# Removing of Atrazine from Water using Advanced Oxidation Processes

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## ABSTRACT:

The performance of Advanced Oxidation Processes UV, Ozone ($O_3$), UV+$O_3$, $O_3$+ $H_2O_2$, UV+$H_2O_2$ were investigated in this study using a semi batch lab scale experiments for the mineralization of Atrazine which is used as a model pollutant. 1mg/L of Atrazine was investigated. The effect of pH was studied in the presence of sodium hydroxide (NaOH). UV photolysis (UV only) shows no COD reduction in treating Atrazine. The treatment with $O_3$ gives 43% of COD reduction. For $O_3$ +UV the COD reduction achieved was 45% at pH=12. Treatment with $O_3$+$H_2O_2$ gives 95% reduction in COD at $H_2O_2$ concentration of 140mg/L and pH=12. The treatment with UV+$H_2O_2$ gives 95% in COD reduction at $H_2O_2$ concentration 600 mg/L and pH=7.

**Key words: Atrazine, AOP, Ozone, Hydrogen peroxide, UV**

## 1. INTRODUCTION

Atrazine (ATZ) is a white, crystalline solid that is used as a synthetic herbicide to treat broadleaf weeds in agricultural and roads applications. EPA's Pesticides in ground water database indicates frequent recognitions of ATZ at concentrations above the maximum concentration level (MCL) in ground water in several states, [1] [9]. The maximum contaminant level (MCL) for ATZ in drinking water is 3 µg/L, as set by the EPA, [2] [22]. The European Union banned the use of ATZ due to its persistence in the environment [22]; [11]; [7]; [24]. ATZ is a pesticide that's commonly used as model compound in research of advanced oxidation processes, it remains hard to degrade due to the triazine ring, [23]. ATZ is used in Iraq in spite of its banned and many researchers evaluate its validity and its impact on environment. Al Naqeeb et al.,(2010), [27] and Al Chalabi and Al Jbouri (2012), [25] stated that using ATZ with yellow maize led to improve some growth analysis parameters. Al

Kutubi (2012),[26] found that the residue of the ATZ had a significant effect on density and percentage of weed control of broad bean. Ali (2013) [29], studied the degeneration effect of the injection of different dose of ATZ into the body of white mice. The results show that the fatal rates were increased. Also a degenerations and necrosis in kidney, lung, heart and liver were observed. In general the main problem that associated with treatment of water polluted with pesticides that the traditional treatment methods such as secondary biodegradation, cannot appreciably remove these contaminants. While advanced treatment technologies such as activated carbon and reverse osmosis which produce high quality water, they only transfer and concentrate the pollutants from one phase to another, requiring further processing to render the compounds inert [1]. Advanced oxidation process (AOP), which is defined as, the process of generation of hydroxyl radical in water in sufficient amount to oxidize the pollutant, this process seems to be very promising option especially in presence of suitable infrastructure [12]. The hydroxyl radical (OH) is a powerful, non-selective chemical oxidant that react very quickly with most organic compounds, A free radicals is not an ionic species but is formed from an equal cleavage of two electron bond [21]. Table 1 shows relative oxidation power of hydroxyl radical and other oxidants taking chlorine as reference.

**Table 1. Relative oxidation power of hydroxyl radical [15].**

| Oxidizing species | Relative oxidation power |
|---|---|
| Chlorine | 1.00 |
| Hypochlorous acid | 1.10 |
| Permanganate | 1.24 |
| Hydrogen peroxide | 1.31 |
| Atomic oxygen | 1.78 |
| Hydroxyl radicals | 2.05 |
| Ozone | 1.52 |
| Positively charged hole on titanium dioxide, $TiO_2^+$ | 2.35 |

Most of AOP systems use combination of $H_2O_2$ , $O_3$, catalysts like ($Fe^{2+}$, $Mn^{2+}$, $Ni^{2+}$, $Co^{2+}$, $Cd^{2+}$, $Cu^{2+}$, $Ag^+$, $Cr^{2+}$, $Zn^{2+}$), photocatalysis and irradiation (ultraviolet (UV), ultrasound (US) or electron beam)

## 1.1 Ozonation

As early as 1893, $O_3$ was used as a disinfection agent for drinking water in Europe. By 1986, over 1000 potable water plant using $O_3$was reported. Also US began to use the $O_3$ process to control odor and taste in wastewater at Philadelphia. Since then, much research has been done on surface and groundwater, as well as industrial wastewaters. Ozonation of water include introducing ozone to water through spargers to begin the process, ozone must first be created from an oxygen source: air or pure oxygen tank. To do this, electric current is run through air or oxygen, charging the molecules and converting diatomic oxygen ($O_2$) to ozone ($O_3$). The ozone is then fed through the water, [8].

### 1.2 Treatment by $O_3$+$H_2O_2$

Coupling of ozone with hydrogen peroxide is known as Peroxone process. Many researchers (Beltran et al., (1998),[5]; Scheideler et al., (2011), [20] ) showed that the conjugate base of hydrogen peroxide, $OH_2^{-}$ may triggered the $O_3$ decomposition cycle and as a result accelerate hydroxyl radical formation. In the peroxone process, the ozone residual is short lived because the added peroxide greatly accelerates the ozone decomposition. The oxidation is more reactive and much faster in the peroxone process compared to the ozone molecular process [10]. Summarizing benefits of $O_3/H_2O_2$ system it can be stated that it is more reactive and usually faster compared with ozonation alone, The Peroxone treatment transforms organic carbon to more biodegradable by-products and usually accelerates the degradation of intermediates. The addition of hydrogen peroxide is quite simple and does not require much additional space.

### 1.3 Treatment by $O_3$+UV and $H_2O_2$+UV

Many substances that absorb radiation can decompose through the pathway known as direct photolysis. The photolytic reactions proceed if the given compound absorbs a fraction of the incident UV radiation. UV radiation can also enhance the decomposition of ozone and subsequently the generation of radicals. Both in a gaseous phase and in an aqueous solution, ozone absorbs UV radiation. Maximum absorption is at 253.7 nm. Since ozone absorbs at 254 nm, traditional UV sources can be applied in $O_3$/UV process, [21].

In $O_3$/UV process the contaminants can be degraded by a combination of reaction mechanisms that include direct UV photolysis, direct ozonation and indirect $^{\bullet}OH$ radical oxidation. Direct photolysis of the pollutant can take place if it absorbs the wavelength used. The photodecomposition of ozone leads to in situ production of hydrogen peroxide, [3]. Thus, three components to produce $^{\bullet}OH$ radicals and/or to oxidize the pollutants, UV radiation, ozone, and hydrogen peroxide, exist in this system. The relative contribution of these mechanisms depends on UV radiation, wavelength and intensity, ozone concentration and the type and the concentration of given pollutant. The reaction mechanisms may include the above mentioned ozone/hydrogen peroxide reactions as well as hydrogen peroxide photolysis. Increasing the concentration of $H_2O_2$ leads to the reaction of hydrogen peroxide with $OH^\bullet$ radicals, competing with organic pollutants, then reducing the efficiency of the treatment, [14], also hydroxyl radical may recombine and participate in radical-radical reactions to form $H_2O_2$, lead to a low reaction rate [6].

The main aim of this research is to study the effectiveness, the best conditions and combination of advanced oxidation processes (AOPs) especially the addition of ozone in the treatment of ATZ.

## 2. Experimental work

### 2.1 Chemical used

Atrazine (ATZ), pesticide were in commercial form and bought from Hebei Chinally International Trade Co. LTD (China). The purity of the Atrazine, were 99%, its chemical structure is depicted in Fig. (1). Table 2 shows the physical and chemical properties of this pesticide

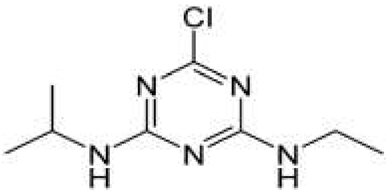

**Fig.1 Chemical structure of Atrazine.**

**Table 2. List of pesticides properties**

| | |
|---|---|
| Formula is | $C_8H_{14}ClN_5$ |
| Molecular weight is | 215.68 g/mol |
| Solubility in water | 34 mg/L at 25C |
| Vapor Pressure | 3x10-7 mm Hg at 20 C |
| Kow | Log Kow = 2.75 |
| Solubility in water | 40 mg/L |

Table 3, 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 NaOH.

**Table 3: List of chemicals used**

| Compound | Formula | Vender | Assay |
|---|---|---|---|
| Oxygen gas | $O_2$ | Local market | 99.99% |
| Hydrogen Peroxide | $H_2O_2$ | Hopkin and williams | >35 % |
| Sodium hydroxide | NaOH | BDH | 99% |
| Acetonitrile HPLC grade | $C_2H_3N$ | Sigma-Aldrich | 99.9% |

### 2.2 Equipments

A semi batch experiments were carried out in the present work using laboratory scale system, Fig. (2). A glass cylindrical reactor with 5L volume, was used. Ozone gas was generated by Enally-Woodland model OZX-03K generator, USA. Ozone generated from oxygen with inlet flow rate of 3L/min, which gives 3gm/hr ozone output. Generated ozone was introduced to the reactor by using Teflon pipes and a ceramic diffuser. Peristaltic pump (BT300-2J) from Longer Company, China with flow rate of 1L/min was used to maintain the desired flow for circulation in the reactor. The Photo-reactor is a stainless steel cylindrical reactor, 2.4 litter volume with the dimensions of 85 cm x 6.5 cm. Irradiation was achieved by using low-pressure mercury vapor lamp, 4-pin single end, 40W, UV-C 254 nm (Philips Company). The lamp was sheathed in glass sleeve for protection and fixed inside along the reactor. The pH of solution was monitored using pH meter from (WTW Co., German, INOLAB 720). The whole system was fixed inside a large fume hood from LabTech Company, to ventilate possible excess ozone and generated gasses.

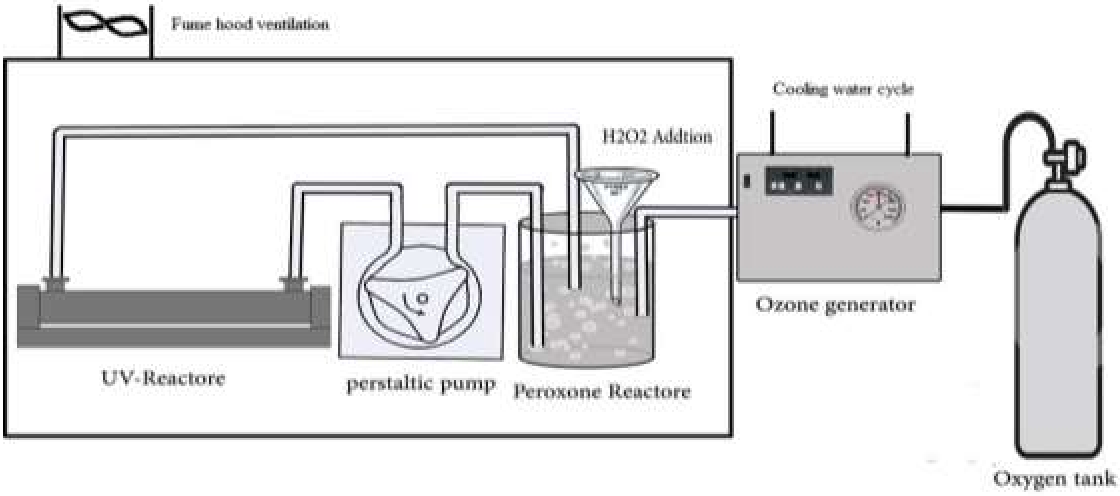

**Fig. 2 schematic diagram of AOP system set-up.**

## 2.3 Analysis

All the analyses were done in the laboratories of Environmental Engineering Department / University of Baghdad. High Performance Liquid Chromatography (HPLC), from Perkin Elmer series 2.00, (USA), was used to identify the pesticide concentration and the intermediate compounds which evolve through the oxidation reaction. The stationary phase is C18 column (with 250mm in length and 4.6mm internal diameter, while its particle size was 5µm), Discovery, from Supelco. The mobile phase used was a mixture of acetonitrile and distilled water (45:55). Flow rate of 1mL/min was used to pump the mobile phase. Peaks were detected with a UV detector at 220nm. All samples were filtered using 0.2μm syringe filter. Fig.(3), shows the standard curves for ATZ using HPLC.

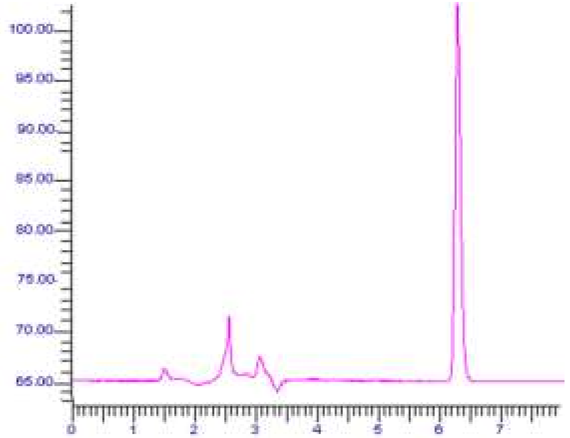

**Fig.3 Standard curves for ATZ using HPLC .**

Chemical Oxygen Demand of samples was analyzed by COD Photometer system. It was used to measure the total organic contents in the solution along the treatment. Appropriate amount of sample (2ml) was introduced into digestion solution (MR-Rang: 0-1500mg/L) containing ($K_2Cr_2O_7$, $H_2SO_4$ and $HgSO_4$). The mixture was then incubated for 120 min at 150°C in the COD reactor (model RD-125, Lovibond Company, Germany). After oxidation is complete, the CODs value was measured by colorimetric method at 605 nm by DR/2010 (model MD-100, Lovibond,Germany) spectrophotometer.

### 2.4 Experimental Procedure

The experimental procedure was performed at room temperature which was about 25°C, flowing the below steps:

1. The desired amount of pesticides were measured and diluted in distilled water. This solution was introduced into the glass reactor. Initial sample at t=0 was taken for analysis.
2. The pH value was adjusted to the desired values by adding NaOH.
3. Ozone generator was turned on and the generated ozone was introduced into the glass reactor by a Teflon pipe and dispersed through the solution by using a ceramic diffuser.
4. The peristaltic pump was turned on and 2.4L of the solution was transfer to the UV reactor. Continues recirculation was maintained between the glass reactor and the UV reactor throughout the experiment.
5. When the oxidation by UV was used the UV lamp was turned on, and when the ozone treatment was not used the ozone generator was turned off.
6. $H_2O_2$ was added to the pesticide solution in the glass reactor when the oxidation process was proceeds with addition of $H_2O_2$.

7. Sample was taken at different period of experiment for the analysis of pesticide concentration and pH.

## 3. Results and discussion

### 3.1 UV Photolysis

Direct photolysis was carried out by passing the atrazine solution with initial concentrations of 1 mg/L through the UV-reactor without any addition of reagents. The temperature was maintained at 25°C throughout the experiments and the solution was irradiated for 180 min. The results were plotted in Fig. (4).

From this Fig. it can be noticed that there was an obvious reduction in ATZ concentration within the first 15 min, Fig.(4a), but COD value remains constant, Fig.(4b). This may be attributed to the fact that ATZ molecule absorb that wavelength very well but the resulted intermediate dose not. This result was in a good agreement with Acero et al., (2000), [2]. The increase in COD values which may be noticed in Fig (4) is a result of the formation of hydrogen peroxide as suggested by Aguinaco, (2014) [3]. Interference between $H_2O_2$ and COD is well known phenomena which lead to overestimation in COD value and this is resulted from consuming of oxidizing agents such as potassium dichromate by $H_2O_2$.

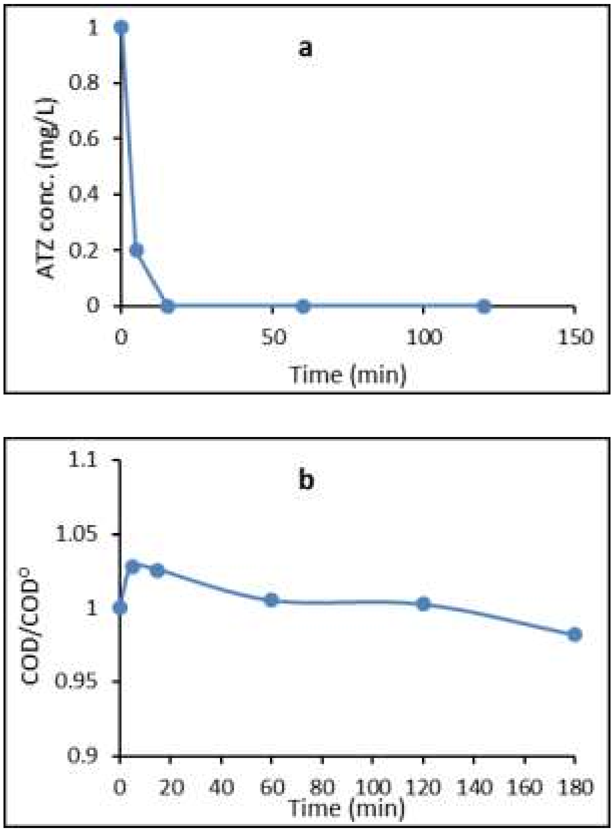

**Fig. 4 UV-photolysis of 1 mg/L Atrazine, pH=7 and Temp.25°C, (a) ATZ degradation (b) COD degradation,**

The variation of pH value during the UV photolysis is illustrated in Fig. (5), it can be seen that the pH decreased with time, this may be a result from the weak acidic effect of the atrazine losses fragments during the process as stated by Hequet et al., (2001), [13].

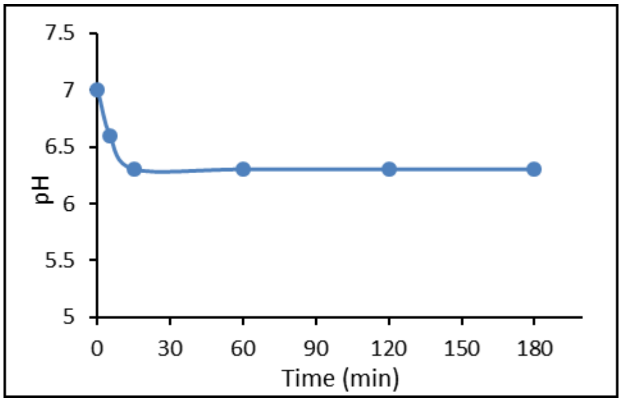

**Fig. 5 pH variation during ATZ irradiation.**

## 3.2 Ozonation Treatment

Ozonation of 1 mg/L of ATZ solution was carried out in a reactor with three liter volume. The temperature was maintained at 25°C throughout the experiments, the inherent pH value =7 and the ozonation time =180 min. The results were plotted in Fig. (6).

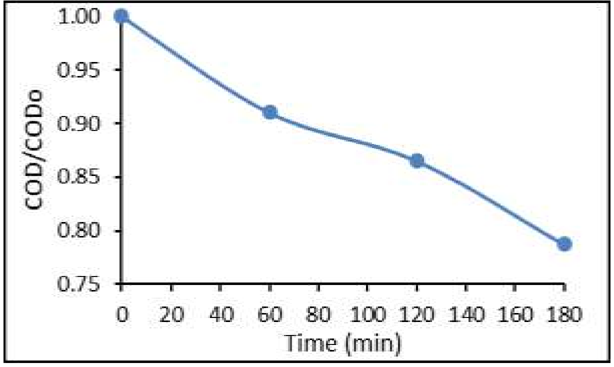

**Fig. 6 COD degradation of 1 mg/L ATZ by ozone**

From Fig. (6), it can be noticed that the COD/COD$_o$ value at the end of experiments have been reduced by 21% after 180 min of treating with ozone, these results were in agreement with Nealieu et al., (2000), [17] and Acero et al., (2000), [2]. Fig. (7) illustrate the variation in pH during the ozonation treatment; it can be observed that there is a decline in the pH from its inherent value 7 to about 4.4, this might be a result of acidic nature of degradation products. This fall in pH values turns solution into acidic so it diminishes the OH radical formation and push towards direct ozone reaction, [10].

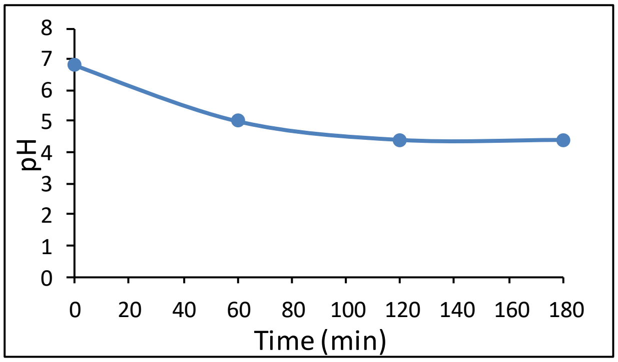

**Fig. 7 pH variation during ATZ ozonation, initial pH=7.**

In order to improve the ozonation treatment, the rate of radical formation can be enhanced by increasing the pH value of the solution, as mentioned by [18]; [10]; [12]. Initial pH equal to 12 was used in the present experiments, as suggested by (Prado and Esplugas, 1999), [19]. The ATZ solution was maintained at pH=12 by using sodium hydroxide. Fig. (8) shows the COD reduction at pH =12

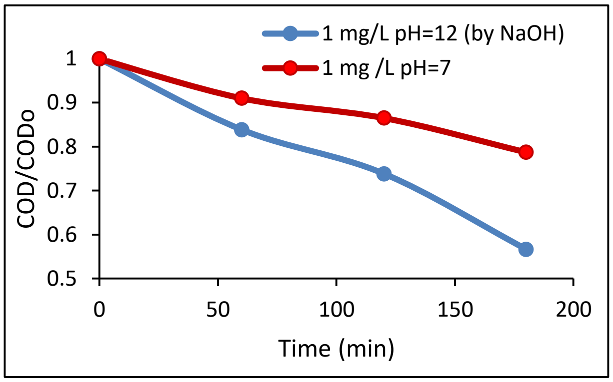

**Fig.8 Ozonation of ATZ at pH =12 and inherent pH=7.**

Fig. (8) shows that at pH=12 and ATZ concentration 1mg/L, the COD reduction attain 43%. It can be observed that the COD reduction was enhanced compared with the COD reduction for ATZ solution at pH=7 which represent the inherent pH of ATZ solution. Fig. (9), shows the variation in the value of pH during the ozonation treatment at initial pH=12. From this figure it can be seen that there is no obvious change in the pH during the experiments, it means that the solution remain in basic environment which is favorable for the ozonation treatment.

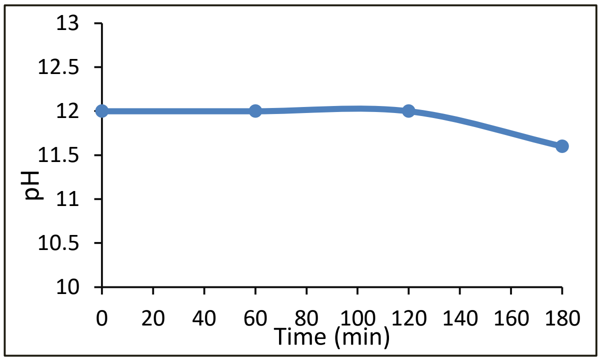

**Fig.9 pH variation during ATZ ozonation, initial pH=12 by NaOH.**

## 3.3 Ozone+UV Treatment

To increase the degradation efficiency of ATZ, $O_3$ was combined with UV light, since the UV irradiation energy is absorbed by $O_3$ molecule and generates the hydroxyl radical $OH^{\bullet}$ which is responsible for the degradation and mineralization of organic compounds. The temperature was maintained at 25°C throughout the experiments and a solution of 1 mg/L ATZ were ozonated and irradiated for 180 min. The results were plotted in Fig. (10). 31% reduction in COD value could be mentioned, which may indicate that the amount of generated radicals are not quite enough to oxidize the whole ATZ.

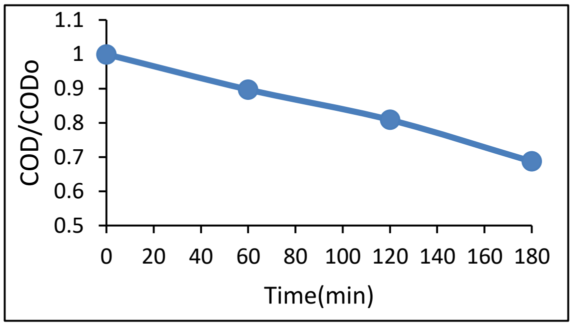

**Fig. 10 $O_{3+}$UV treatment of ATZ at pH=7.**

Fig.(11) illustrate the variation in pH value during $O_3$+UV treatment, it shows that the pH value drops from around 7 to about 3.5, this may be a result from the acidic fragments produced through the generation of intermediate, as mentioned previously

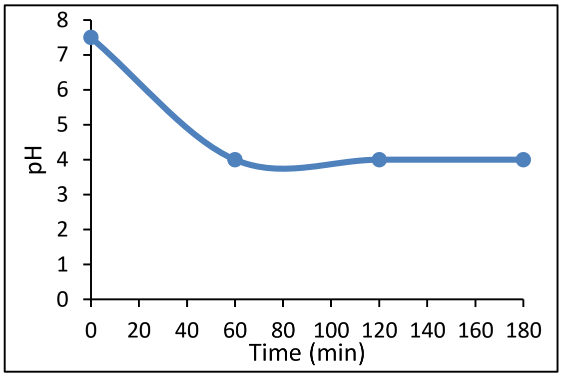

**Fig. 11 pH variation during $O_3$+UV treatment of ATZ , pH=7.**

Fig.(12) shows the COD reduction for ATZ solution at initial pH value of 12. The results are plotted in Fig. (12).

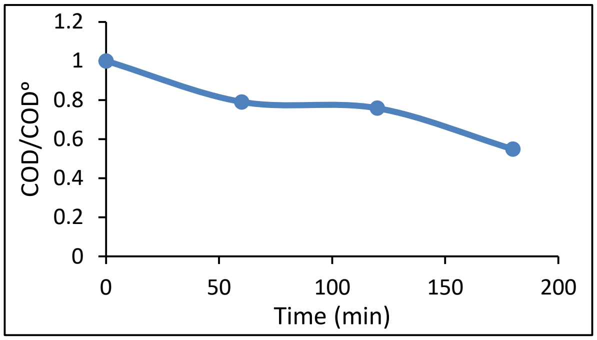

**Fig. 12 $O_3$+UVtreatment of ATZ concentration, pH=12**.

The reduction in COD values obtained was 45%. A comparison between the results obtained by $O_3$+UV treatment at pH=7 and pH=12 was shown in Fig.(13). It was obvious from the comparison that the COD removal efficiency was increased by 25% at pH=12.

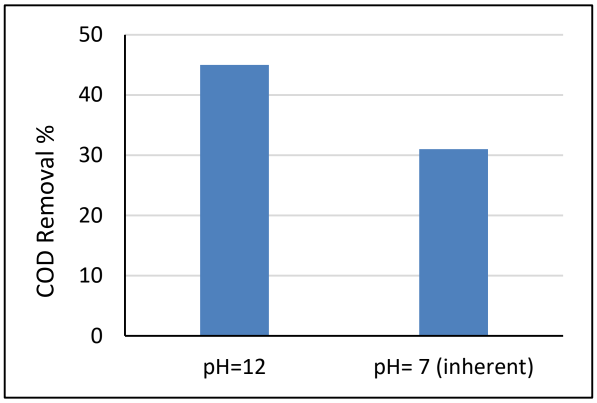

**Fig. 13 Comparison between COD removal efficiency for $O_3$+UV treatment, at pH=7 and pH=12**

## 4.7 Ozone+$H_2O_2$ Treatment at pH=12

From our previous work (Mustafa and Mohammed, 2015) it was found that ozone+$H_2O_2$ treatment is more efficient in alkaline conditions so set of experiments were conducted to determine the optimum $H_2O_2$ concentration that required to achieve the best COD reduction. 70, 140 and 210mg/L $H_2O_2$ were tested, the results were plotted in Fig (14). From Fig.(14a) it can be observed that the COD reduction was increased from 74 to 95 % when $H_2O_2$ increases from 70 to 140 mg/L, then a decrease in the COD reduction to 84 % was observed for 210 mg/L $H_2O_2$ concentration. This can be explained as: $H_2O_2$ is acting as a scavenger at high concentrations i.e. hydrogen peroxide react with new formed radicals and inhabit the degradation reaction [12]; [3]. Fig.(14b) shows that the ATZ with concentration of 1mg/L and $H_2O_2$ concentration of 140mg/L was completely converted to the intermediate product after 60 min of ozone+ $H_2O_2$ treatment. From the previous results as a conclusion the best reduction in COD for 1mg/L ATZ was obtained to be 95% with the $H_2O_2$ concentration of 140 mg/L.

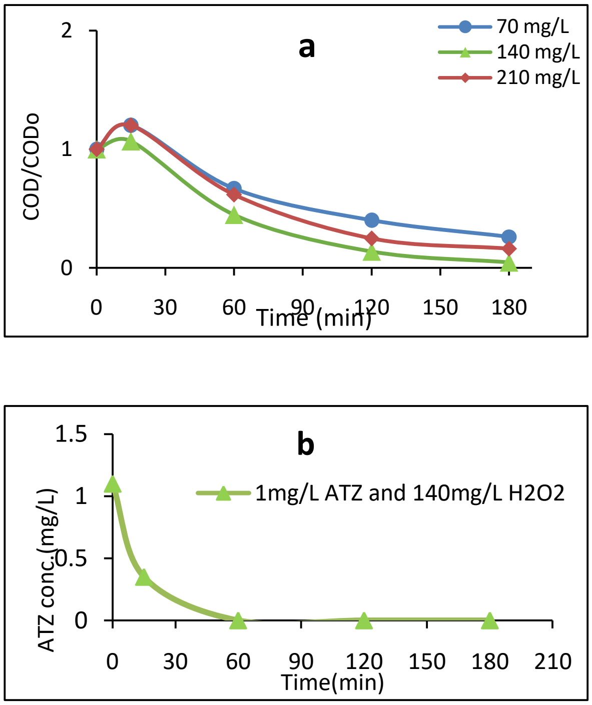

**Fig.14 $O_3$+$H_2O_2$ treatment at different initial $H_2O_2$ concentrations, pH=12, (a) COD degradation (b) ATZ degradation.**

## 4.9 UV+$H_2O_2$ Treatment

UV+$H_2O_2$ treatment was tested in the present experiments to compare its degradation efficiency with other treatments adopted in the present research. 300 and 600 mg/L $H_2O_2$ were used at pH=7(inherent pH) to find the appropriate dosage for the treatment. The results are plotted in Figs.(15). It can be noticed that 95% in COD reduction was achieved after 180min of treatment with 600mg/L $H_2O_2$.

Fig.(15b) shows that ATZ was converted to the intermediate products after 15 min only.

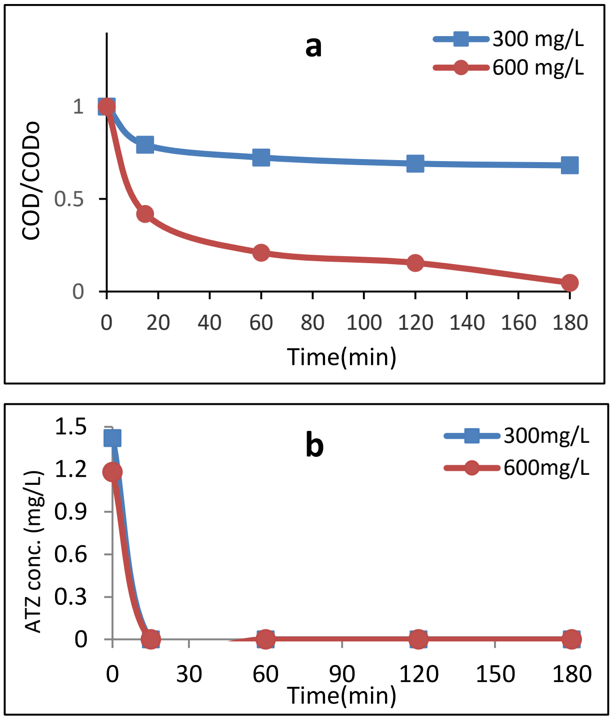

**Fig.15 UV+$H_2O_2$ treatment at different initial $H_2O_2$ concentrations, at pH=7, (a) COD degradation (b) ATZ degradation.**

Fig.(16) shows the variation of the pH values at different initial $H_2O_2$ concentration. The pH all along the initial time of experiment, specifically after 15 min decreases sharply then no observed decrease was noticed, this was because of the acidic nature of the mineralization and intermediate products.

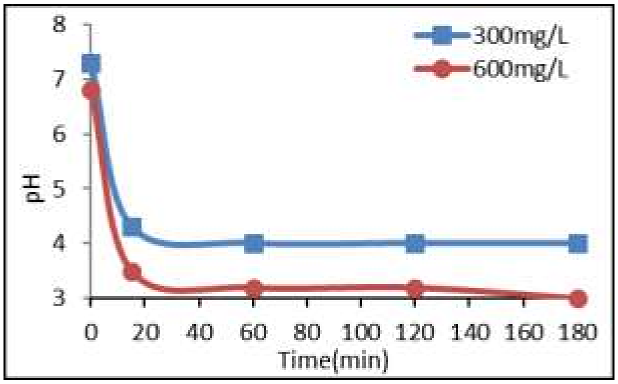

**Fig. 16 Effect of initial $H_2O_2$ concentrations on the pH variation by UV+$H_2O_2$ treatment at ATZ concentrationa1mg/L and initial pH=7.**

## 5. Conclusion

There are many conclusions that may be taken from the experimental work of this study, they are as follows:

1. Complete conversion of ATZ to intermediate products was achieved after 15min of treatment when using UV photolysis (UV only), but there was no reduction in the COD values was observed along the treatment indicating that the intermediate products is recalcitrant and is most difficult to degraded by UV photolysis only.
2. The pH plays an important role in the ozonation treatment (ozone only). The COD reduction achieved 43%, at pH=12, compared with 21% at pH=7.
3. An enhancement in ozonation treatment was obtained by combined $O_3$ with UV. The COD reduction attained was 45% at pH=12.
4. $O_3$+$H_2O_2$ treatment improve the COD reduction of ATZ to reach 95% at 140 mg/L of $H_2O_2$ and pH=12.
5. UV+$H_2O_2$ treatment was investigated to compare its degradation efficiency with $O_3$+ $H_2O_2$ treatment. 95% was achieved at 600mg/L $H_2O_2$ and pH=7. UV+$H_2O_2$ treatment seems to be efficient to treat ATZ but it required a relatively high amount of$H_2O_2$

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# ازالة الاترازين من الماء بأستخدام عمليات الاكسدة المتقدمة

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## الخلاصة

تم تقييم كفاءة عمليات الاكسدة المتقدمة $UV, O_3, UV+O_3, O_3+ H_2O_2, UV+H_2O_2$ في هذه الدراسة باستخدام مفاعل مختبري لغرض ازالة الاترازين . تم استخدام المبيد بتركيز 1 ملغم/لتر وتم دراسة اثر الحامضية على هذه العملية باضافة هيدروكسيد الصوديوم. لم تحدث ازالة لـ (COD) عند استخدام اشعة UV بينما اعطى الاوززن 43٪. عند استعمال الاوزون مع الاشعة فوق البنفسجية وبوسط قاعدي دالته الحامضية 12 اعطى 45٪ ازالة للملوث. الاوزون مع بيروكسيد الهيدروجين اعطى ازالة 95٪ باستخدام 140 ملغم/لتر من بيروكسيد الهيدروجين وبوسط قاعدي دالته الحامضية 12 ايضاً. كما تم الحصول على ازالة 95٪ للملوث باستخدام الاشعة فوق البنفسجية و 600 ملغم/لتر من بيروكسيد الهيدروجين في الوسط القاعدي بدالة حامضية 12.