REMOVAL OF OIL FROM WASTEWATER BY ADVANCED OXIDATION PROCESS / HOMOGENEOUS PROCESS

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ABSTRACT

In the present work advanced oxidation process, photo-Fenton ($UV/H_2O_2/Fe^{+2}$) system, for the treatment of wastewater contaminated with oil was investigated. The reaction was influenced by the input concentration of hydrogen peroxide H_2O_2, the initial amount of the iron catalyst Fe^{+2}, pH, temperature and the concentration of oil in the wastewater. The removal efficiency for the system $UV/ H_2O_2/Fe^{+2}$ at the optimal conditions and dosage (H_2O_2 = 400mg/L, Fe^{+2} = 40mg/L, pH=3, temperature =30°C) for 1000mg/L load was found to be 72%.

KEYWORDS: Advanced oxidation process; Fenton reagents; photo-Fenton process; H_2O_2; oil degradation.

ازالة الزيت من المياه الملوثة باستخدام عملية الاكسدة المتقدمة / الطريقة المتجانسة

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الخلاصة

تم في هذا البحث دراسة معالجة المياه الملوثة بالزيت باستعمال احد طرق الاكسدة المتقدمة/المتجانسة بواسطة طريقة photo-Fenton ($UV/H_2O_2/Fe^{+2}$) .

تاثرت التفاعلات عند اضافة تراكيز مختلفة من بيروكسيد الهيدروجين (H_2O_2) ومن الحديد(Fe^{+2}) كعامل مساعد والدرجة الحامضية (pH) والحرارة وتركيز الزيت في المياه الملوثة .

اظهرت التجارب ان نسبة الازالة للزيت بلغت 72% بواسطة الاكسدة المتقدمة / المتجانسةباستعمال طريقة photo - Fenton $UV/H_2O_2/Fe^{+2}$ بالظروف المثالية (بيروكسيد االهيدروجين = 400 ملغم /لتر، اوكسيد الحديدوز= 40 ملغم /لتر، الـ دالة الحامضية pH=3 ودرجة الحرارة = 30 درجة مئوية وعند تركيز 1000ملغم /لتر للزيت في الماء) .

الكلمات الرئيسية : عمليات الاكسدة المتقدمة، محاليل الفنتون، الهيدروجين بيروكسايد ، تحلل الزيت

1. INTRODUCTION

In the recent years, the increasing in the human being activities (industrial, agricultural and domestic fields) results an extremely organic load in the wastewater treatment plants, thus results a discharge to the river which will not agreed with the health and environmental standards. Oil-contaminated wastewater can cause serious environmental problems due to its hazardous nature. The volume of diesel/gas oil contaminated wastewater from petroleum filling stations has increased in line with the number of such stations required to cater for the growing number of vehicles. According to data provided by International Energy Agency (IEA), the world consumption of diesel oil increased during the period 1990 to 2003 from 454,747 to 684,022 million liters. In Iraq there is 507 car-wash facilities (except Kurdistan), (Ministry of Environment report 2011) and some of them discharge its wastewater (almost emulsifiable oils) to sewer systems without treatment. Also as a result of human activities, there are many accidental discharges of hydrocarbon oil to the natural environment during its processing, transportation and storage. Photo in **Fig.1**, illustrate oil contaminated wastewater in Iraqi stream. Diesel /gas oil causes eye and skin irritation in humans, but otherwise its effects on humans are considered to be poorly investigated (Muzyka et al. 2002). Diesel is considered to be harmful and possibly carcinogenic to humans and it contains PAHs that create a risk for human health because of their carcinogenic, mutagenic and teratogenic properties (Grant et al. 2007). Standards and regulation were adopted for discharge of oily wastewater into surface water or sewage systems. These regulations may vary from country to country, and even within a country itself. The allowable hydrocarbons and its derivatives discharge into water bodies about 10 mg/L in Iraq (Iraqi preservation law, Law No. 2, 2001).Several conventional techniques such as gravity separation; dissolved air flotation; demulsification ; coagulation; flocculation; biological treatment have been employed for the treatment of oily wastewater. However, these processes can only transform the pollutants from one phase to another without destroying them. Also these processes result in concentrated sludges which require further processing and disposal. In addition, conventional treatment processes have difficulty in fully removing emulsified oil or small oil droplets. (Neyens and Baeyens 2003). Advanced oxidation processes (AOPs) have been investigated for the oil-contaminated wastewater treatment as an alternative to conventional treatment techniques. AOPs are characterized by the use of highly reactive intermediates, hydroxyl radicals (HO•), which attack the organic pollutants in the wastewater and mineralize them. Advanced oxidation processes (AOPs) have the advantage of rapid oxidation of pollutants to harmless end products. Such processes include homogenous processes such as Fenton's reagent, H_2O_2 and ozone and heterogeneous photocatalysis using semiconductors such as TiO_2 and ZnO (Neyens and Baeyens 2003) .The possible reaction pathways involving the attack of hydroxyl radicals onto organic compounds are:

1- Electrophylic addition of a hydroxyl radical to organic compounds (unsaturated or aromatic) that contain a π bond leading to the formation of organic radicals.

2- Hydrogen abstraction by reacting the hydroxyl radical with a saturated aliphatic compound

3- Electron transfer with reduction of the hydroxyl radical into a hydroxyl anion by an organic substrate, (Bossmann et al., 1998).

Tiburtius et al.(2005) investigated the oxidation potential of the Fenton and photo-Fenton processes to degrade a solution containing benzene, toluene and xylenes (BTX) and water contaminated with gasoline (containing 25% ethanol). Rapid degradation of the BTX compounds was observed as well as the formation of intermediate phenolic compounds in the initial reaction periods that were completely degraded after 30 minutes of reaction. A level of 75% in degradation of total hydrocarbons present in gasoline-contaminated water was achieved, and the BTX compounds were removed in the first minutes of the reaction (when phenolic compounds were formed), after which they degraded slowly. Galvao et al., (2006) studied the photo-Fenton process for treating water containing diesel oil. They conducted a study to optimize the amount of reagents used, in order to achieve high levels of degradation with a smaller amount of reagents. With Fe^{+2} ion concentrations of 0.1 mM, an amount below the maximum discard limit permitted by Brazilian law (0.27 mM), there was 99% degradation of the organic compounds originally present in the wastewater, expressed as total organic carbon (TOC).Dincer et al.(2008) investigated the degradation of wastewater from oil recovery industry by the Fenton, photo Fenton and UV/H_2O_2 processes. The COD removal efficiency was strongly affected by many factors such as the concentration of H_2O_2, Fe^{+2} and the ratio of organic materials to the Fenton reagents. The optimal mass ratio for H_2O_2/Fe^{+2} yielding the highest COD removal was found to be 8.658 corresponding to 200.52 g L^{-1} H_2O_2 and 23.16 g L^{-1} Fe^{+2} concentrations for 60 minutes reaction time. Fenton process gave a maximum COD reduction of 86% (for 21000 to 2980 mg/L).Tony et al.(2009) demonstrates the suitability of advanced oxidation processes for the treatment of oil-water emulsion and car-wash wastewater. The Photo-Fenton process using the artificial UV light is more efficient than the Fenton and UV-radiation treatment processes. After 150 min of the Photo-Fenton reaction, the COD removal rate ranged from 60-82 %, depending on the initial concentration of the emulsion (for the concentration range 1500-200 mg-COD/L, respectively).

Tony et al., (2012) studied the application of advanced oxidation processes (AOPs) to the treatment of wastewaters contaminated with hydrocarbon oil. Three different oil-contaminated wastewaters were examined and compared:

- Real hydrocarbon wastewater collected from an oil refinery;
- Real hydrocarbon wastewater collected from a car-wash facility located at a petroleum filling station; and
- Synthetic hydrocarbon wastewater generated by emulsifying diesel oil and water. The AOPs investigated were Fe+2/H2O2 (Fenton's reagent), Fe+2/H2O2/UV (Photo-Fenton's reagent). Laboratory-scale batch and continuous flow experiments were undertaken. The photo-Fenton parametric concentrations to maximize COD removal were optimized: pH = 3, H_2O_2= 400 mg/L and Fe^{+2} = 40 mg/L. In the case of the oil-refinery wastewater, photo-Fenton treatment achieved approximately 50% COD removal and, when preceded by physicochemical treatment, the percentage removal increased to approximately 75%. In the present study the photo-Fenton system was used for the mineralization of oil in wastewater. The effect of the reagent dosage and operating conditions were investigated.

2. MATERIALS AND METHODS

2.1 Reagents

Commercial gas oil was used in this study as the model pollutant for the degradation of oil from wastewater by AOP.

Gas oil was analyzed in Petroleum Research and Development Center / Ministry of Oil. The chemical compositions are (Paraffins 62.5%, Aromatics 18.7% and Naphthenes 18.8%.

The paraffinic fraction composition of gas oil was identified by gas chromatography and revealed more than 66 alkane compounds, **Fig.2**. From this figure it can be observed that gas oil has a higher relative concentration in C_9, C_{11}, C_{12}, C_{13}, C_{14} and C_{15}.

The functional groups present in gas oil were detected by FTIR analysis, **Fig. 3**.

Table 1 shows all the chemicals that used in the experiments. All the samples were prepared by dissolving requisite quantity in distilled water. The pH of the solution was adjusted by using H_2SO_4 solution.

2.2 Equipments

The experiments were carried out in a batch mode laboratory-scale reactor, **Fig.4** .The reactor consists of Pyrex glass cylinder 3 litter volume mounted at magnetic stirrer with heater (MSH-300N, BOECO, Germany).

UV radiation (254 nm) was generated from UV lamps (TUV 11W 4P-SE, Philips, England), which was fixed vertically at the top of the reactor. The lamp was totally immersed in the cylindrical reactor. UV lamp was sheathed in quartz sleeve for protection. The distance between the lamp and the reactor was fixed to be 5 cm to ensure maximum light irradiation as mentioned by Chiu et al., (1999).

The turbidity of the solution for three experiments was measured using turbidity meter (Hanna microprocesser, Italy).

Initial pH of solution was monitored using a pH meter from (WTW Co., German, INOLAB 720).

2.3 Experimental Procedure

The procedure was performed as follows:

The desired concentration of gas oil (500-2000) mg/L was prepared and the pH was adjusted

Before adding the reagents by adding a dilute H_2SO_4 solution in the reactor.

The Fenton reagents were introduced to the solution by adding ferric chloride (10-100 mg/L) and then the hydrogen peroxide (100-800 mg/L). The solution was then subjected to stirring using a magnetic stirrer at 200 rpm for 150 min. The heater was adjusted at the required temperature.

Samples at a regular time intervals (30 min) were taken for COD analysis.

2.4 Analysis

Chemical Oxygen Demand of samples was analyzed by using COD Photometer. The appropriate amount of sample (0.2ml) was introduced into commercially available digestion solution (HR-Rang: 0-15000mg/L) containing potassium dichromate, sulfuric acid and mercuric sulfate. The mixture was then incubated for 120 min at 150°C in a COD reactor (model RD-125, Lovibond Company, Germany). After oxidation is complete, the COD concentration was measured colorimetrically at 605 nm using a DR/2010 spectrophotometer (model RD-125, Lovibond, Germany).

3. RESULTS AND DISCUSSION

3.1 The effect of initial H_2O_2concentration

The effect of initial concentration of H_2O_2 (100, 200, 400, 600 and 800 mg/L) on photo-Fenton process was tested to optimize the amount of H_2O_2 required to degraded the oil. Initial amount of Fe^{+2} (40 mg/L), initial oil concentration 1000 ppm (1550±60COD) with pH equal to 7 were used in these experiments. The temperature was maintained at 20°C through these experiments. **Fig. 5** shows the relation between the removal efficiency and the irradiation time for different initial concentration of H_2O_2. From this figure it can be noticed that the degradation of oil increased as the concentration of H_2O_2 increased from 100 to 400 reaching a maximum removal efficiency of 54% at 400 mg/L after 150 minutes of irradiation time. The addition of H_2O_2 above this value had a negative effect, 47% at 800 mg/L of H_2O_2, as shown in **Fig. 6**.

Many investigators give more than one explanation for these phenomena, among them (Shihab, 2011):

- Hydroxyl radicals generated from the direct photolysis of hydrogen peroxide were the main responsible species of the degradation process. It was expected that increasing the concentration of H_2O_2 reducing the rate of degradation due to the reaction of hydrogen peroxide with these radicals, and hence acts as an inhibiting agent (i.e. self–scavenging of $HO^\bullet$ by H_2O_2), eq.(1).

$$H_2O_2 + HO^\bullet \rightarrow HO_2^\bullet + H_2O \qquad (1)$$

- Hydroxyl radical may recombine and participate in radical-radical reactions to form H_2O_2, eq.(2).

$$2HO^\bullet \rightarrow H_2O_2 \qquad (2)$$

- At higher H_2O_2 concentrations lower light intensity available for oil degradation, since H_2O_2 also absorb lights in the system.
- Hydroxyl radical efficiently reacts withH_2O_2 and produce $HO^\bullet_2$. Since $HO^\bullet_2$ radicals are not as reactive a $HO^\bullet$, then low degradation may obtained, eq.(1).H_2O_2 is $HO^\bullet$ scavenger and so an excess H_2O_2 can result in a net decrease in the treatment efficiency.

However, if H_2O_2 does is low, $HO^\bullet$ formation will also be low, decreasing the treatment efficiency. Therefore, a balance must be maintained between excess and low levels of H_2O_2.

3.2 Effect of initial iron salt (Fe^{+2}) concentration

The effect of initial Fe^{+2} concentrations on photo-Fenton process was tested by carrying out experiments with various amounts of Fe^{+2} (10, 20, 40, 50, 60 and 100) mg/L. The optimum H_2O_2 concentration (400 mg/L) from the previous section was used in these experiments. The initial oil concentration1000mg/L and initial pH=7 were used for these experiments. Temperature was maintained at 20°C.From **Figs.7 and 8. ,** it can be noticed that the degradation rate of oil distinctly increased with the increasing amounts of iron salt. It reached its maximum value (54% removal) at 40 mg/L after about 150 min of irradiation time .The addition of the iron salt above this value did not affect the degradation, it had a negative effect, it reaches 20% removal at 100 mg/L Fe^{+2} . This finding is in agreement with the previous observation of Tony et al. (2009) and Tony et al., (2012).

The negative effect can be explained as:

- Addition of ferrous ions increases wastewater brown turbidity during the photo-treatment, which hinders the absorption of the UV light, required for the photo-Fenton process, (Dincer et al., 2008).

- Excess ferrous ions can react with hydroxyl radical according to eq. 3 decreasing the attack of hydroxyl radical on organic substrates.

$$Fe^{2+} + HO^{\bullet} \rightarrow Fe^{3+} + HO^{-} \qquad (3)$$

- Excess ferrous ions can react with OH radical producing compound which inhibit reaction rate, (Neyens and Baeyens, 2003).

3.3 Effect of initial pH

The effect of the pH plays an important role in the photo-Fenton treatment process. Different values of pH were examined in this study (2, 3, 4, 5,7and 8) keeping the other parameters and dosage constant (Fe^{+2}= 40mg/L, H_2O_2= 400mg/L, oil concentration=1000 mg/L and temperature=20°C).The results were shown in **Figs. 9 and 10.** From these figures it can be observed that a maximum removal efficiency (66%) was obtained at pH=3. Above this value the removal efficiency was decreased gradually except for pH=8, a marked decrease in removal efficiency (28%) was observed.

The optimum pH has been observed to be 3 in many studies (Pignatello 1992 and Tony et al., 2012).

The pH affects the activity of both the speciation of iron, and hydrogen peroxide decomposition. The decrease in removal efficiency can explain as follows:

At lower pH (< 2.5), the formation of $[Fe(H_2O)_6]^{2+}$ complex occurs rather than $[Fe(OH)(H_2O)_5]^{+}$ complex , which reacts more slowly with hydrogen peroxide and, therefore, produces less amount of reactive hydroxyl radicals thereby reducing the degradation efficiency as mentioned by Mota et al.,(2008). In addition, the scavenging effect of hydroxyl radicals by hydrogen ions becomes important at a very low pH and also the reaction of Fe^{3+} with hydrogen peroxide is inhibited (Pignatello, 1992).

At operating pH of > 4, iron reacts with the hydroxide ions (HO−), precipitating the iron hydroxide ($Fe(OH)_2$ or $Fe(OH)_3$), which does not react with H_2O_2, precluding the Fenton reaction. Also, the oxidation potential of OH radical is known to decrease with an increase in the pH (Kwon et al., 1999).

3.4 Effect of temperature

Reaction temperature is another important process parameter that affects the degradation process. Different temperatures (20, 30 and 40 °C) were used. The dosage of the reagents and other parameters were remain constant (Fe^{+2}= 40 mg/L, H_2O_2= 400 mg/L, oil concentration=1000 mg/L and pH=3.The results were plotted in **Figs. 11 and 12**.These figures show that the removal efficiency of oil increases from 66% at 20°C to 72% at 30°C and then decreased to 69% at 40°C. The increase in temperature accelerate the decomposition of H_2O_2 thus increase the generation of OH radicals which enhances the degradation process.

The result shows that only a marginal effect on degradation performance was obtained. This was coinciding with Rivas et al. (2004), they reported that the degradation efficiency is unaffected even when the temperature is increased from 10 to 40°C. Above 40°C, efficiency of hydrogen peroxide utilization decreases due to accelerated decomposition of hydrogen peroxide into water and oxygen as mentioned by Nesheiwat and Swanson, (2000).

3.5 Effect of the initial oil concentration

Different concentrations of gas oil (500, 1000 and 2000) mg/L were used at H_2O_2 = 400 mg/L, Fe^{+2} = 40 mg/L, pH= 3 and temperature=30°C.The results were plotted in **Figs.13 and 14**. From these figures it can be observed that the removal efficiency decreases linearly from 79% to 50% as the concentration of gas oil increases from 500 to 2000 mg/L respectively. This can be attributed to the increase in turbidity of the solution. The turbidity for 2000 mg/L oil solution was measured to be 47 NTU, whereas for 500 mg/L oil solution the turbidity was 6 NTU only. Decreasing turbidity clearly enhance the penetration of the UV light, resulting in enhanced oil removal, this observation was reported by (Najjar et al. 2001).

4. CONCLUSIONS

The main conclusions that can be drawn from the experimental work of this study are as follows:

1. The removal efficiency of the system $UV/H_2O_2/Fe^{+2}$ which considered in this study for the degradation of oil in wastewater was found to be 72%.

2. The dosage of the reactants, H_2O_2= 400 mg/L and Fe^{+2}= 40 mg/L, were found to be the optimum.
3. The efficient operating temperature was found to be 30°C.
4. The best pH for the system $UV/H_2O_2/Fe^{+2}$ which considered in this study was found to be equal to 3.

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Table 1: List of chemicals used

Compound	Formula	Vender
Hydrogen peroxide	H_2O_2	Scharlau (30 % wt/wt)
Ferrous chloroide tetrahydrate	$FeCl_2.4H_2O$	BDH (97% purity)
Sulfuric acid	H_2SO_4	Riedel-deHaen (99 % purity)

Fig. 1: Surface water oil contaminated in a AL- Fataha

Fig. 2: The paraffinic fraction composition of gas oil.

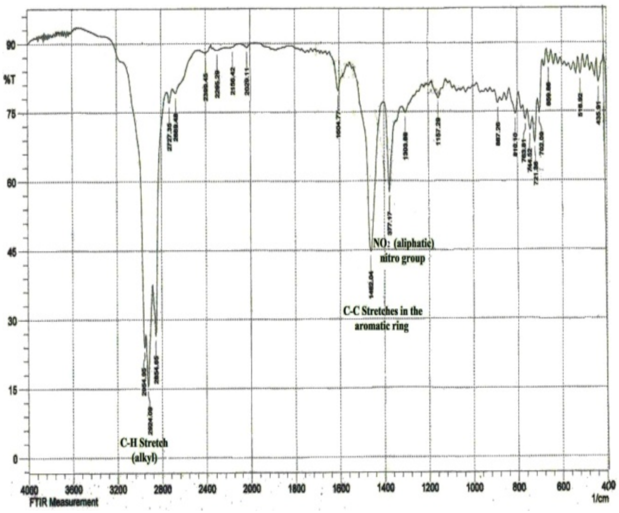
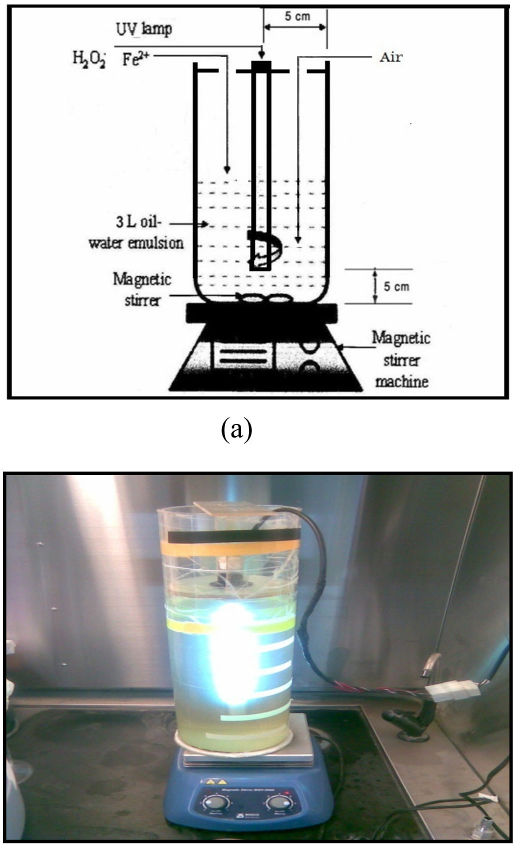


Fig. 3: The functional groups of gas oil detected by FTIR analysis.



(a)

(b)

Fig.4: Laboratory scale batch reactor
(a) Schematic diagram of batch reactor
(b)Photograph of batch reactor

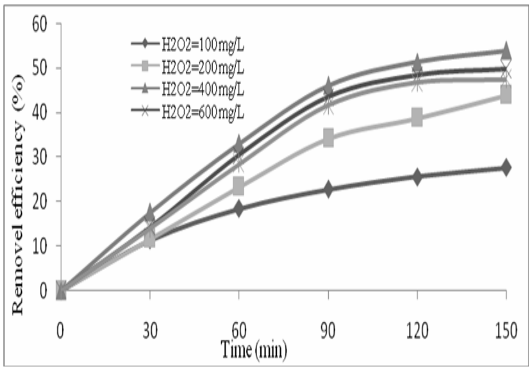


Fig.5: Effect of initial hydrogen peroxide (H_2O_2)concentrations on the degradation of oil by photo-Fenton system at Fe^{+2}= 40 mg/L, pH= 7, oil conc.=1000 mg/L and temp. =20°C.

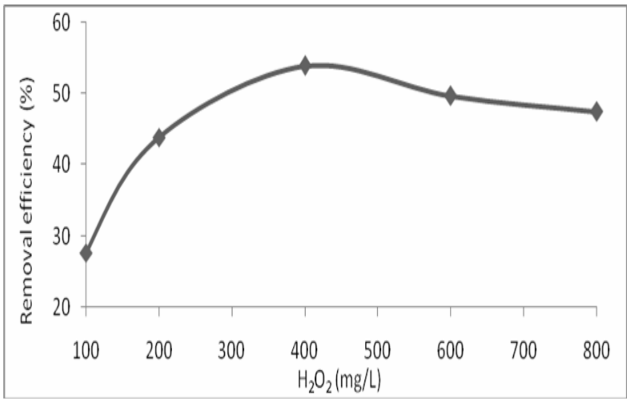


Fig.6: Degradation of oil as a function of hydrogen peroxide(H_2O_2)initial concentration by photo- Fenton system at Fe^{+2}=40 mg/L, pH=7,oil conc.=1000 mg/L ,temp. =20ºC and, irradiation time 150 min.

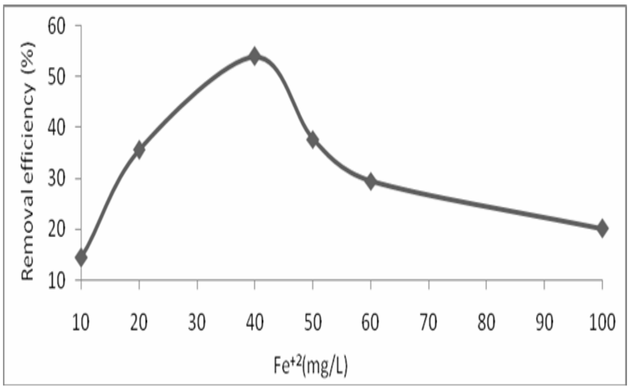


Fig.7: Effect of initial iron salt (Fe^{+2}) concentrations on the degradation of oil by photo Fenton system at H_2O_2=400 mg/L, pH=7, oil conc.=1000 mg/L and temp.=20°C.

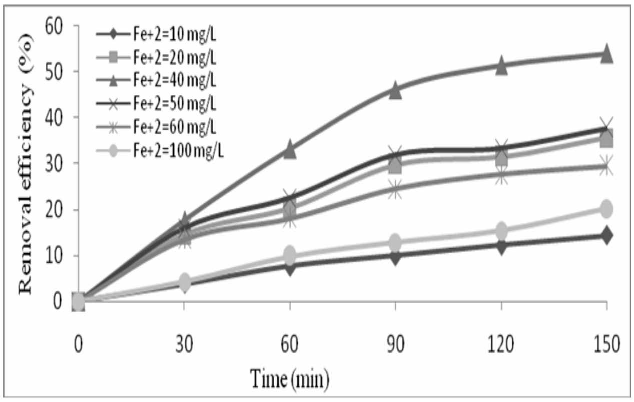


Fig.8: Degradation of oil as a function ofFe^{+2} initial concentration by photo Fenton system at H_2O_2=400 mg/L, pH=7, oil conc.=1000 mg/L , Temp.=20ºC and irradiation time 150 min.

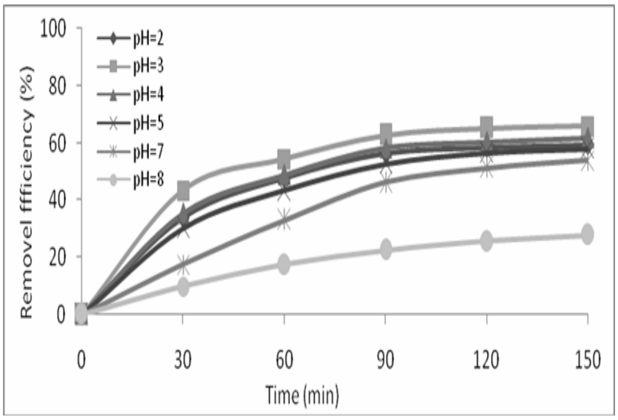


Fig.9: Effect of difference pH on degradation of oil by photo Fenton system at H_2O_2=400 mg/L, Fe^{+2} = 40 mg/L, oil conc.=1000 mg/L and temp.=20ºC.

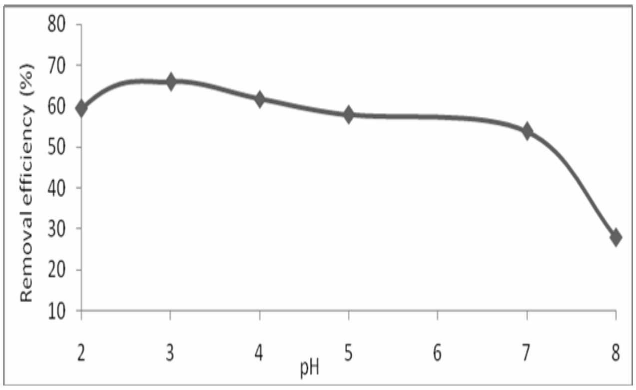


Fig.10: Degradation of oil as a function of pH by photo Fenton system at H_2O_2=400 mg/L, Fe^{+2} = 40 mg/L, oil conc.=1000 mg/L ,temp. =20ºC and irradiation time 150 min.

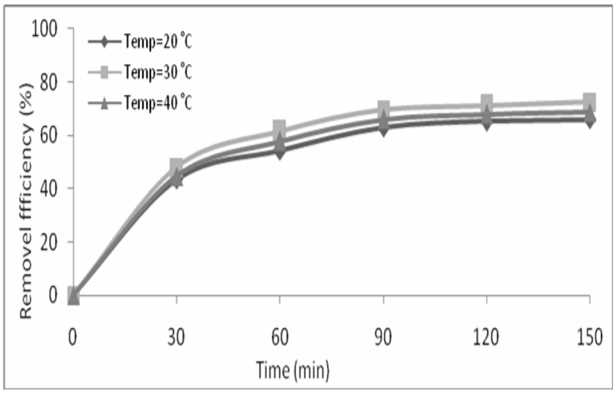


Fig.11: Effect of difference temperatures on degradation of oil by photo Fenton system at H_2O_2=400 mg/L, pH=3, oil conc.=1000 mg/L and Fe^{+2}=40 mg/L.

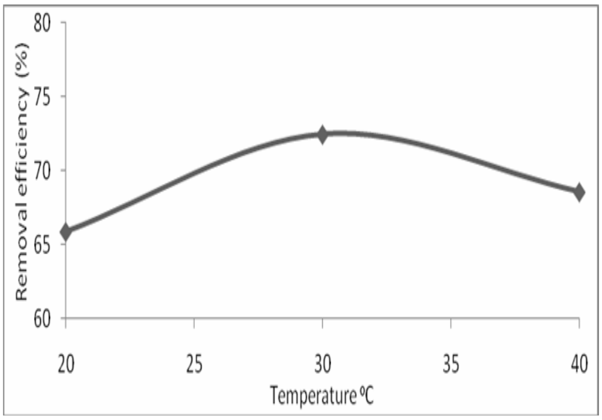


Fig.12: Degradation of oil as a function of temperature by photo Fenton system at Fe^{+2}=40 mg/L, pH= 3, oil conc.=1000mg/L , H_2O_2 =400 mg/L and irradiation time 150 min.

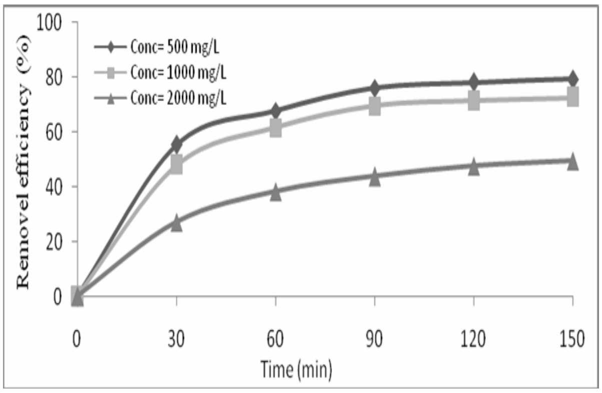


Fig.13: Effect of different oil concentrations on the degradation of oil by photo Fenton system at H_2O_2=400 mg/L, Fe^{+2}=40 mg/L, temp. = 30°C and pH=3.

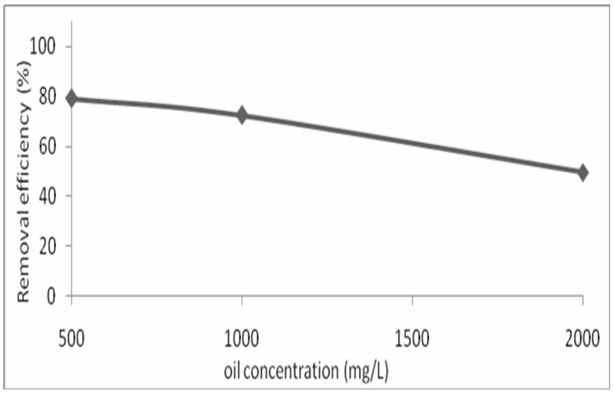


Fig.14: Degradation of oil as a function of different oil concentrations by photo Fenton system at H_2O_2= 400 mg/L, Fe^{+2}=40 mg/L, pH=3, oil conc.=1000 mg/L ,temp. =30ºC and irradiation time 150 min