 hours.

A greater level of hydrocarbon destruction was achieved by using the following peroxide and iron concentrations: 35% H_2O_2 and 50 mg/L Fe, and 17.5% H_2O_2 and 500 mg/L Fe. It should be noted that this reduction may not be due to chemical oxidation alone, but may also include losses due to increased volatilization, and increased desorption from soil in response to higher temperatures.

6. CONCLUSION

In summary, this study aimed to provide a preliminary characterization of Fenton's reagent for the purpose of in-situ chemical oxidation of soil contaminated with petroleum hydrocarbons. In agreement with previous studies, Fenton's reagent was characterized as being a highly reactive process, which may produce temperatures well above 100 °C and significant amount of gas very rapidly. Consequently, the application of Fenton's reagents must be approached carefully with safety at the forefront.

Results from aqueous trials suggest that H₂O₂:gasoline (wt/wt) ratios of 5:1 are sufficient for near complete oxidation of gasoline so long as iron levels are optimized. Iron optimization appears to occur around 200 mg/L Fe when using Fe(NO₃)₃·9H₂O as a source catalyst, but will likely vary as other factors such as iron source type are explored. Preliminary results indicated that acetate or organic salts might not be an appropriate choice.

Experimental trials using gasoline contaminated sand columns indicated that removal of petroleum hydrocarbons is generally favoured for the following combinations: 35% peroxide and 50 mg/L iron; and, 17.5% peroxide and 200 mg/L iron. It was observed that temperature produced with combinations of iron over 200mg/L paired with peroxide levels greater than 17.5% in lab experiments were i.) outside the optimal Fenton's reagent temperature range and ii.) potentially unsafe because of extreme temperature increases and gas production. Further refinement of process factors such as injection rate and iron type at peroxide and iron concentrations around 17.5% and 200mg/L would likely provide for better optimization.

7. ACKNOWLEDGEMENT

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