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Investigation of amoxicillin removal from aqueous solution by Fenton and photocatalytic oxidation processes

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Abstract

This study investigated the removal of amoxicillin (AMO) antibiotic and chemical oxygen demand (COD) by Fenton (Fe^{2+}/H_2O_2) and photocatalytic (UV-A/TiO_2) oxidation processes in aqueous solutions. In experiments, pH, antibiotic, Fe^{2+}, H_2O_2, TiO_2 concentrations and reaction time parameters were examined. In the Fenton process, the removal efficiencies of initial AMO and COD concentrations were 83% and 66%, respectively. The Fe^{2+}/H_2O_2 molar ratio was determined as 1/15. In the photocatalytic process, AMO and COD removal efficiencies were 62% and 52%, respectively. The results indicate that these two processes may enhance the rate of AMO removal in polluted water and could be used as a preliminary treatment or as an alternative to existing treatment systems.

Keywords: Amoxicillin; antibiotic removal; chemical oxygen demand (COD); Fenton oxidation; photocatalytic oxidation.

1. Introduction

The most common usage of antibiotics is to treat bacterial infections in humans and animals. Other uses are in hospitals, food and pharmaceutical industries, aquaculture cultivation, and in scientific research (Savcı, 2016). Antibiotics are complex and hardy biodegradable organic compounds, having been a cause for concern due to their toxic effects on the human body and natural life (Kummerer, 2009b; Luo *et al.*, 2014). By oral application of antibiotics, only 10-20% is metabolized, while the remaining 80-90% is expelled from the body directly and indirectly into the ecosystem (Hernando *et al.*, 2006).

The presence of antibiotics and their residues in various water sources can lead to the occurrence of antibiotic-resistant bacteria. These compounds damage or destroy natural aquatic micro-organisms required for biological wastewater treatment. Existing conventional biological wastewater treatment plants are not efficient enough to remove pharmaceuticals (Rizzo *et al.*, 2013; Matongo *et al.*, 2015).

Advanced oxidation processes (AOPs) are an efficient and environmentally-friendly method which are used to generate hydroxyl radicals ($OH^\bullet$, E=2.8 V) in ambient conditions. Then $OH^\bullet$ oxidizes, emerging contaminants to H_2O and CO_2 end products.

Many studies discuss the removal of antibiotics from different water sources (see Trovó *et al.*, 2011; Li *et al.*, 2012; Dimitrakopoulou *et al.*, 2012; Oros-Ruiz, 2013; Sheydaei *et al.*, 2014; Santos, 2015; Dehghani *et al.*, 2015). Fenton and photocatalytic processes are highly applied methods of AOPs. Fenton technology is based on an electron transfer between ferrous ion (Fe^{2+}) as a catalyst and H_2O_2 as an oxidant in acidic conditions (Dehghani *et al.*, 2015). In photocatalytic process, ultraviolet light (UV) is used to excite the electrons with a semiconductor catalyst (such as TiO_2, ZnO etc.), having valance and conduction energy bands, based on the action of positively charged holes. These holes react with water on the surface of a photocatalyst and generate oxidants such as $OH^\bullet$ and a superoxide ($O_2^{-\bullet}$) radical (Meeroff *et al.*, 2012).

Penicillins, as a classification, are the most widely consumed antibiotics in Turkey and other countries (Turkdogan & Yetilmezsoy, 2009; WHO,2011-2014). AMO belongs to the penicillin, used as a β-lactam antibiotic for human and animal health for the prevention of bacterial infections caused by gram-negative and gram-positive organisms such as various allergies, infections (Jung *et al.*, 2012; Napoleao *et al.*, 2015).

This study evaluated the advanced oxidation of antibiotic amoxicillin in aqueous solution by Fenton and photocatalytic processes in terms of amoxicillin and COD removal and to see if these systems could be a pertinent way to eliminate the drug in wastewater treatment plants. The effects of pH, initial antibiotic, Fe^{2+}, H_2O_2, and TiO_2 concentrations, reaction time, and the Fe^{2+}/H_2O_2 molar ratio on the degradation of AMO and COD were investigated. Optimum values of these operating parameters were determined. The Fenton and photocatalyitc reagent dosages were specified based on the initial COD value for the efficient treatment because COD is a specified oxidant reacting with the sample under these conditions.

2. Materials and methods

2.1. Chemicals

Amoxicillin ($C_{16}H_{19}N_3O_5S$ 365.4 g mol^{-1}) was purchased from Sigma-Aldrich and used as received. The chemical structure of AMO is given in Figure 1.

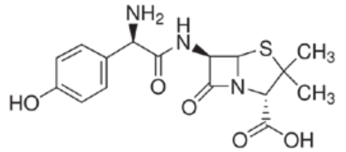


Fig. 1. Chemical structure of AMO

Other chemicals were obtained from Merck. COD test kits (0-1500 mg O_2 /L) were supplied from Hach Lange, USA.

2.2. Experimental procedure

A stock solution of AMO (500 mg L^{-1}) was prepared in ultrapure water and stored at 4° C. The experiments were performed at different pHs 2-7 for the Fe^{2+}/H_2O_2 process and 3, 7, 11 for the UV-A/TiO_2 process, different concentrations of Fe^{2+} (28-280 mg L^{-1}), H_2O_2 (170-1700 mg L^{-1}), AMO (10-100 mg L^{-1}), TiO_2 (200-1000 mg L^{-1}), and for time ranges from 5 to 60 minutes.

For the Fenton experiments, synthetic wastewater samples contained in 100 mL a dark Pyrex reactor were stirred at 120 rpm with a magnetic bar at 25° C for 30 min (Figure 2). To prevent the interferences of H_2O_2 on COD removal, 1 M of MnO_2 was also added. After sedimentation, 15 mL of the sample were centrifuged (6000 rpm, 15 min) and filtered using a membrane filter (0.45 µm) to measure of AMO and COD amounts.

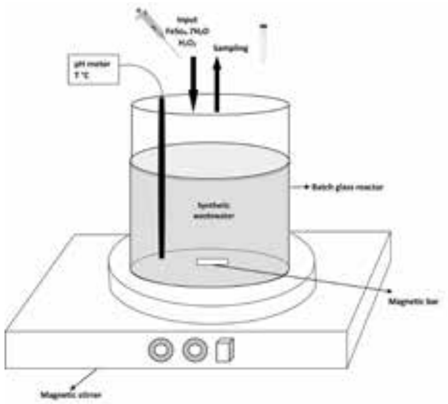


Fig. 2. Fenton process setup

A volume of 1000 mL of the sample was introduced into 2000 mL of water in a jacketed pyrex glass UV reactor. The sample was then exposed to a UV-A black light bulb (T5 6 W, 24 cm, 315- 400 nm) with an irradiation time of 30 min at 150 rpm (Figure 3). During the experiments, the temperature (25° C) of the reactor was controlled by using a thermometer. The samples were analyzed at the same conditions as the Fenton experiments to determine the removal of AMO and COD.

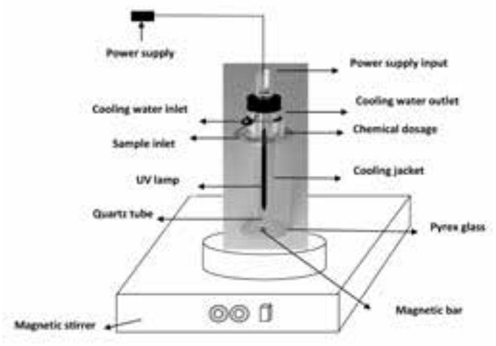


Fig. 3. Photocatalytic process setup

2.3. Analytical methods

A UV-visible spectrophotometer (T90+ UV-visible, PG Instruments) was used and the absorbance of AMO was determined as 230 nm. COD of all the samples was measured by using a Hach DR/2010 spectrophotometer according to standard methods (APHA, AWWA, WPCF, 2005). pH was measured by a WTW Multi 340i pH meter.

The experiments were performed in IKA-Werke RT-5 Power and Yellow Line magnetic stirrers. For the centrifuge processing of samples, Hettich EBA20 was used. The residual H_2O_2 was determined by the iodometric titration method (Jeffery *et al.*, 1989; Kang & Chang, 1997).

3. Results and discussion

3.1. Effect of pH

In Fenton experiments, the effect of pH shows that as pH increased from 2 to 7, the removal of AMO and COD decreased (Figure 4). The data show that the maximum reduction rates of AMO and COD were achieved at pH 3 for more than 77% and 85% at 30 min.

pH is very important for oxidation influencing the production of $OH^{\bullet}$, the substrate and oxidant activities, the formation of iron, and the decomposition of H_2O_2. The removal efficiency of AMO and COD reduce for lower and higher pHs due to the ionization states of the substrate and the catalyst. This is because Fe^{2+} is tended a better dissolution at acidic pHs (2-5) in water (Kuo, 1992).

H_2O_2 has greater stability due to the formation of an oxonium ion ($H_3O_2^{+}$) reducing the regeneration of Fe^{2+} at low pHs (Kwon *et al.*, 1999). Also, the pH value below optimal can inhibit the reaction of Fe^{3+} ion and H_2O_2 each other (Pignatello, 1992). Some researchers also suggest that the production of the $OH^{\bullet}$ is scavenged in the presence of a high concentration of H^+ (Lucas & Peres, 2006).

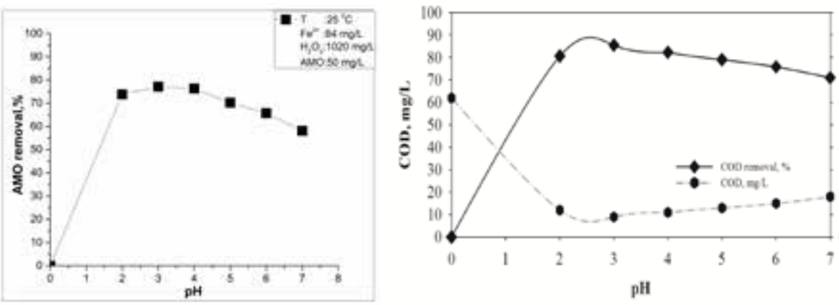
A pH value above optimal can affect H_2O_2 decomposition range due to the absence of H+ decreasing the generation of OH•. H_2O_2 is more unstable reduced ability to oxidation at higher pH (Walling, 1975). Besides, the Fe^{2+} catalyst loses its activity with the formation of ferric oxyhydroxide ($Fe(OH)_3$), precipitating and scavenging generated OH• under alkaline conditions (CO_3^- and HCO_3^-) (Buxton *et al.*, 1988). In the UV-A/TiO_2 process, a pH of 7 was optimal for AMO and COD removal for more than 90% at 30 min. AMO and COD removal did not change considerably for pH 7 and higher (Figure 5). Photocatalytic oxidation efficiency increases in neutral and alkaline pH values. pH affects the electrostatic load of the TiO_2 surface at photocatalytic reactions.

TiO_2 particles are shown to be granule in an aqueous medium due to the pH and ionic strength (French *et al.*, 2009). It is known that agglomeration of TiO_2 particles is lower at acidic conditions than alkaline ones (Li *et al.*, 2010).

3.2. Effect of Fe^{2+} concentration

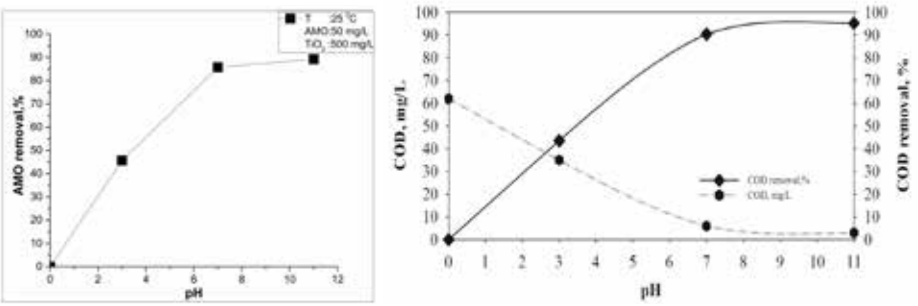
The removal efficiency of AMO and COD increased from 68.4% to 82.06% as the concentration of Fe^{2+} increased from 28 to 112 mg L^{-1}. It did not change significantly above this value.

On the other hand, the minimum COD removal efficiency was 82.26% at 28 mg L^{-1} Fe^{2+}, and the mineralization of COD was completely achieved at 280 mg L^{-1} Fe^{2+} dosage at 30 min. As shown in Figure 6, the oxidation of AMO and COD increased with an increasing Fe^{2+} value. As the dosage of Fe^{2+} increases, the redox reaction and coagulation become complete. When Fe^{2+} is above 112 mg L^{-1}, it is consumed rapidly or regenerated slowly and then oxidized to Fe^{3+} at the end of the reaction (Kavitha & Palanivelu, 2004). Moreover, Fe^{3+} may react with OH• and precipitate as $Fe(OH)_3$ above 112 mgL^1. Therefore, 112 mg L^1 of Fe^{2+} was reported as the optimum concentration.



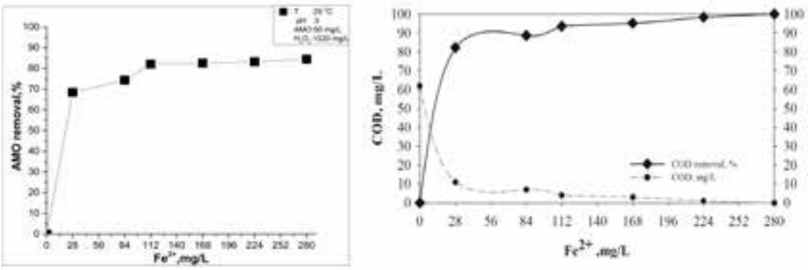
(a) **(b)**

Fig. 4. Effect of pH on (a) AMO and (b) COD removal in Fe^{2+}/H_2O_2 process



(a) **(b)**

Fig. 5. Effect of pH on (a) AMO and (b) COD removal in UV-A/TiO_2 process



(a) **(b)**

Fig. 6. Effect of Fe^{2+} on (a) AMO and (b) COD removal in Fe^{2+}/H_2O_2 process

3.3. Effect of H_2O_2 concentration and residual H_2O_2

The effect of H_2O_2 concentration on AMO and COD removal is shown in Figure 7. The best removal of AMO and COD concentration was observed at 1020 mg L^{-1} of H_2O_2 (78.55%). AMO removal did not change significantly for 1020 mg L^{-1} H_2O_2 dosage and higher. At higher peroxide concentrations, the excess H_2O_2 can act as a scavenger for $OH^\bullet$, forming hydroperoxyl ($^\bullet HO_2$), which is a free radical that has a lower oxidation capacity than $OH^\bullet$ (Arslan-Alaton & Dogruel, 2004; Tony *et al.*, 2009a).

A higher dosage of H_2O_2 increases in percent degradation. However, some organic compounds, which are hard to oxidize, reduce the performance of H_2O_2 (Kang & Hwang, 2000; Lin & Lo, 1997). Furthermore, residual H_2O_2 in water can be toxic to microorganisms, and require a different water treatment process to deal with biological debris (Barbusiński & Filipek, 2003; Wang *et al.*, 2017) Therefore, the additional concentration of H_2O_2 into a sample reduce the system's efficiency. A lower dosage of H_2O_2 does not product sufficient $OH^\bullet$ radicals to cause full decomposition. For this reason 1020 mg L^{-1} H_2O_2 were accepted as the optimal value. After Fenton oxidation, the residual concentration of H_2O_2 interferes with COD analysis in the treated water, which causes the COD value to increase. Table 1 shows the change in the amount of H_2O_2 during the reaction. It was observed that an Fe^{2+} ion reacted with 85% H_2O_2 after approximately 20 min.

Table 1. The results of the residual hydrogen peroxide concentration of AMO for optimum conditions

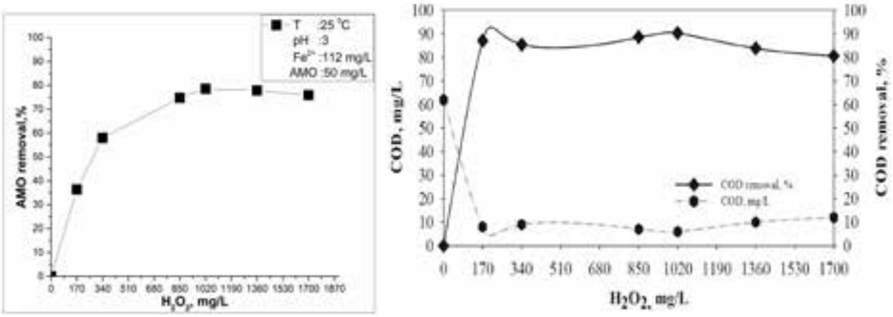
Parameter	Initial H_2O_2 (mg L^{-1})	Residual H_2O_2 (mg L^{-1})
pH, 3	1020	102
Fe^{2+},112 mg L^{-1}	1020	153
H_2O_2, 1020 mg L^{-1}	1020	289
Conc.,100 mg L^{-1}	1020	85
Time, 20 min	1020	153

3.4. Effect of TiO_2 concentration

Data regarding the effect of the TiO_2 concentration is given in Figure 8. The results reveal that as the concentration of TiO_2 increased from 200 to 1000 mg L^{-1}, there was a decrease in the removal of AMO and COD. Hence, the optimum value of TiO_2 was determined to be 200 mg L^{-1}. This is advantageous in terms of cost for the use of chemicals.

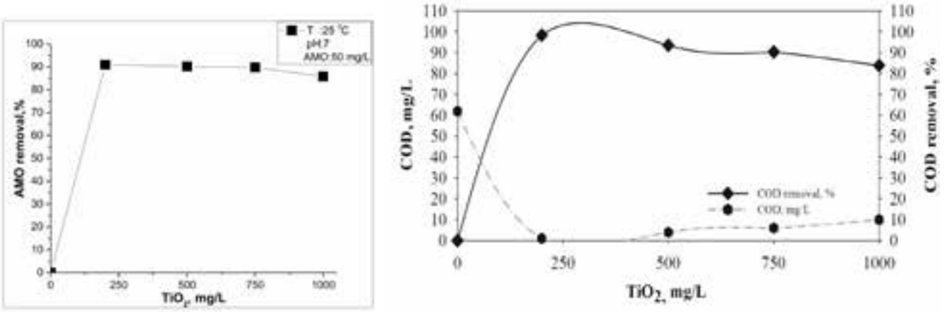
Dimitrakopoulou *et al.* (2012) reported that Degussa P25, a catalyst consisting mainly of anatase and rutile, could achieve total degradation of AMO after 25 min. The researchers also showed that 93% could be mineralized after 90 min of reaction.

Elmolla & Chaudhuri (2010b) found 42% AMO degraded after 300 min with UV-A irradiation and at 500 mg L^{-1} pure anatase TiO_2. However, in this study, the best efficiencies of AMO degradation and COD removal were 90.96% and 98.32% using 200 mg L^{-1}



(a) (b)

Fig. 7. Effect of H_2O_2 on (a) AMO and (b) COD removal in Fe^{2+}/H_2O_2 process



(a) (b)

Fig. 8. Effect of TiO2 on (a) AMO and (b) COD removal in UV-A/TiO_2 process

pure anatase TiO_2 at 30 min. This is completely related to the experimental design factors, such as antibiotic and TiO_2 concentrations, reaction times, mixing speeds, type of catalyst, reactor geometry, and light source.

3.5. Effect of antibiotic concentration

The initial concentration of AMO has an important role due to the performance limitation of Fe^{2+}/H_2O_2 and UV-A/TiO_2 processes. Figure 9 shows that AMO degradation efficiency improved from 52.92% to 82.01% with the increase of AMO concentration, and the COD removal efficiency also increased from 23.08% to 79.67% from 10 to 100 mg L^{-1} within 30 min. It was shown that the removal efficiency of AMO and COD increased with the increase of initial concentrations by the Fenton process. Thus, high doses of H_2O_2 and Fe^{2+} may be required to degrade AMO effectively.

According to Figure 10, the efficiencies of AMO degradation and COD removal slowly decreased while the initial AMO concentration increased from 10 to 100 mgL^{-1} in the photocatalytic process. The increase of antibiotic concentration also increases the amount of antibiotic adsorbed to the photocatalyst surface. However, the photoactive regions on the catalyst surface are reduced and finally the rate of degradation of antibiotics is decreased (San *et al*., 2002).

3.6. Effect of initial COD

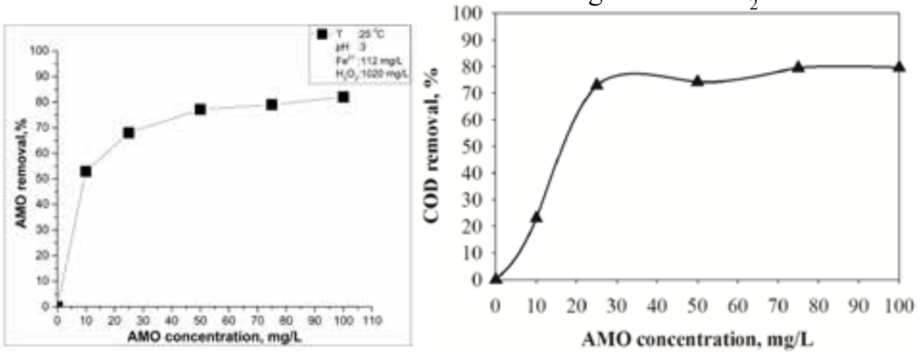
The initial COD is one major factors for determining the reagent dosages for the efficiency of Fenton and photocatalytic processes. For this reason, in this study the effect of COD removal was also studied for both processes. The experiments showed that although AMO degradation was quite efficient, the removal efficiency of COD was lower than AMO. This may be due to the solution of $OH^\bullet$ at high Fe^{2+}, H_2O_2 and TiO_2 doses and/or formed metabolites.

3.7. Effect of reaction time

For the 60 min reaction time, the degradation of the initial AMO concentration and COD removal with two processes are given in Figure 11(a) and (b). In the Fenton process, the reduction rate of AMO increased in 20 min (82.67%) when AMO and H_2O_2 concentrations were high, which was due to $OH^\bullet$ destruction.

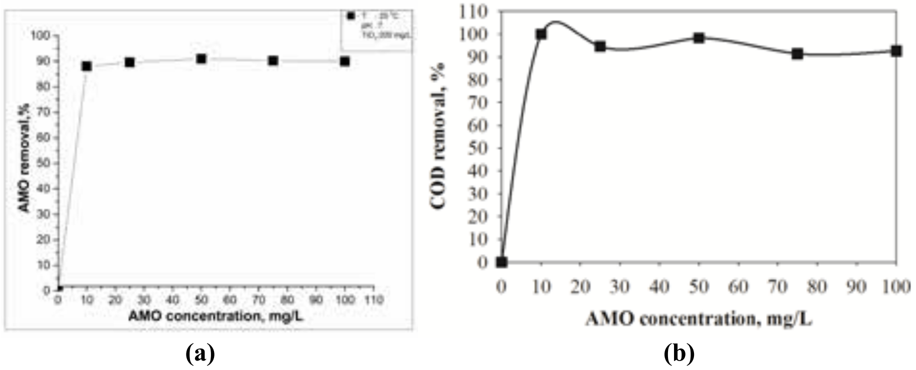
On the other hand, the removal efficiency did not significantly increase from 20 to 60 min because of low AMO and H_2O_2 concentrations. COD removal also increased slowly during the 60 min reaction time. Belal *et al.* (2000) reported that the hydrolysis of penicillins increases with time.

According to the photocatalytic results, AMO and COD removal efficiencies slightly went up due to the fact that having higher AMO concentrations can cover the active regions on a TiO_2 surface. In addition, the formation of $OH^\bullet$



(a) **(b)**

Fig. 9. Effect of AMO concentration on (a) AMO and (b) COD removal in Fe^{2+}/H_2O_2 process



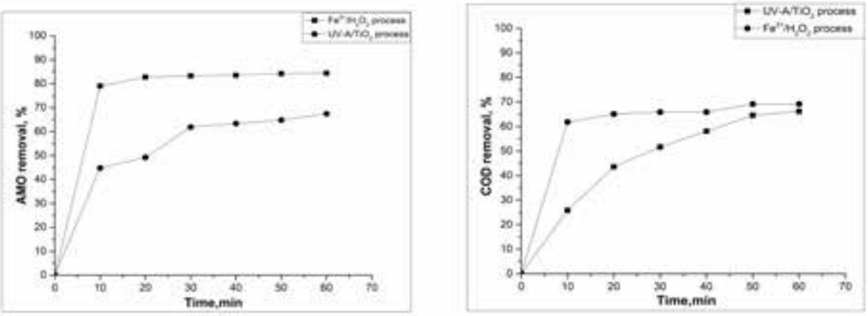
(a) **(b)**

Fig. 10. Effect of AMO concentration on (a) AMO and (b) COD removal in UV-A/TiO_2 process

can decrease so the reduction of AMO may be degraded.

Table 2. Optimum values of AMO for fenton and photocatalytic oxidation processes

Parameter	Fe^{2+}/H_2O_2	UV-A/TiO_2
pH	3	7
Fe^{2+}, mg L^{-1}	112	-
H_2O_2, mg L^{-1}	1020	-
TiO_2, mg L^{-1}	-	200
Con., mg L^{-1}	100	50
Time, min	20	30



(a) (b)

Fig. 11. Effect of reaction time on (a) AMO and (b) COD removal

Finally, it was observed that the Fenton process may efficiently separate organics of AMO than a photocatalytic process can in a short time.

COD removal is also efficient by Fe^{2+}/H_2O_2. This occurs because some intermediate compounds are resistant to mineralization byproducts. The optimal values of both processes are summarized in Table 2.

4. Conclusions

The study results revealed that optimum values vary with the type of oxidation process employed. The best removal of AMO and COD was 83% and 66%, respectively, in acidic pHs by the Fenton process. The next best was 62% and 52% respectively, in neutral pHs by the photocatalytic process. The efficiency of the Fenton process increases with augmented concentrations of AMO, which depends on H_2O_2 and Fe^{2+} doses for maximum COD removal. Thus, the molar ratio of Fe^{2+} and H_2O_2 is also significant in order to avoid scavenging effects, increase COD removal, reduce final sludge volume, and in terms of overall cost. Therefore, the optimum molar ratio of Fe^{2+}/H_2O_2 was determined to be 1/15. After the Fenton reaction Fe^{3+} formed could also contribute to COD removal with coagulation and flocculation.

The efficiency of the photocatalytic process increases with rising doses of AMO and TiO_2. Advantages of using pure TiO_2 are that it is inexpensive, available, non-toxi. and photochemically stable.

The results indicated that initial AMO and COD could be effectively destroyed by both oxidation processes in a short reaction time, meaning that Fenton and photocatalytic processes could be used as a preliminary treatment or as an alternative to existing treatment systems.

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تحري انتزاع الأموكسيسيلين من محلول مائي بواسطة عمليات فينتون والأكسدة بالتحفيز الضوئي

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الملخص

هدفت هذه الدراسة إلى تحري انتزاع مضاد الالتهاب أموكسيسيلين (AMO) ومطلوبية الأوكسجين الكيميائية (COD) بواسطة عمليات فينتون (Fe^{2+}/H_2O_2) والأكسدة بالمحفز الضوئي ($UV\text{-}A/TiO_2$) في محلول مائي. تمت معاينة التراكيز ومعايير مدة التفاعل في التجارب لكل من الـ pH، مضاد الالتهاب، H_2O_2، Fe^{2+} و TiO_2. بلغت كفاءات الإزالة للتراكيز الأولية لـ AMO و COD في عملية فينتون 83% و66% على التوالي. وقد تحددت النسبة المولية لـ Fe^{2+}/H_2O_2 بـ 1/15. وبلغت كفاءات الإزالة لـ AMO و COD في عملية التحفيز الضوئي 62% و52% على التوالي. وأشارت النتائج إلى أن هاتين العمليتين قد تحسنان من نسبة إزالة الـ AMO في الماء الملوث وقد تستخدم كعلاج أولي بديلاً عن أنظمة العلاج السارية.