

Mineralization of organic compounds in wastewater contaminated with petroleum hydrocarbon using Fenton's reagent: a kinetic study

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Abstract: In the present work, the possibility of using chemical oxidation through Fenton's reaction for the pre-treatment of wastewater contaminated with petroleum hydrocarbon was investigated as laboratory-scale experiments. The effect of different operational conditions, namely, hydrogen peroxide, ferrous ion concentrations, temperature, and initial pH were evaluated. Operating at initial pH of 3, with a temperature of 28 °C and a molar ratio $H_2O_2/Fe^{2+} = 9:1$, it gave us 70.58% removal of COD. A kinetic study was carried out using a modified pseudo-first-order model. The experiment was performed at different temperatures hence allowed the calculation of Arrhenius equation parameters and the global activation energy for the first-order reaction. [Journal of American Science 2010;6(4):58-66]. (ISSN: 1545-1003).

Key words: Chemical oxidation, wastewater, COD, activation energy

1-Introduction

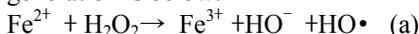
The world is facing the challenge of purification of surface and groundwater contaminated with petroleum hydrocarbon. In spite of its many uses, petroleum hydrocarbons are toxic in nature and environmental contamination by these toxic products is emerging as a serious global problem. Hydrocarbons are quantitatively most important constituents of petroleum, and arise from natural as well as anthropogenic sources. Their accidental releases in the natural environment have resulted into the gross contamination of soil, surface and groundwater. In this case, they constitute a major environmental problem due to the high organic compounds load and the great volume of contaminated water generated. In the last few years there has been a great effort to develop new solutions for the remediation of hydrocarbon from water. The best and economical way is to control the release of petroleum in the natural environment.

Biological treatments have been suggested by many authors and institutions. One of those institutions is RIMAX BIO which suggested the use of BIO PETRO TREET – Bioremediation system for removing petroleum hydrocarbon from soil and water. The Bio Petro Treet system consist of a highly concentrated microbial formulation

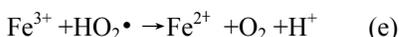
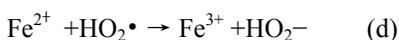
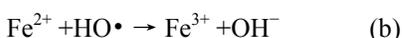
containing a proprietary blend aerobic bacteria having petroleum molecule metabolizing capacities for hydrocarbon removal from soil and aqueous environments. Although, the high concentration of organic compounds load make the petroleum hydrocarbon recalcitrant to aerobic degradation and inhibit the efficiency of anaerobic digestion. Physico-chemical processes have been also applied for petroleum hydrocarbon treatment. The reduction of hydrocarbon pollution can be increased if biological treatments are combined with chemical or physical processes. Advanced oxidation processes (AOPs) are known for their capacity to mineralize a wide range of organic compounds. The concept has been defined by Glaze et al. in 1987 as “near ambient temperature and pressure water treatment processes which involve the generation of hydroxyl radicals in sufficient quantity to effect water purification”.

AOPs employ reactive oxidizing agents such as hydrogen peroxide or ozone, with or without addition of catalysts or photolysis. These oxidation systems have been used to treat both individual organic and inorganic substances in water under laboratory conditions as well as real effluents from the different sources. Among AOPs, the Fenton's reagent (Fe^{2+}/H_2O_2) becomes more and more common for the treatment of

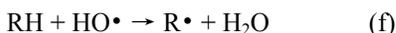
contaminated water. Many researches had demonstrated that the oxidation mechanism by Fenton's reagent was due to the reactive hydroxyl radicals generated in an acidic solution by the catalytic decomposition of hydrogen peroxide (Lopes De Moraes and Zamora, 2005; Kang and H. Wang 2000). In Fenton's reagent, iron and hydrogen peroxide are two major chemicals determining operation cost as well efficacy (Zhang, 2005). The equation of hydroxyl radicals generation is below:



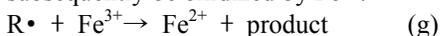
In addition to the main reaction, various additional competitive reactions are also possible involving Fe^{2+} , Fe^{3+} , H_2O_2 , superoxide and hydroxyl radicals (E. Neyens, J. Baeyens, 2003).



In the presence of organic substrates (RH) the highly reactive hydroxyl radical, a species with a relatively short life-span (rate constants in the range 10^7 to $10^{10} \text{M}^{-1} \text{s}^{-1}$), undergoes oxidation generating a new radical (C.Walling, 1974).



Possible organic compounds present in reaction mixture can suffer an abstraction of a hydrogen atom or addition of radical $\text{HO}\cdot$, in the case of olefins, creating a new radical ($\text{R}\cdot$), which can subsequently be oxidized by Fe^{3+} :



The equation above regenerates Fe^{2+} which ensures the continuity of the chain reaction that can lead ultimately to the decomposition of organic substrate in carbon dioxide and water.

The purpose of this work is the application of Fenton's reagent to partially degrade the organic load in water contaminated with petroleum hydrocarbon, expressed in terms of COD. The aim is to experimentally determine the optimal conditions for Fenton's reagent application, studying the effect of different operational variables such as pH, temperature and H_2O_2 and Fe^{2+} concentrations. Also, a kinetic study using a modified pseudo-first-order model

was made in order to determine the kinetic constants.

2- Materials and methods

2-1- Contaminated water

The oxidization reaction was conducted as laboratory-scale experiments for lowering the oil-products content dissolved in water solution. To evaluate the effectiveness of petroleum hydrocarbon removal from water by oxidization method, a model wastewater was prepared. It consisted of petroleum hydrocarbon (**5ml of crude oil**) which was left in water (20L) over 3 days to let it completely dissolve in water medium. Before performing the Fenton's oxidation, the initial COD of the sample was tested. The characterization of the wastewater is presented in the table below.

Table1: Characterization of wastewater

COD (mg/l)	pH
98	8.6

2-2 Experiments

Fenton's reagent experiments were carried out at room temperature in a 1 liter reactor using varying hydrogen peroxide and FeSO_4 dosages at varying pH values in order to determine the optimum conditions for best COD removal. Optimum pH determination was carried out for chemical dosages of H_2O_2 and, in order to find optimum chemical dosages at a predetermined optimum pH, H_2O_2 and FeSO_4 dosages varied. The pH adjustment was carried out with diluted sulphuric acid (H_2SO_4) and sodium hydroxide (NaOH) solutions. Typical experiments were carried out with 500mL wastewater to which an amount of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was added and mixed by stirring. The Fenton oxidation began with the addition of hydrogen peroxide solution. To avoid the coagulation of chemical, samples were taken to the mixing machine. The mixing rate was set at 120r/min. Temperature was also regulated. Samples were withdrawn from the reactor at pre-determined times. Control experiments were performed in the absence of either Fe^{2+} or H_2O_2 .

2-3 Reagents

During the experiments, the following reagents were used:

- H_2O_2 (1%, 2% and 3%)
- $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 0.1mol/L
- NaOH
- H_2SO_4
- $\text{K}_2\text{Cr}_2\text{O}_7$
- $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$
- Phenanthroline

peroxide present in the reaction media was checked by peroxides test. Dissolved oxygen was measured with DO machine. Others wastewater parameters were analyzed according to standard methods. All of oxidation experiments were performed, at least, three times and the observed margin of error was always less than 4% of the reported value.

2-4 Analytical methods

The COD analyses were performed by titration method using ammonium sulfate $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$, potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) and phenanthroline. The residual hydrogen

3- Results and discussions

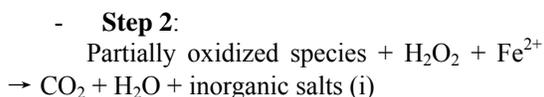
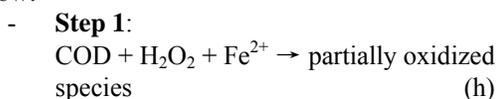
Table 2: Experimental conditions, COD values, COD removal (%) obtained after oxidation of petroleum hydrocarbon with Fenton's reagent

Experiments	concentration H_2O_2 (%)	T ($^{\circ}\text{C}$)	pH	Molar ratio ($\text{H}_2\text{O}_2/\text{Fe}^{2+}$)	COD Initial (mg/l)	CODFinal (mg/l)	Removal COD (%)
Exp 1	1%	28	3	9:1	98.34	87.33	11.20
Exp 2	2%	28	3	9:1	94.56	57.13	39.25
Exp 3	3%	28	3	9:1	97.25	28.61	70.58
Exp 4	3%	28	2	9:1	97.15	40.53	58.28
Exp 5	3%	28	5	9:1	95.48	32.96	65.48
Exp 6	3%	28	7	9:1	93.83	48.57	48.23
Exp 7	3%	28	9	9:1	95.35	73.84	22.55
Exp 8	3%	10	3	9:1	94.56	50.83	46.24
Exp 9	3%	20	3	9:1	95.74	32.60	65.94
Exp 10	3%	40	3	9:1	93.45	28.43	69.57
Exp 11	3%	50	3	9:1	95.43	31.45	67.04
Exp 12	3%	28	3	4.5:1	94.28	26.88	71.48
Exp 13	3%	28	3	18:1	96.50	38.48	60.12
Control experiments							
Experiments	Fe^{2+} (M)	H_2O_2 (%)	T ($^{\circ}\text{C}$)	pH	COD Initial (mg/l)	CODFinal (mg/l)	Removal COD (%)
C1	0	3	28	3	96.50	89.35	7.4
C2	0.1	0	28	3	97.15	95.60	1.6

Fenton's oxidation was carried out at a laboratory scale in order to minimize the impact of

petroleum hydrocarbon on natural water. The Fenton's oxidation pathway system to the

reduction of COD is always summarized as follow:



The extent of oxidation determines the extent of reduction of COD and the costs of application of Fenton's reagent depend on the amount of hydrogen peroxide used. Many cases have been faced whereby the complete mineralization of the organic compounds which produces carbon dioxide and water is not necessary achieved. The intermediate compounds minimize the consumption of chemical reagent and results in the great reduction of COD. Table 2 gives the initial and the final values of COD for each experiment and the removal COD obtained has been defined as:

$$\text{COD}(\%) = \frac{\text{COD}_0 - \text{COD}_f}{\text{COD}_0} \times 100 \quad (\text{k})$$

The table also summarizes also the experiments performed where-by the hydrogen peroxide concentration, temperature, pH and the initial concentration of ferrous ions were modified. As it can be observed, the removal COD for Fenton experiment after 2h of reaction, lay between 11.20% and 71.48% depending on the operating condition. These COD removal is attributed by the high reactivity of organic compounds with hydroxyl radicals. In Fenton's reagent, the hydrogen peroxide dosage could also be referred to as the stoichiometric weight ratio between the hydrogen peroxide and COD, which are calculated assuming the complete oxidation of COD.

3.1 Effect different dosage of hydrogen peroxide.

In the first set of experiments, the pH of the medium and the molar ratio (hydrogen peroxide and ferrous ions) were kept constant at 3.0 and 9:1 respectively. The values were chosen based on many researches and recommendations. (Stuart M. Peters, et al, 2001). The temperature was kept at 28 °C. The hydrogen peroxide concentration

was changed between 1 and 3%. Figure 1 shows the rate of COD reduction over time. It can be observed that COD reduction occurs principally at an initial period (20- 40 min) and as the reaction time increased the COD concentration profile was approaching a plateau. It is possible to observe that COD is significantly reduced within two hours of reaction. The kinetic attention will be focused on two hour of reaction in this study. The figure shows also that increasing the concentration of hydrogen peroxide (and proportionally ferrous ions concentration) has a positive effect on the COD removal rate. For the maximum hydrogen peroxide concentration studied (3%), the COD removal reaches 70.58%. This shows the Fenton's reagent ability to degrade a great amount of oxidizable organic matter in our water sample. The increase in COD conservation with the raise of hydrogen peroxide concentration results from the generation of hydroxyl radicals and, therefore, a greater extent of oxidation reaction. (P. Bautista, et al, 2007)

3.2 Effect of initial pH

The pH effect was studied through experiments Exp 3, Exp 4, Exp 5, Exp 6 and Exp 7. Figure 2 shows the COD reduction of petroleum wastewater for different initial values of pH. It reveals that Fenton's reagent is a highly pH sensitive process. The COD removal performance declined for pH values greater than 5.0. This occurs due to the formation of the ferric hydroxo-complexes, namely the precipitation of Fe^{3+} as $\text{Fe}(\text{OH})_3$, hindering the reaction between Fe^{3+} and H_2O_2 , and therefore the regeneration of Fe^{2+} . Besides, $\text{Fe}(\text{OH})_3$ catalyzes the self-decomposition of H_2O_2 (particularly unstable in basic media) to oxygen molecular and water, resulting in a decline of its oxidative capacity (P. Bautista, et al, 2007). On the other hand, for pH values lower than 2.0 the reaction of hydrogen peroxide with Fe^{2+} is seriously affected due to the formation of complex species $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$, which reacts slower with peroxide when compared to that of $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$. In addition, the peroxide gets solvated in the presence of high concentration of H^+ ion to form stable peroxone ion $[\text{H}_3\text{O}_2]^+$. The peroxone ion it leads to an electrolytic behavior on the part of hydrogen peroxide improving its stability and substantially reducing the reactivity with ferrous ion.

Therefore, the initial pH value has to be in the acidic range (3–4) to generate the maximum amount of $\text{HO} \cdot$ and oxidize the organic

compounds. In particular, the optimal value of pH is 3, which is in agreement with previous results obtained in other studies using Fenton's reagent.

3.3 Effect of temperature

A temperature range from 10 to 50°C was studied in order to observe the effect of temperature on Fenton's reagent (Exp 3, Exp 8, Exp 9, Exp 10 and Exp 11). The obtained COD removal efficiencies after oxidation with Fenton's reagent are presented in figure 3. It can be deduced from this figure that the optimal range for the temperature is in the range of 28 to 40 °C. For lower temperatures studies, the COD reduction indicates that the extent of the reaction was not complete within 2h of reaction. For 10 °C the removal COD 46.24% and for 20 °C was 65.94%. This hypothesis is confirmed by the presence in the reaction medium of residual levels of hydrogen peroxide. The removal COD rate went down above the optimal range of temperature. The hydrogen peroxide self-decomposition into molecular oxygen and water is being significant at 50 °C, partially reducing the effectiveness of the oxidation process and the generation of hydroxyl radicals. The Fenton's reaction is an exothermic process. The moderation of the temperature in the reactor is important for safety reasons and to avoid runaway situations.

3.4 Effect of ferrous ions concentration

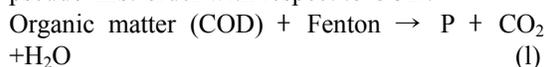
To determine the effect of the concentration of ferrous ions they were carried out as experiments 3, 12 and 13. As a control experiment, it is possible to confirm that using only hydrogen peroxide, the obtained COD reduction is very small (7%). The results indicated that by increasing the Fe²⁺ ion concentration in the solution, the COD removal increased. Oxidation rate increases with Fe²⁺ ion concentration as the results of higher production of hydroxyl radicals. In spite of the improvement in reaction kinetics achieved with higher Fe²⁺ concentration, it is important to reduce costs in chemicals and minimize the production of Fe³⁺ that will probably precipitate as Fe(OH)₃. Consequently, it appears that in the case of Exp 12 where H₂O₂:Fe²⁺ = 4.5:1, the final conversion of COD (71.48%) is only slightly above the Exp 3 (70.58%), where the initial concentration of iron was half (H₂O₂:Fe²⁺ = 9:1). Probably a greater concentration of Fe²⁺ can cause the recombination of radical HO•. In this case, the Fe²⁺ reacts with the hydroxyl radical functioning as a scavenger

(C. Walling, 1974). It is therefore desirable that the amount of iron used must be a compromise that can minimize the production of sludge from iron complex. The value of H₂O₂:Fe²⁺ = 9:1 was selected and used as a reference for other tests. Finally, to confirm the symbiotic importance of hydrogen peroxide in Fenton's reagent using only ferrous ions (C2), there is practically no decrease on COD (only about 1.6%).

3.5 Kinetic study

It must be remarked that since we have a very complex reaction mixture, the overall kinetics will be analyzed by taking into the total organic compound present within the reactor. Due to the complexity of chemical compounds formed as intermediates in the Fenton reagent oxidation, make virtually impossible carry out a detailed kinetic study with the different individual reactions that take place during chemical oxidation. However, it is possible to conduct an approximated kinetic study according to some parameter that represents the overall organic matter, in this case, the COD. Figure 4 shows the effect of temperature in COD reduction during Fenton's reagent application to the wastewater contaminated with petroleum hydrocarbon. Through this study it will be possible to assess and obtain a series of apparent kinetic constants that reflect the greater or lesser speed with which follows the process of oxidation in the different conditions set operative.

The progressive disappearance of organic load, measured as COD, by Fenton reagent may be represented by a simple irreversible reaction of pseudo-first-order with respect to COD:



P symbolizes the intermediate products of degradation.

By assuming that these reactions follow pseudo-first-order kinetics with respect to COD, we may write:

$$-\frac{d\text{COD}}{dt} = k\text{COD} \quad (m)$$

which can be integrated between $t = 0$ and $t = t$, yielding:

$$\ln\left(\frac{\text{COD}_o}{\text{COD}}\right) = kt \quad (n)$$

According to this expression, a linear representation of the first term versus time, should lead to a straight line for each experiment whose slope is k

Figure 5 shows this kinetic of pseudo-first-order for the Fenton system in the case of experiments in which we varied the temperature. Least squares regression analysis gave the k values listed below:

$K=0.00635 \text{ min}^{-1}$ at 10°C , 0.01205 min^{-1} at 20°C , 0.02165 min^{-1} at 28°C , 0.02465 min^{-1} at 40°C and 0.03 min^{-1} at 50°C . The kinetic reaction for the Fenton system is generally limited by the speed of $\text{HO}\cdot$ radical generation but also dependent on specific wastewater to be treated.

A multiple regression analysis permits to calculate the value of k for experiments with Fenton's reagent at pH 3. To analyze the experimental data, the integral method was used for the first step. The results are shown in figure 5, which clearly gives evidence and is in agreement with the equation (n). From the slopes of lines, the apparent second-order kinetic constants were obtained and from the corresponding Arrhenius-type plot (figure 6) k values can be correlated by an Arrhenius-type expression. The graphical representation of $\ln k$ on the basis of $1/T$ (Figure 6) allows the calculation of the parameters of Arrhenius equation:

$$\ln k = \ln A_0 - \frac{E_a}{RT} \quad (o)$$

Where E_a is the energy of activation (kJ mol^{-1}) and A_0 the pre-exponential factor (min^{-1})

The regression analysis with expression of constants against temperature led the following values of: $E_a = 31.877 \text{ kJ mol}^{-1}$ and $A_0 = 4.29 \times 10^{-2} \text{ min}^{-1}$

For the zero-order reaction rate data, no correlation could be established. This is probably due to the decomposition of hydrogen peroxide, which strongly increases with the temperature - approximately 2.2 times for each 10°C rise in the range $20 - 100^\circ\text{C}$ (US peroxide, 2001). This work shows that the use of the Fenton's reagent requires a very fine control of the operating conditions to achieve high performances, otherwise, limitations will unavoidably arise from hydrogen peroxide decomposition via non-producing $\text{HO}\cdot$ reactions, reaction of the $\text{HO}\cdot$ species with non-target chemicals or even other well-know reactions reported in the literature that reduce the amount of hydrogen peroxide available for Fenton's reaction (Hunling SG, et al. 2000).

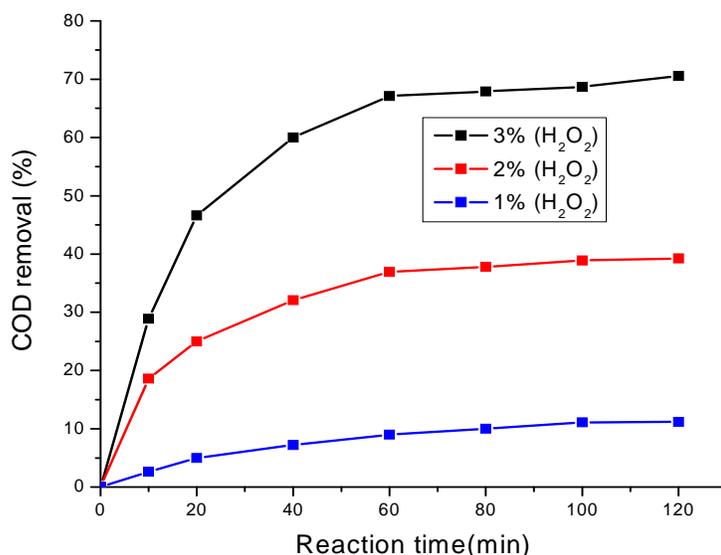


Figure1: Effect of hydrogen peroxide concentration on the COD removal

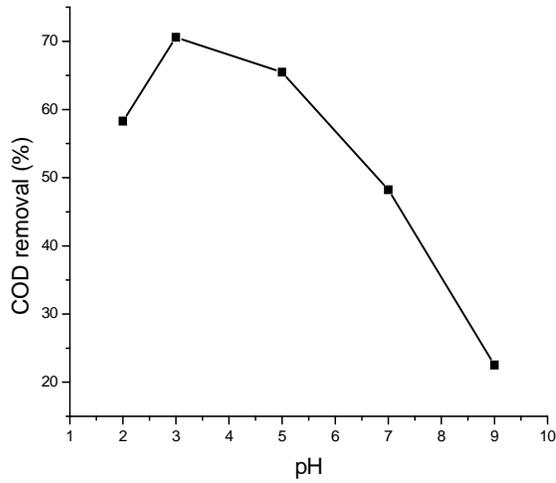


Figure2: influence of pH on the COD removal

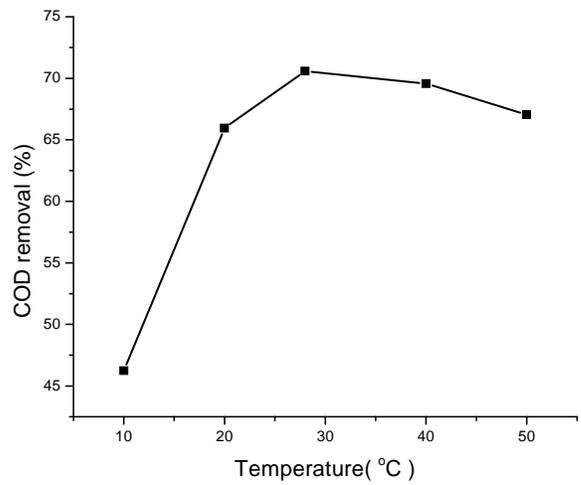


Figure3: initial temperature influence on the COD removal

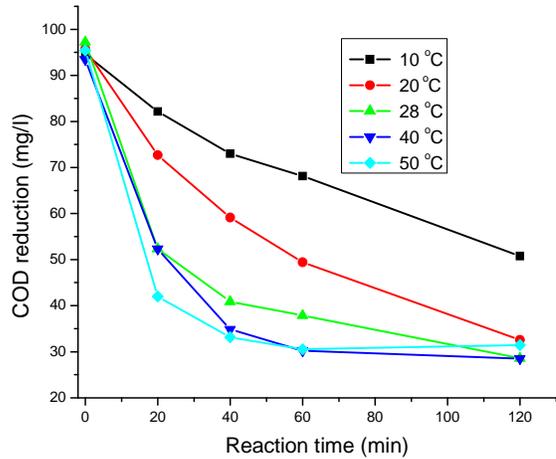


Figure4: Effect of temperature on the COD reduction

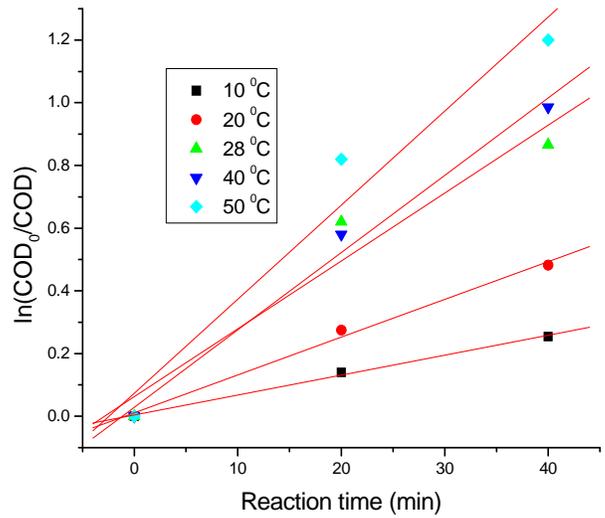


Figure5: Determination of the first-order rate constants k for different temperatures

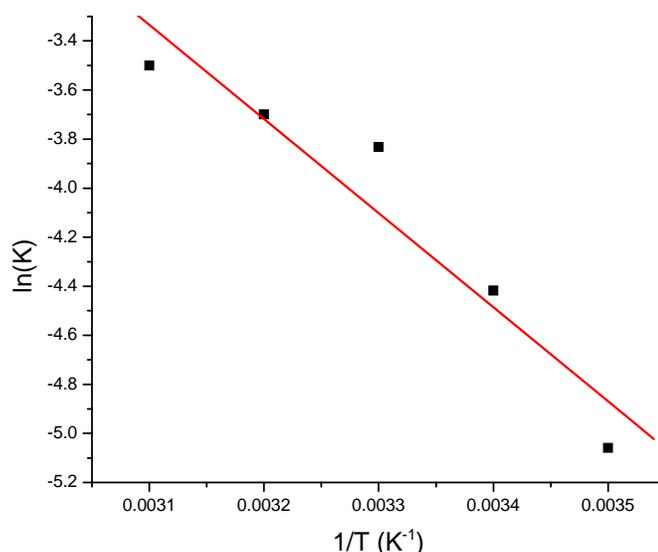


Figure6: Arrhenius plot of the apparent first-order kinetic constants for the reaction

4- Conclusion

The overall results of this study indicate that the application of Fenton's reagent is a feasible method to partially treat water contaminated with petroleum hydrocarbon achieving a significant decrease of COD. Fenton's reagent at initial pH 3, temperature of 28 °C, molar ratio $H_2O_2:Fe^{2+}=9:1$ and H_2O_2 3%, leads to a COD reduction of 70.58%. A simple kinetic analysis based on COD was carried out. A modified pseudo-first-order equation allowed to describe the process well and led to the determination of kinetic parameters useful for the design of industrial reactors. A value of 31.877kJmol^{-1} was obtained for the apparent activation energy (E_a) in Arrhenius equation.

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