Industrial Wastewater Treatment in North Gas Company By Using Coagulation – Flocculation Process

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Abstract

Recently, water pollution considered a major problem that faced the human. Large quantities of water consume in various industrial oil refinery processes, where the wastewater discharged from these processes contains high organic, phosphate, ammonia, nitrite compounds, and toxic substances. Regarding that, making this resource a fresh is a major concern.

Furthermore, a great attention has been given for the removal of these contaminants discharged by Fenton process combined with coagulation process. Combined Fenton/Coagulation process was used and applied in this study as a novel wastewater treatment to remove Chemical Oxygen Demand (COD), Phosphate (PO_4), Ammonia (NH_3), Nitrite (NO_3) compounds, and turbidity (NTU) from industrial wastewater in North Gas Company (NGC), Iraq. The industrial wastewater used in this study was characterized with 114 mg/L COD ,10.28 mg/L PO_4, 4.123 mg/L NH_3, 95.6 mg/L NO_3, 98 NTU Turbidity. The main goal from this work was to evaluate the performance removal efficiency of combined Fenton/Coagulation process and examine the effect of various operational parameters such as Fenton's dosages (H_2O_2, Fe^{2+}), the concentration of coagulant (Alum) on the removal efficiency. The results obtained in this work revealed that maximum removal efficiency of COD, PO_4, NH_3, NO_3, turbidity was 89.43%, 72.94 %, 91.065%, 90.96%, 89.85%, respectively was achieved using Fenton combined with coagulation process at 60 mg/L of Alum and 2.5 Fenton's mole ratio. Overall it can be established that Combined Fenton/Coagulation process plays an important role in obtaining good results and had better removal efficiency. All the experiments were carried out using jar test apparatus at constant temperature (room temperature).

Keywords: Wastewater; Removal Efficiency; Chemical Oxygen Demand; Phosphates; Nitrates; Ammonia; Turbidity.

معالجة مياه المطروحات الصناعية في شركة غاز الشمال بوساطة أستخدام عملية تجميع فنتون/ التخثير

الخلاصة

حاليا احد المشاكل الرئيسة التي تواجه الانسان هو تلوث الماء. كميات كبيرة من الماء تستهلك في مختلف العمليات الصناعية لتكرير النفط، إذ إن مياه المخلفات المطروحة من هذه العمليات الصناعية تحتوي على كميات عالية من مختلف المواد العضوية، الامونيا، النترات، و المواد ذات السمية. في هذا الصدد لهذا فان جعل هذا المصدر الطبيعي صالح للشرب و غير ملوث يعد مشكلة اجتماعية و اقتصادية كبيرة. لذلك جهود كبيرة قد كرست لأزالة هذه الملوثات المطروحة في مياه المخلفات بأستخدام عملية فنتون احد طرق الاكسدة المتقدمة مع عملية التخثير. في هذه الدراسة تم استخدام عملية فنتون/التخثير كطريقة حديثة لأزالة كل من المتطلب الكيميائي للأوكسجين، و الفوسفات، و الأمونيا، و النترات من مياه المطروحات الصناعية في شركة غاز الشمال، في العراق. مواصفات مياه المطروحات الصناعية المستخدمة في هذه الدراسة هي 114 ملغ/لتر المتطلب الكيميائي للأوكسجين، 10.28 ملغ/لتر فوسفات، 4.123 ملغ/لتر أمونيا، 95.6 ملغ/لتر نترات، 98 وحدة عكورة. الهدف الرئيسي من هذه الدراسة هو تقييم أداء كفاءة إزالة عملية فنتون/التخثير و فحص تأثير العوامل التشغيلية مثل تراكيز فنتون (بيروكسيد الهيدروجين، الحديدوز)، و تركيز المخثر (الشب) على كفاءة الأزالة. طبقاً للنتائج المستحصلة في هذه الدراسة وجد أن أعلى كفاءة إزالة للمتطلب الكيميائي للأوكسجين، الفوسفات، الامونيا، النترات، و العكورة هي 89.43%، 72.94%، 91.065%، 90.96%، 89.85% على التوالي تحققت بأستخدام طريقة فنتون مع عملية التخثير عند افضل تركيز للمخثر (الشب). بصورة عامة يمكن ان نقول إن طريقة فنتون/التخثير تلعب دورا مهما في الحصول على نتائج كفاءة ازالة جيدة. جميع النتائج العملية تم أجراؤها بأستخدام جهاز فحص الجار عند درجة حرارة ثابتة (درجة حرارة الغرفة).

الكلمات الافتتاحية: مياه المخلفات، كفاءة الأزالة، المتطلب الكيميائي للأوكسجين، الفوسفات، الامونيا، النترات.

1. Introduction:

Water plays a vital, and important role in our daily life. Nowadays, making this natural source a fresh and available is a major environmental problematic [1]. The major concerns that can face the water quality is related with the chemical contaminants detected in both municipal and industrial wastewaters. However, large amounts of wastewater can be generated and associated with crude oil refining processes (0.4 to 0.6 times the volume of oil processed) [2] and discharged to the environment. The crude oil through the refinery processes converts into petroleum and its other useful by-products. Thoroughly, these processes large quantities of water consumed and therefore their corresponding huge quantity of wastewater produced which consists of storm water, cooling water, process water and sewage [3]. Furthermore, the wastewater produced may contain some oil due to the origin of the wastewater [4].

The effluents from petroleum refinery processes contain high quantities of toxic and recalcitrant of compounds such as Toluene, Xylene, Phosphates, Nitrates which are recognized hazardous materials discharged into the environment [5]. A variety of conventional treatment methods (mechanical and physiochemical processes) used for treating of refinery wastewater such as coagulation, floatation, water separation with further biological treatment [6]. A wide range of these toxic and polluted compounds cannot be removed by using conventional methods.

Advanced oxidation processes (AOPs) are an efficient, and appropriate can be used to destroy and remove the pollutants that cannot be removed by the conventional methods, based on the generation of hydroxyl radicals ($OH^{\cdot}$). AOPs have been successfully applied for treating various of water pollutants such as textile wastewater[7], phenolic wastewater [8], [9], pharmaceutical wastewater [10], pulp and paper industrial processes [10]. Fenton process one of the advanced oxidation process that utilized a mixture of hydrogen peroxide (H_2O_2) and ferrous ions (Fe^{+2}) to generate hydroxyl radicals according to equation (1) [11].

$$Fe^{++} + H_2O_2 \rightarrow Fe^{+++} + HO^{\cdot} + {}^{-}OH \qquad (1)$$

Fenton/Coagulation process was used for treating real industrial. Coagulation process found to be an effective in removing color, and turbidity in wastewater [12]–[15]. Where, a coagulant is a chemical which in solution furnishes ionic charges opposite to charges in colloid particle in the water, where these coagulants are neutralize repelling charges on the colloidal particles and produce flocs.

The main objectives in this study to treat real industrial wastewater using Fenton/Coagulation process and examining the effect of different operational parameters involved in Fenton/Coagulation process including Fenton's (H_2O_2, Fe^{2+}), and coagulant (Alum) doses.

2. Experimental and Methodology

2.1. Materials and Chemicals

In this study the materials and chemicals used are sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), Phosphate (PO_4), Ammonia (NH_3), Nitrite (NO_3) compounds, hydrogen peroxide (50% H_2O_2 w/v), ferrous sulfate hepta hydrate ($FeSO_4.7H_2O$), silver sulfate($AgSO_4$), mercuric sulfate ($HgSO_4$), and potassium dichromate ($K_2Cr_2O_7$), Alum (AL_2SO_4) and distilled water. All the chemicals used were of analytical grade and high purity without further purification.

2.2. Wastewater

In this study a real industrial wastewater has been used and taken from industrial water unit treatment in North Gas Company (NGC), Iraq as a case study. The main characteristics of this wastewater are listed in Table (1).

Table (1) The main characteristics of wastewater.

Item	Value
COD, (**mg/L**)	114
PH, (-)	7.11
PO_4, (**mg/L**)	10.28
NO_3, (**mg/L**)	95.6
NH_3, (**mg/L**)	4.123
Turbidity, (**NTU**)	98

2.3. Fenton/Coagulation Procedure.

All Fenton/Coagulation experimental runs were conducted by Jar test apparatus. The Jar consisting of six glass cylindrical beakers, the capacity of each beaker 2000 ml. Each beaker has a magnetic stirrer can operate at various mixing speeds ranged from 0 to 200 rpm. In each run, about 1800 ml of industrial wastewater sample was filled into each beaker with the required doses of Fenton (H_2O_2 and Fe^{2+}). The final solution volume completed to (2000 mL) by adding a few amounts of industrial wastewater. The solution was mixed and agitated vigorously for 10 min at 14 rpm which is the same of homogeneity speed in order to achieve good and efficient mixing. Then, the required doses of alum were added and agitated at the same of mixing speed (14 rpm) for 1 min. And, the solution mixing speed reduced gradually till reach to the coagulation tank speed (10 rpm). Finally, the solution mixing speed was stopped and waited about 30 min (coagulation time) for precipitation. After precipitation the samples were taken from each beakers for testing, and the beakers washed again by distilled water to remove the impurities into these beakers completely and repeated the same process for the other experiments.

Moreover, it is necessary to know that before each run the solution pH was monitored by using pH meter and adjusted by $NaOH/H_2SO_4$ till reach the required pH solution. In this study optimal conditions for each parameter alone was estimated, i.e. for estimating optimum hydrogen peroxide (H_2O_2) concentration, we added constant doses from ferrous ions (Fe^{2+}) and then start the process. The same method was repeated for the other parameters (Fe^{2+} and alum). All the experimental runs, were carried out at 25 ($\mp$2)℃, and 1 atm.

2.4. Analytical Methodologies:

After each treatment and precipitation about 200 ml sample were taken from each beaker to measure the contents of COD, PO4, NH3, NO3, and turbidity. COD measurements were measured according to standards methods by closed reflux trimetric methods using UV/Vis spectrophotometer (Thermo Scientific, Genesys 20, German). The values of COD were estimated by taking 50 ml of each sample and filled into a round bottom flask containing (5 ml concentrated H_2SO_4 and 25 ml $K_2Cr_2O_7$) and heated by using heater at 150℃ for a period 120 min. After heating, the samples were performed to cool and then titrated it with ammonium ferrous sulfate, taken to measure COD based on the the initial and the final value of COD according to equation (2). While the other parameters such as PO_4, NH_3, and NO_3 were measured by using the spectrophotometer method (Thermo Electron Corporation). The turbidity parameter was measured by using the turbidity meter depend on ASTM D-1889-18 method Nephelometric turbidity.

$$\% \ COD = \frac{COD_i - COD_t}{COD_i} * 100 \qquad (2)$$

Where: COD_i, and COD_t are the initial (114 mg/L) and the final COD, respectively.

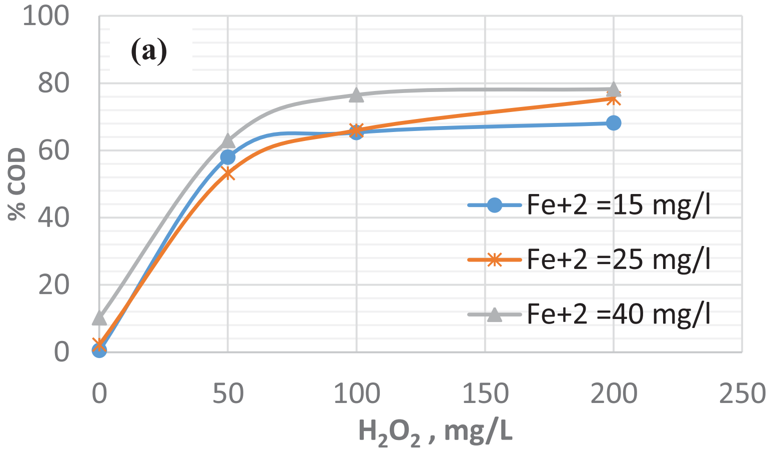
3. Results and Discussion:

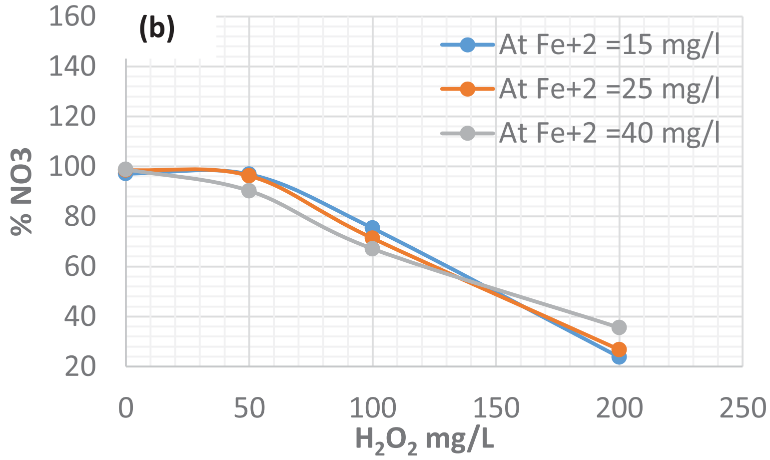
3.1. The effect of Fenton's Reagents (H_2O_2, Fe^{2+}) on the Removal efficiency:

The initial concentrations of Fenton's process are very important and plays a critical role in the degradation and cost process. In this work the effect of Fenton doses (H_2O_2, Fe^{2+}) on the removal efficiency of COD, NO3, and NH3 from wastewater has been studied and presented in Figure (1) (a, b, c). Figure (1a), presents the effect of Fenton's doses on the COD removal efficiency. Figure (1a) shows an increasing in the initial concentration of H2O2 from (50 to 200 mg/L) at constant initial concentration of Ferrous ions (Fe^{2+}) (40 mg/L) the removal efficiency could also increase from 62.84 to 78.24 %, respectively. On the other hand, the behavior of COD degradation process can be seen by varying the other initial concentrations of hydrogen peroxide with other constant doses of Ferrous ions (Fe^{2+}) (15 and 25 mg/L) but with lower efficiency. The high removal efficiency of COD with increasing the initial concentrations of hydrogen peroxide can be explained due to the increasing of hydroxyl radicals generated, But, it is very important to take into account further amounts of initial concentrations of H_2O_2 can act a scavenger for the hydroxyl radicals generated and this decreases the COD removal efficiency[16].

These results obtained are agree with the results obtained by other researchers [17] and [16]. It is important to know these experiments were performed by varying the initial concentration of hydrogen peroxide with constant dose of initial concentration of Ferrous ions. The increasing of Ferrous ions causes increasing of the production rate of hydroxyl radicals, which leaded to a higher effluent mineralization level. Taking into account, the excess amount of Ferrous irons could have a radical scavenger effect. It can be concluded from Figure (1a) that 78.24% COD removal efficiency was achieved at 200 and 49 mg/L of H_2O_2 and $Fe^{2+,}$ respectively.

On the other hand, Figure (1b) and Figure (1c), show the effect of Fenton's reagents on the removal efficiency of Nitrate (NO_3) and ammonia (NH_3), respectively. Figure (1b) shows that that removal efficiency of NO_3 decreased from 96.27 % to 26.75% with an increasing the initial concentrations of hydrogen peroxide from 50 to 200 mg/L, respectively at constant dose 25 mg/L of Ferrous ions. Figure (1c) shows the same behavior of NH_3 removal efficiency. It can be concluded from Figure (1) (a, b) Fenton's reagents are negative response on the NO_3 and NH_3 removal efficiency. Furthermore, Fenton's reagents are unaffected on the PO_4 and turbidity removal efficiency, data are not shown.





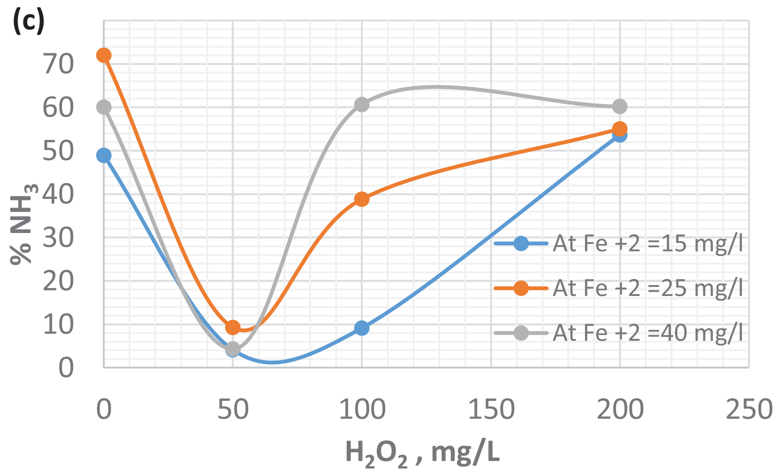
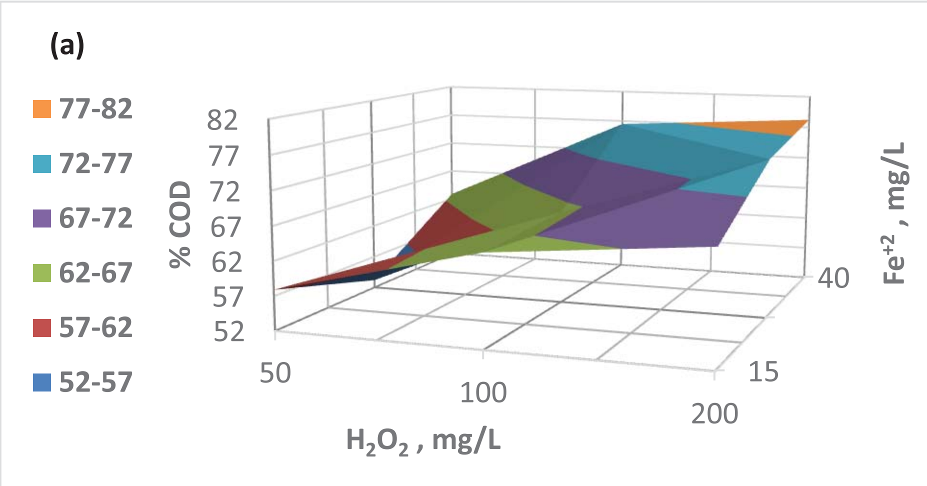
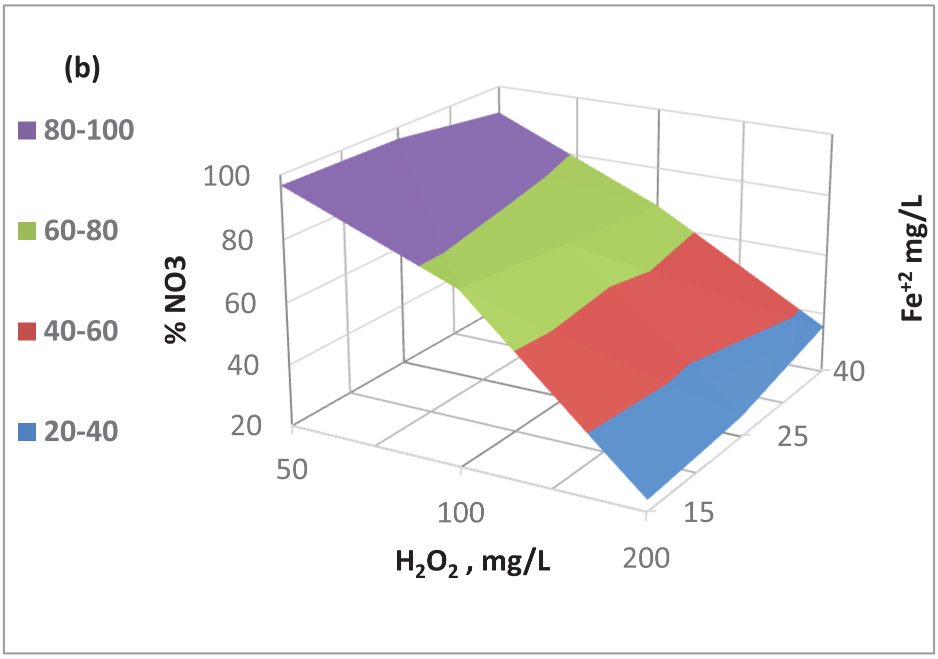


Fig. (1) The effect of initial concentration of hydrogen peroxide (H_2O_2) and ferrous ions (Fe^{2+}) on the removal efficiency of: (a) % COD (b) % NO_3 (c) % NH_3.

It showed that an increasing in initial concentrations of each parameter can effects on the COD, ammonia, and nitrite removal efficiency. H_2O_2/ Fe^{2+} were a critical parameter for improving the efficiency of the Fenton's process. The effect of mole ratio on COD, NO_3, NH_3 removal efficiency has been studied by changes the two initial concentrations of H_2O_2 and Fe^{2+} simultaneously as presented in Figure (2) (a, b, c), respectively. The results obtained in Figure (2a) shows the effect of removal efficiency of COD increase gradually and the best removal efficiency was when the molar ratio (2.5), by increasing the concentration of Fe^{+2} continuously. Further, increasing in mole ratio more than this limit the behavior of removal efficiency decreased due to the activity of hydroxyl radicals in oxidizing the organic substances decreased. These results are match with best value of removal ratio according to the most researches such as they mentioned the best molar ratios between (1-10), it depends on the quantity of pollutant in the water and concentration of the pollutant[17][16]. Moreover, lower efficiency in NO_3 and NH_3 removal efficiency with increasing the mole ratio, data presented in Figure (2) (b, c). The higher doses of H_2O_2 result more hydroxyl radicals, which, can be improved the COD removal efficiency. Also, pointed at that optimal molar H_2O_2 to Fe^{2+} ratio of 3.29 improved the biodegradability of mature landfill leachates. Moreover, it is reported that a much smaller optimal molar ratio of 1.9-3.7 to achieve the maximal degradation efficiency.

The researchers found the best molar ratio equal 4. All these findings indicated that the optimal H_2O_2/Fe^{2+} ratio varied highly with type of waste to be oxidized.





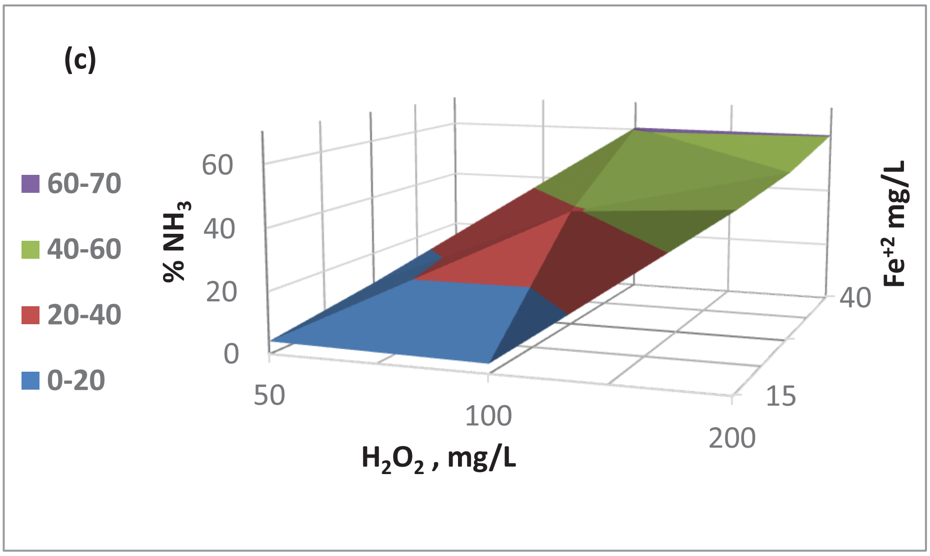
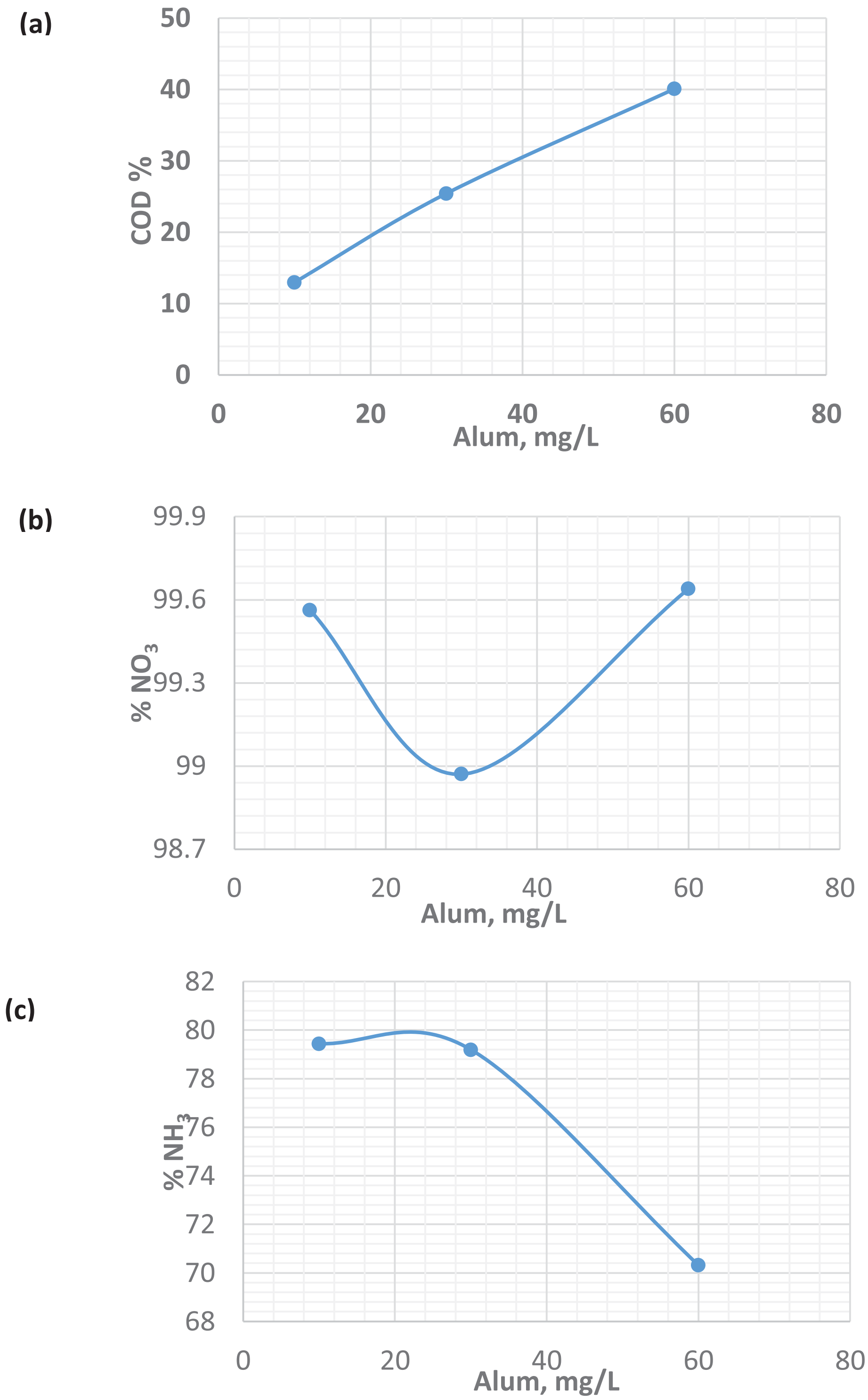
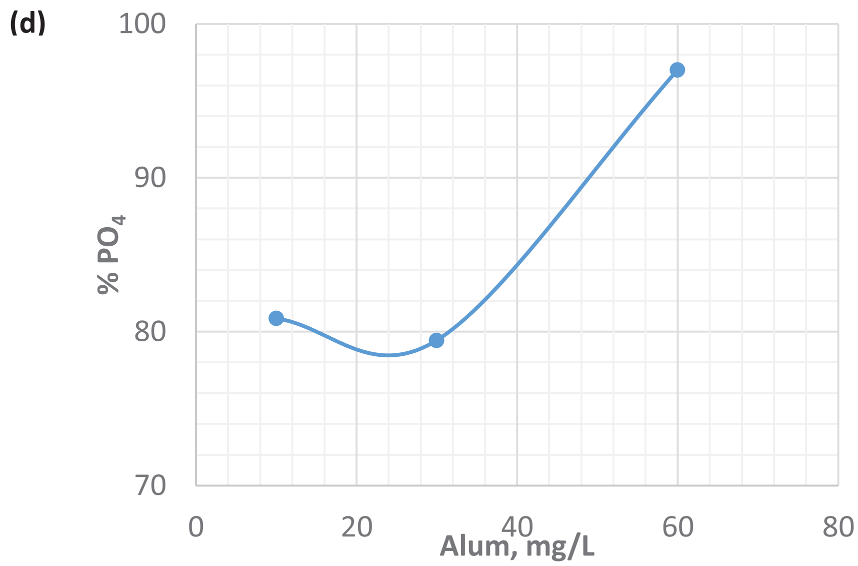


Fig. (2) The effect of mole ratio on: (a) % COD removal efficiency (b) % NO_3 removal efficiency (c) % NH_3 removal efficiency.

3.2. The Effect of Alum on Removal Efficiency

The effect of Alum (AL_2SO_4) coagulant dose on the COD, NO_3, NH_3, PO_4, and Turbidity removal efficiency from wastewater has been studied at different doses of Alum (10, 30, 60 mg/L) as shown in Figure (3) (a, b, c, d). As can be seen in in Figure (3a) the COD removal efficiency increased from 12.90 to 40.09 % (maximum value) with an increasing the dose of Alum from 10 to 60 mg/L, respectively. It can be concluded from Figure (3a) that maximum removal efficiency 40.09 % was achieved at optimum Alum coagulant 60 mg/L, where sharply increasing in removal efficiency is proportionally with an increasing the initial concentration of Alum. These results were in agreement and the same behavior investigated in color removal from landfill leachate by using coagulation/flocculation process[18], [19]. On the other hand, the effect of Alum dose on the NH_3, PO_4, Turbidity removal efficiency has been studied and presented in Figure (3) (b, c, d, e), respectively. Figure (3e) shows the impact of Alum on the removal of Turbidity from wastewater, it can be seen from these presented results the Turbidity removal efficiency increased from 79.18 to 97.5 % when the Alum coagulant dose increased from 10 to 60 mg/L. Finally, it can say that Alum coagulant achieved high removal efficiency in removing NO_3, NH_3, PO_4, Turbidity but lower COD removal.


(a)
50
40
30
20
10
0
COD %
0
20
40
60
80
Alum, mg/L
(b)
99.9
99.6
99.3
99
98.7
% NO3
0
20
40
60
80
Alum, mg/L
(c)
82
80
78
76
74
72
70
68
% NH3
0
20
40
60
80
Alum, mg/L



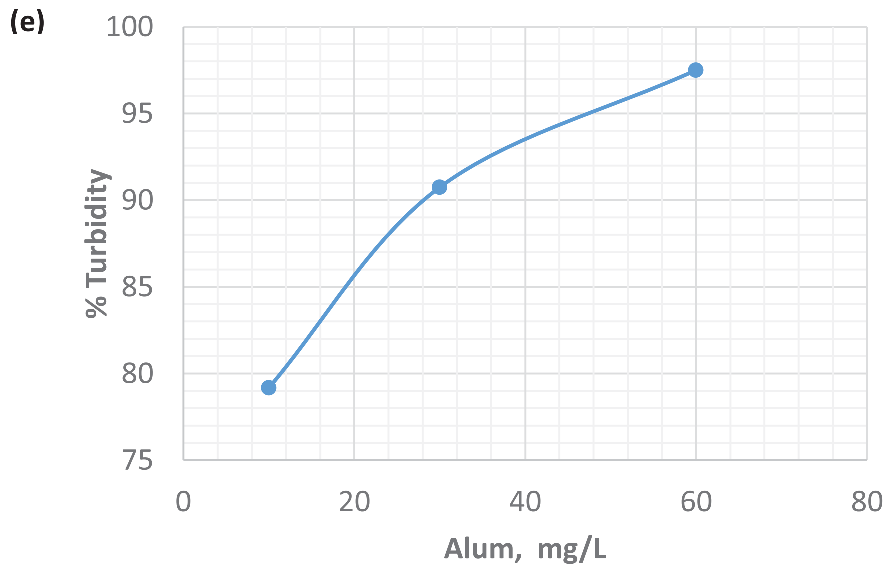
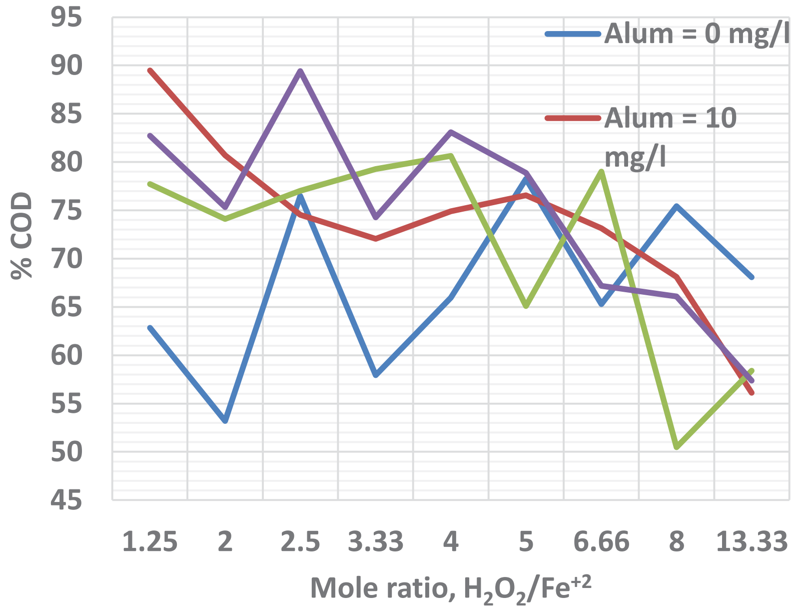


Fig. (3) The effect of Alum on: (a) % COD removal efficiency (b) % NO_3 removal efficiency (c) % NH_3 removal efficiency (d) % PO_4 removal efficiency (e) % Turbidity removal efficiency.

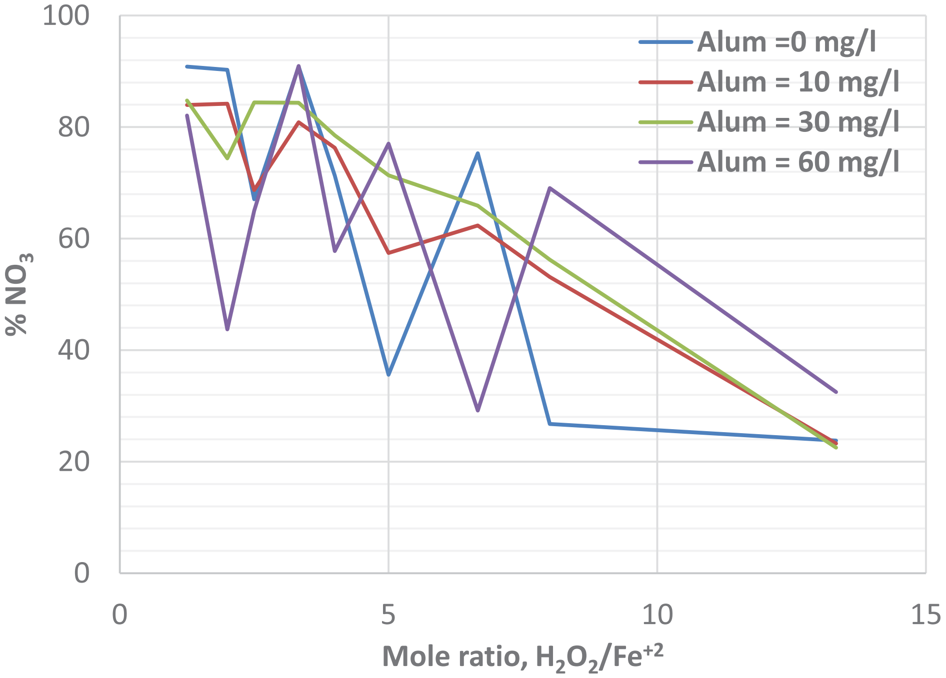
3.1.3. The Effect of Fenton/Coagulation on the Removal Efficiency.

COD, NO_3, NH_3, PO_4, and Turbidity removal efficiency depends on the operating conditions mentioned in Figure (1-3) (a, b, c, d, e) where each figure showed the effect of each parameter on the COD, NO_3, NH_3, PO_4, and Turbidity removal efficiency. However, and in order to increase the removal efficiency, various parameters were mixed together to obtain higher values of removal efficiency. In this study Fenton (H_2O_2, Fe^{2+}) combined with coagulation (AL_2SO_4) process was used to remove COD, NO_3, NH_3, PO_4, and Turbidity as shown in Figure (4) (a, b, c, d, e). Data presented in these figures as Fenton process was expressed as mole ratio was varied from (1.25 to 13.33 mole ratio) with constant doses of each Alum coagulants (0 – 60 mg/L AL_2SO_4). Figure (4a) shows that the COD removal efficiency fluctuates with increasing the mole ratio where maximum removal efficiency was obtained at 2.5 mole ratio and 60 mg/L of Alum coagulant. The same behavior could be obtained at different other doses of Alum coagulants but in lower efficiency. The effect of Fenton/Coagulation process on the NO_3, NH_3, PO_4, and Turbidity removal efficiency presented in the Figure (4) (b, c, d, e). The results obtained in these figures revealed that the COD, NO_3, NH_3, PO_4, and Turbidity removal efficiency enhanced by using combined Fenton/Coagulation process was enhanced and increased highly compared with each separated process.

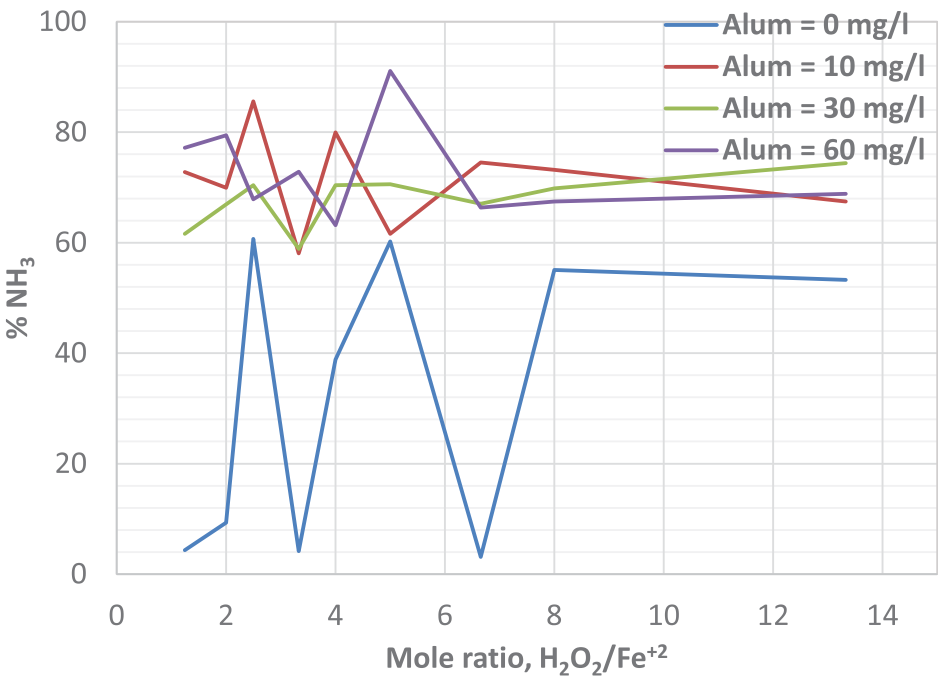
(a)

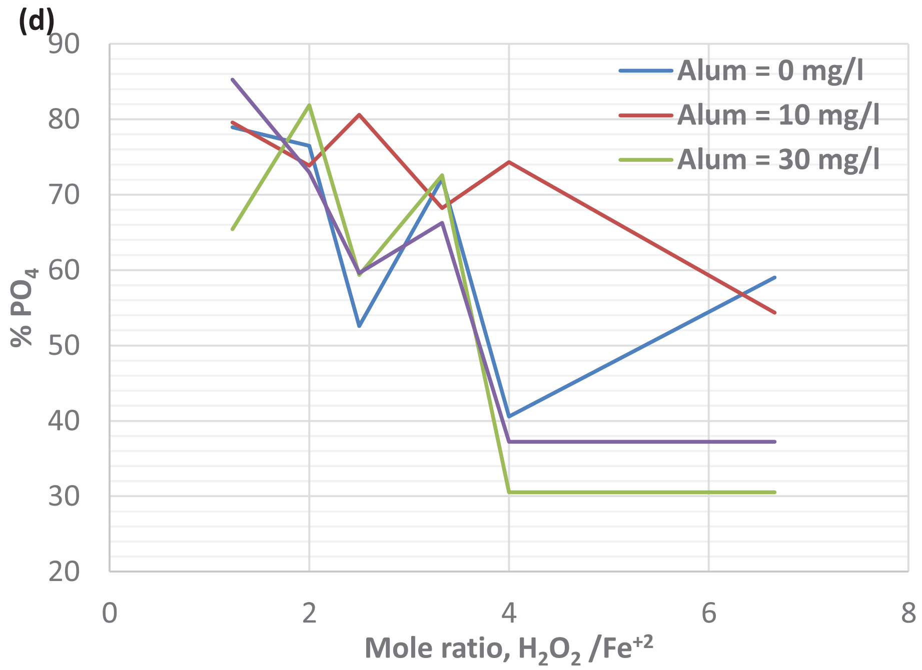


(b)


100
80
60
40
20
0
% NO_3
0
5
10
15
Mole ratio, H_2O_2/Fe^{+2}
Alum =0 mg/l
Alum = 10 mg/l
Alum = 30 mg/l
Alum = 60 mg/l

(c)


100
80
60
40
20
0
% NH_3
0
2
4
6
8
10
12
14
Mole ratio, H_2O_2/Fe^{+2}
Alum = 0 mg/l
Alum = 10 mg/l
Alum = 30 mg/l
Alum = 60 mg/l



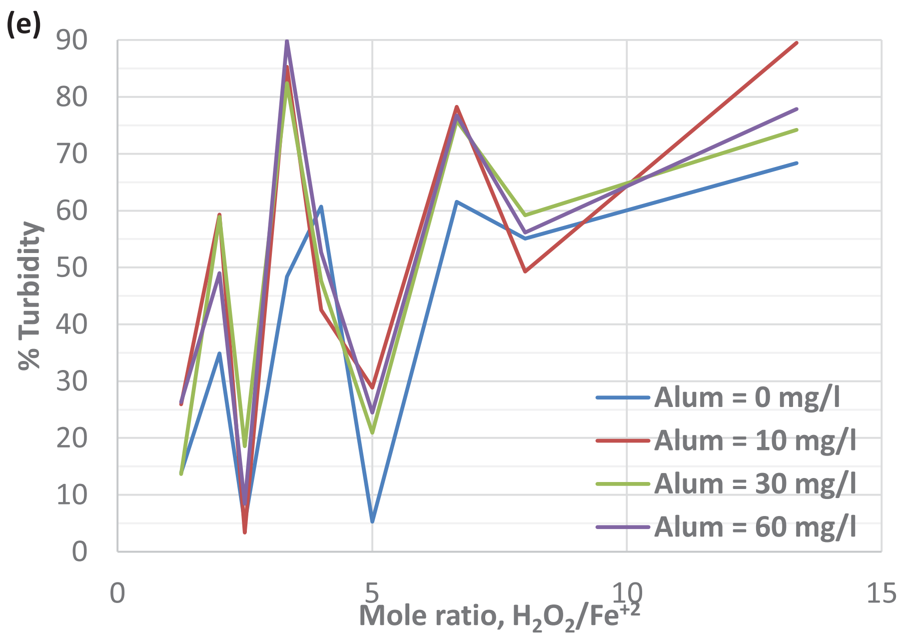


Fig. (4) The effect of Fenton/Coagulation on: (a) % COD removal efficiency (b) % NO_3 removal efficiency (c) % NH_3 removal efficiency (d) % PO_4 removal efficiency (e) % Turbidity removal efficiency.

4. Conclusions

In this study Fenton/Coagulation process as Advanced oxidation processes (AOPs) has been used to treat real industrial wastewater, and based on the experimental results obtained in this study it can be concluded that:

1- It is very difficult to treat wastewaters discharged from petroleum refineries using conventional treatment methods.
2- AOPs are found to be efficient process for the degradation of refractory compounds. Fenton combined with coagulation process as AOPs was used for this issue.
3- Fenton/Coagulation was successfully applied in terms of COD, PO4, NH3, NO3, turbidity removal efficiency to increase the biodegradability of real refinery wastewater.
4- As a results of this study the maximum removal efficiency of COD, PO4, NH3, NO3, turbidity was 89.43%, 72.94 %, 91.065%, 90.96%, 89.85% achieved at optimum operating conditions are 1.23 mole ratio and 60 mg/L of Alum.

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