

## Effect of chloride concentration on the oxidation of EDTA in UV-FSR oxidative system

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### Abstract

This study focused on the effect of chloride concentration and pH on the photochemical degradation of ethylenediaminetetraacetic acid–disodium salt ( $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$ , EDTA) under advanced oxidation using UV/ $H_2O_2$  system. The treatment of this chemical by UV/ $H_2O_2$  oxidation using a laboratory scale UV-free surface reactor (UV-FSR) with (1000 and 10,000 mg/l  $Cl^-$ ) and without  $Cl^-$  addition at different pH values (pH 3, 7 and 10) was compared. Results of this study indicated that  $Cl^-$  concentration is more decisive than pH in the oxidation process. There was no AOX at the start of the experiments but as a result of oxidation a de novo synthesis of AOX was observed, and these AOX<sub>de novo</sub> compounds were destroyed during the treatment. Treatment was followed by TOC and AOX measurements. Approximately 95% TOC removal efficiency was obtained for the treatment of EDTA-containing synthetic water. Owing to kinetic studies, it was found that the TOC degradation in all experiments fitted to the first order reaction law.

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**Keywords:** AOX<sub>de novo</sub> formation; AOX removal; oxidation processes; UV-free surface reactor; EDTA

### 1. Introduction

Ethylenediaminetetraacetic acid (EDTA) is a common chelating agent used in many different industrial applications for sequestration of metal ions. Thus, it is employed in the photographic industry, in textile and paper manufacturing, in the metal industry and in chemical cleaning processes but, because of its metal binding and metal solubilizing properties, it may produce deleterious effects when released into the environment. For example, EDTA can make difficult the removal of heavy metals present in water and wastewater by conventional precipitation processes mainly because of the dramatic increase in the solubility of heavy metal ions. In addition, the presence of organic chelating agents such as EDTA may also cause some metal industry wastewater discharges to violate the COD requirement for effluent discharge, because the toxicity of heavy metal complexes prevents the reduction of COD by biological treatment

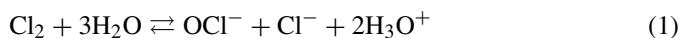
processes. Removal of EDTA from wastewaters by conventional techniques is not simple because it is not easily biodegradable [1]. There is great environmental interest in destruction of this substance because it is found in many rivers and lakes under anthropogenic stress; the microbial degradation of EDTA is relatively poor and takes place only under very special conditions; treatment with chlorine or other disinfectants does not result in any significant degradation of EDTA [1].

Adsorbable organic halogens (AOX) is a measure for halogenated compounds and is an important parameter for the characterization of industrial wastewaters. A large number of AOX causing substances show a significant eco-toxicity [2].

Sewage water legislation in Germany provides very strict regulations for the discharge of AOX containing wastewaters. Those wastewaters often cause technical and financial problems for specific industries [3]. It is well known that, chlorination of drinking water or swimming pool water causes unwanted formation of chloroform and other halogenated derivatives such as chloramines. Another source of AOX in wastewater is created by the application of chlorous budget cleaners (chloramine T, chloramine BARS, sodium hypochlorite, chloric

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lime). Addition of Chlorine to water produces hypochlorite by the following reaction [4]:



Chlorine addition can be formulated in a simplified manner as follows:

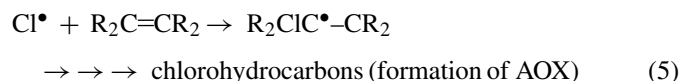


In the last decade, the so-called “advanced oxidation processes” (AOPs) have been proposed for the waste-free degradation of toxic pollutants in industrial wastewaters. Several AOP-methods are based on the combination of classical oxidants such as  $\text{H}_2\text{O}_2$  or  $\text{O}_3$  with UV irradiation with or without catalysts. In these processes, extremely reactive hydroxyl radicals are primarily generated and these are able to oxidize and destroy most of the wastewater constituents, including halogenated organic compounds [5].

Many industrial wastewaters such as tannery and dye manufacturing wastewaters have extraordinarily high chloride ion concentrations due to extensive use of sodium chloride. High  $\text{Cl}^-$  concentrations may cause serious corrosive problems with reactor components consisting of stainless steel. Dissolved chloride ions react rapidly with hydroxyl radicals ( $\bullet\text{OH}$ ) as described by the following equations (Eqs. (3) and (4)) [6]:



$\bullet\text{ClOH}^-$  can decompose to yield chlorine atoms. Chlorine atoms can add to  $\text{C}=\text{C}$  double bonds of the compounds present during advanced oxidation processes (AOP) treatment, generating chlorinated hydrocarbons (Eq. (5)), and leading to an undesirable increase of the global parameter AOX [6]:



Some researchers have reported the effect of chloride ion concentration on the efficiency of AOP at a constant pH value, while some others have reported the effect of pH in the absence of chloride ions. For example, chloride ion was reported to inhibit the decomposition of dichlorvos insecticides [7] and 2,4-dichlorophenol [8] in the Fenton process, which produces  $\bullet\text{OH}$  efficiently under acidic pH conditions. In addition, chloride ion was also found to inhibit color removal, oxidation of tetrachloroethylene (PCE), and mineralization of natural organic matter in processes where  $\text{TiO}_2$  [9],  $\text{O}_3/\text{H}_2\text{O}_2$  [10] or sonolysis/ $\text{O}_3$  [11] were used. Gurol and Akata [12] used a  $\text{Cl}^-$  as  $\bullet\text{OH}$  trap to determine the primary quantum yield in ozone photolysis.

Some researchers carried out experiments to examine the effects of  $\text{Cl}^-$  on the photocatalytic degradation of Methylene Blue, and Orange II in aqueous  $\text{TiO}_2$  suspensions under UV light illumination [13]. They found that the high chloride concentration adversely effected the degradation rate of the dyes.

In another study the toxicological effect of disinfections using sodium hypochlorite on aquatic organisms, and its contribution to AOX formation in hospital wastewater were investigated [14]. The same workers found that low chloride concentrations corresponded to low concentrations of AOX, and the high chloride concentrations generated high AOX and high toxicity on *D. magna*.

As can be seen, in the literature there are no significant publications concerning the amount of AOX formation, or elimination in wastewaters containing different amounts of chloride ions when UV/ $\text{H}_2\text{O}_2$  treatment is applied.

Today most of the UV- $\text{H}_2\text{O}_2$  photoreactors used for industrial wastewater treatment are equipped with submerged UV-radiators—a rather disadvantageous design which originates from the sterilization technique for clear effluents. If high loaded wastewaters are treated with submerged radiators, dirty coatings on the surface of the UV-radiator quartz cover tubes cause considerable reduction of the effectiveness of the UV- $\text{H}_2\text{O}_2$  process. In order to overcome this problem, in cooperation with industry, a completely new reactor type for UV wet oxidation was developed at the University of Stuttgart, namely, the UV-free-surface-reactor (UV-FSR), and was used in this work. In contrast to the commonly used UV-reactors with submerged radiators the new concept is marked out by the position of the UV radiator which is a few centimeters above the surface of the wastewater.

The main goal during the UV-FSR-development was simplification of the entire technique to obtain a low price, robust, and user friendly reactor. The aim was to operate the UV-FSR with a single push-button, and to be able to replace the UV radiator or the UV reflectors with a few simple manipulations. The UV-FSR is completely air cooled and equipped with 10 cm UV medium pressure radiators with a specific power of approximately 170 W/cm. The electrical equipment consists of a newly developed power semiconductor unit without transformers.

The main objectives of this study were to observe the effect of chloride ion concentration, and pH on  $\text{AOX}_{\text{de novo}}$  formation using this recently developed UV-free surface reactor (UV-FSR) and to determine the degradation efficiency of EDTA and to optimize reaction conditions.

## 2. Materials and methods

### 2.1. Materials

EDTA (ethylenediaminetetraacetic acid, disodium salt) ( $\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$ ) was obtained from Merck Chemical Company. Concentration of substance was 1000 mg/l in the synthetic wastewater, and the COD value was calculated theoretically. In this study only one concentration of Hydrogen peroxide was applied. The Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) (50%, w/w) dosage was based on the stoichiometric ratio with respect to COD. Calculated hydrogen peroxide concentration is 2539 mg/l for EDTA.

During the experiments, pH was kept constant at pH 3, 7 or 10 with the addition of either 25%  $\text{H}_2\text{SO}_4$  or 33%  $\text{NaOH}$  depending on the experimental conditions.  $\text{NaCl}$  was used as a  $\text{Cl}^-$  source

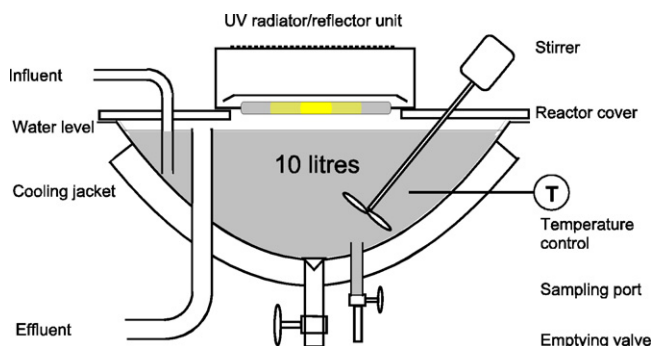


Fig. 1. Schematic sketch of the 10 l UV-FSR.

and purchased from Merck. The experiments were continued until all  $\text{H}_2\text{O}_2$  was consumed, and reaction times were 4 h for EDTA.

## 2.2. UV-FSR reactor

The reactor used in this work is a bowl shaped, laboratory scale, 10 l glass reactor with 300 mm diameter and 140 mm depth (Fig. 1). It is gold coated on the outside to prevent emission of light from the reactor. Gold coating was used for its inert and durable nature. The reactor is equipped with a cooling jacket, influent inlet, safety outlet, a sampling port, temperature control equipment and, a stirrer. The UV-FSR is a completely mixed reactor, mixing is obtained with a mechanical stirrer having a Teflon coated blade. The UV irradiation source (10 cm) is a 170 W/cm (UVH 100 26 S1) air cooled medium pressure mercury UV radiator encased in a quartz tube and it was purchased from UV Technique Mayer Ltd. Bleichenbach Germany.

## 2.3. Experimental procedure and analytical methods

For a standard reaction run, 10 l of aqueous solution was used. As a first step in this study it was necessary to remove the free chlorine present in the tap water to study the effects of free chlorine on the photo-degradation of organic substances. Prior to each experiment, the feed tank was filled with hot tap water (60–70 °C) and the free chlorine in tap water was removed overnight by aeration. It was observed that the elimination of free chlorine by this method was successful. Dechlorinated water was pumped into the UV-FSR reactor and following the adjustment of EDTA ( $\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$ ) concentration in the solution, the required amount of sulphuric acid or sodium hydroxide was added to obtain the desired pH in the solution where needed. The pH of the solutions was not buffered; therefore, during experiments, pH was always controlled with a pH control system (a pH meter, a controller and a dosing pump) located within the reactor, and kept constant. Each substance was treated in the presence of 1000 mg/l  $\text{Cl}^-$ , 10,000 mg/l  $\text{Cl}^-$  or without  $\text{Cl}^-$  at pH 3, pH 7 and pH 10. In the batch experiments, all of the required  $\text{H}_2\text{O}_2$  was added into the reactor at the beginning of the test run. The time at which the ultraviolet lamp was turned on was considered time zero. Then, a stopwatch was started and samples were taken at predetermined time intervals to be anal-

ysed for TOC, AOX and  $\text{H}_2\text{O}_2$ . The 250 ml samples were taken at beginning of the experiments and after 6, 18, 30, 60 min and 4 h until the  $\text{H}_2\text{O}_2$  had completely disappeared. Because large volumes are necessary, samples were not taken during 1–4 h of the experiments. The samples were analysed immediately to avoid further reaction. It is possible that VOC's could be produced during the oxidation; to prevent the VOC's escape from the reactor the temperature of the solution was kept constant at  $30 \pm 2$  °C throughout all the experiments.

AOX was determined in a TOX analyser (TOX-10  $\Sigma$ , Abimed, Düsseldorf, Germany) after enrichment on activated carbon [15]. TOC measurements were carried out by using a Jena Type Multi NC 3000 TOC Analyzer equipped with an auto sampler.  $\text{H}_2\text{O}_2$  concentration was measured according to German Standard Methods [16].

## 3. Results and discussion

### 3.1. Results and discussion of the experimental studies

The first step was to determine the effect of chloride concentration on the process. Therefore, experiments were carried out at three different chloride ion concentrations (without  $\text{Cl}^-$ , or in the presence of 1000 mg/l  $\text{Cl}^-$  or 10,000 mg/l  $\text{Cl}^-$ ) at a constant initial EDTA concentration of 1000 mg/l and a hydrogen peroxide concentration of 2539 mg/l (1  $\times$  stoichiometric). Under these conditions the amount of  $\text{H}_2\text{O}_2$  (47 ml/10 l) concentration was nearly zero after 4 h batch treatment. There was no AOX at the beginning of the experiments, but AOX appeared and increased after 18–30 min in the presence of  $\text{Cl}^-$  ions. After 4 h of batch treatment, this newly formed AOX<sub>de novo</sub> was decomposed. The maximum AOX<sub>de novo</sub> production was obtained in the presence of 10,000 mg/l  $\text{Cl}^-$ , whereas no AOX production occurred when  $\text{Cl}^-$  was not added (Fig. 2). Vollmuth et al. [17] as well as other authors [18] have already observed the formation of AOX during UV oxidation.

TOC concentration of synthetic wastewater was 357 mg/l. The degradation curves of TOC were almost the same at three  $\text{Cl}^-$  concentration (0, 1000, and 10,000 mg/l) and after 4 h batch treatment TOC concentrations of all cases were treated to approximately 45 mg/l (Fig. 3).

The effect of different pH values and chloride concentration on TOC removal efficiencies for EDTA was given in Table 1 as a function of irradiation time. TOC removal efficiency is between 85 and 95% after 4 h batch treatment. Under alkaline condition TOC removal efficiencies are higher than under acidic and neutral conditions for EDTA degradation. It was found that chloride concentrations are not very effective on the TOC removal efficiency. Although Seiss et al. [19] found that different pH values and chloride concentrations effect TOC removal efficiencies greatly; in our studies it has been found that this is not true under all the reaction conditions.

Ghiselli et al. [1] performed degradation experiments using 5 mmol/l ethylenediaminetetraacetic acid (EDTA) solutions at pH 3 in the presence of  $\text{H}_2\text{O}_2$  and metals such as  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$  and mixtures of  $\text{Fe}^{2+}/\text{Cu}^{2+}$  and  $\text{Fe}^{3+}/\text{Cu}^{2+}$  under UV-A irradiation (366 nm) photo-Fenton and photo-Fenton-like reactions at

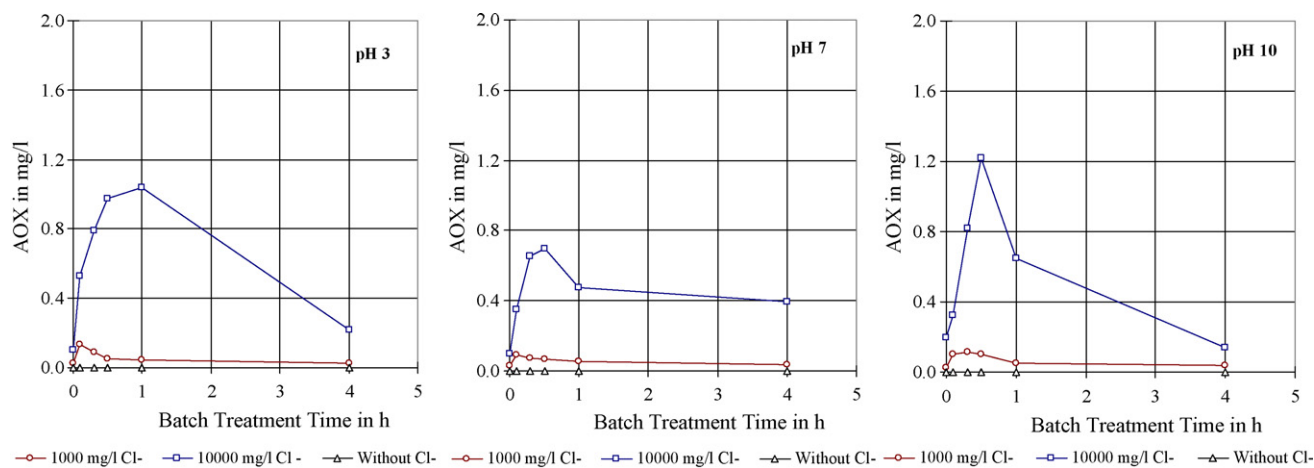


Fig. 2. AOX formation (and decomposition) in the treatment of EDTA.

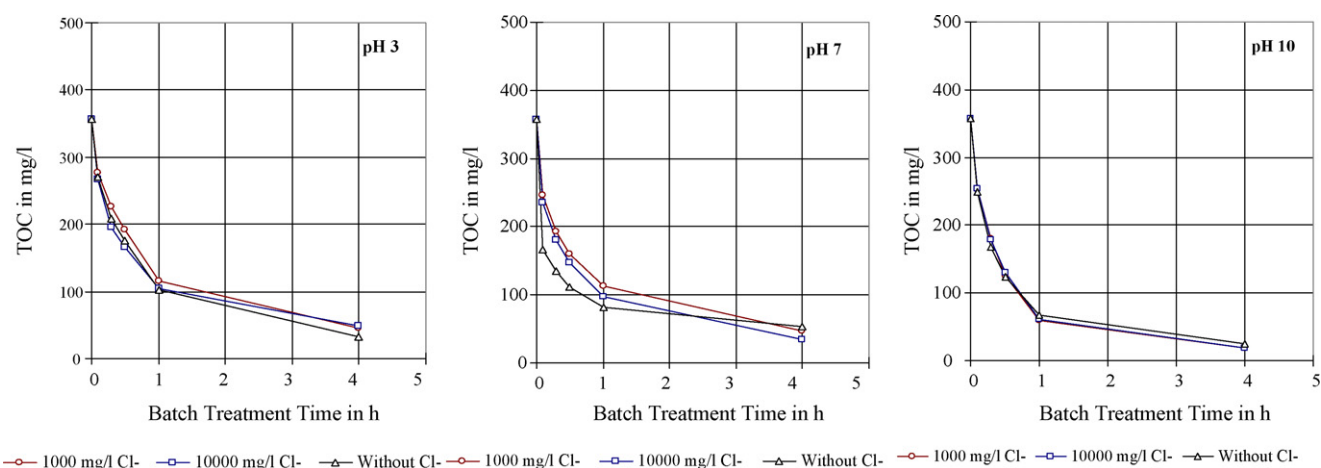


Fig. 3. TOC degradation results of EDTA.

different metal/EDTA concentration ratios in order to determine the best conditions for EDTA photochemical removal. And they determined that, in Fenton systems, TOC removal was very low at the end of 4 h for all experiments, reaching a maximum of 31.9% removal in the case of 1:1 EDTA:Fe<sup>2+</sup> and 13.0% in the case of 1:1 EDTA:Fe<sup>3+</sup>. The Fenton's reaction in the absence of photons can be ineffective due to competition between the substrate and H<sub>2</sub>O<sub>2</sub> for the hydroxyl radical ( $\bullet$ OH). In addition, some by-products formed in this process, in particular oxalic acid, can be accumulated in the reaction media due to its refractory nature to Fenton's reagent. Rivas et al. [20] observed this

behaviour when *p*-hydroxybenzoic acid, a phenolic model compound, was oxidized in aqueous solutions by Fe<sup>2+</sup>/Fe<sup>3+</sup>/H<sub>2</sub>O<sub>2</sub>, in the dark.

In contrast to this, Ghiselli et al. [1] found that all photo-Fenton systems (except for the one using only Cu<sup>2+</sup>) yielded a TOC removal above 80% after 4 h irradiation.

Based upon the fact that TOC removal was very efficient under irradiation, a more detailed work evaluating EDTA destruction was carried out. Due to the fact that EDTA degradation was very fast in light mediated experiments (UV/H<sub>2</sub>O<sub>2</sub>), such as previously observed by Ku et al. [21] in which EDTA

Table 1  
Effect of pH and chloride concentration on TOC removal efficiencies for EDTA in UV/H<sub>2</sub>O<sub>2</sub>

Time (h)	TOC removal efficiency (%)								
	pH 3			pH 7			pH 10		
	Without Cl <sup>-</sup>	1000 mg/l Cl <sup>-</sup>	10,000 mg/l Cl <sup>-</sup>	Without Cl <sup>-</sup>	1000 mg/l Cl <sup>-</sup>	10,000 mg/l Cl <sup>-</sup>	Without Cl <sup>-</sup>	1000 mg/l Cl <sup>-</sup>	10,000 mg/l Cl <sup>-</sup>
0.1	24	22	25	54	31	34	30	29	29
0.3	42	37	45	62	46	49	53	49	50
0.5	51	46	54	69	55	59	65	65	64
1	71	68	71	77	68	73	81	83	83
4	91	87	86	85	87	90	93	95	95

Table 2a

Effect of pH 3 and chloride concentration on the reaction rate constants with EDTA containing wastewater in UV/H<sub>2</sub>O<sub>2</sub>

Cl <sup>-</sup> concentration	Zero order		First order		Second order	
	<i>k</i> <sub>0</sub> (mg/l min)	<i>r</i> <sup>2</sup>	<i>k</i> <sub>1</sub> (min <sup>-1</sup> )	<i>r</i> <sup>2</sup>	<i>k</i> <sub>2</sub> (× 10 <sup>-6</sup> l mg <sup>-1</sup> min <sup>-1</sup> )	<i>r</i> <sup>2</sup>
Without Cl <sup>-</sup>	3.8090	0.8806	0.0177	0.9975	113.205	0.9888
1000 mg/l Cl <sup>-</sup>	3.6376	0.9076	0.0162	0.9984	95.317	0.9850
10,000 mg/l Cl <sup>-</sup>	3.7521	0.8407	0.0167	0.9844	106.735	0.9807

Table 2b

Effect of pH 7 and chloride concentration on the reaction rate constants with EDTA containing wastewater in UV/H<sub>2</sub>O<sub>2</sub>

Cl <sup>-</sup> concentration	Zero order		First order		Second order	
	<i>k</i> <sub>0</sub> (mg/l min)	<i>r</i> <sup>2</sup>	<i>k</i> <sub>1</sub> (min <sup>-1</sup> )	<i>r</i> <sup>2</sup>	<i>k</i> <sub>2</sub> (× 10 <sup>-6</sup> l mg <sup>-1</sup> min <sup>-1</sup> )	<i>r</i> <sup>2</sup>
Without Cl <sup>-</sup>	3.4151	0.5561	0.0128	0.9818	140.559	0.9204
1000 mg/l Cl <sup>-</sup>	3.4772	0.7777	0.0141	0.9809	109.811	0.9682
10,000 mg/l Cl <sup>-</sup>	3.6692	0.7692	0.0161	0.9888	119.675	0.9688

Table 2c

Effect of pH 10 and chloride concentration on the reaction rate constants with EDTA containing wastewater in UV/H<sub>2</sub>O<sub>2</sub>

Cl <sup>-</sup> concentration	Zero order		First order		Second order	
	<i>k</i> <sub>0</sub> (mg/l min)	<i>r</i> <sup>2</sup>	<i>k</i> <sub>1</sub> (min <sup>-1</sup> )	<i>r</i> <sup>2</sup>	<i>k</i> <sub>2</sub> (× 10 <sup>-6</sup> l mg <sup>-1</sup> min <sup>-1</sup> )	<i>r</i> <sup>2</sup>
Without Cl <sup>-</sup>	4.3076	0.8190	0.0341	0.9725	199.483	0.9700
1000 mg/l Cl <sup>-</sup>	4.4774	0.8571	0.0266	0.9977	229.817	0.9770
10,000 mg/l Cl <sup>-</sup>	4.4451	0.8574	0.0262	0.9990	224.839	0.9747

was destroyed almost completely within 1 h, while the organic intermediates were mineralized to CO<sub>2</sub> within 5 h.

In the literature, several authors [22–25] detected that EDTA concentration has dropped after 4 h of reaction in the dark, some organic compounds such as ethylenediaminediacetic acid (EDDA), iminodiacetic acid (IDA) and oxalic acid could be formed as by-products of the decomposition of EDTA in aqueous solution when different Advanced Oxidation Techniques were employed. Thus, the TOC values were not noticeably decreased in these cases, even though the EDTA concentration dropped drastically. In contrast to this, in our study we obtained approximately 95% TOC removal efficiency.

### 3.2. Results of the kinetic studies

For the determination of the order of the reaction for photooxidation of EDTA in the UV-FSR reactor, kinetic studies were done according to TOC degradation curves and several orders such as zero, first and second were tested. Kinetic studies were carried out only for one hour oxidation time. Zero, first and second order constants are given in Tables 2a–2c according to applied pH values.

As a result of the kinetic studies, it was found that the TOC degradations in all studies are fitted to first order reaction law. For this reason only the first order reaction constants are discussed in this section.

It was determined that the initial chloride concentration had no significant inhibitory effect on the first-order reaction rate

constant, *k*, in the oxidation of EDTA containing wastewater (Table 3). The *k* value decreased with increasing chloride concentration at pH 3. And under basic pH conditions (pH 10) obtained higher *k* values (Fig. 4) and TOC removal efficiencies. The maximum first-order reaction rate constant, *k*, was 0.0291 min<sup>-1</sup> and determined at pH 10. Total treatment cost of EDTA containing wastewaters calculated according to this *k* value, and it is approximately 13 €/m<sup>3</sup> to obtain 95% TOC removal efficiency.

According to Bolton et al. [26] if concentration of a substance is high, then the reaction rate will be zero; on the other hand, if concentration of a substance is low, the reaction rate will be first order. But in our studies, it was found that for high concentration of EDTA, the reaction rate was first order. Electric energy per mass (*E*<sub>EM</sub>) and Electric energy per order (*E*<sub>EO</sub>) was calculated as given by Bolton et al. [26] and they were 109 kWh/kg TOC and 38 kWh/m<sup>3</sup> per order, respectively.

Table 3

Effect of pH and chloride concentration on the first-order reaction rate constants with EDTA containing wastewater in UV/H<sub>2</sub>O<sub>2</sub>

Cl <sup>-</sup> concentration	pH 3		pH 7		pH 10	
	<i>k</i> (min <sup>-1</sup> )	<i>r</i> <sup>2</sup>	<i>k</i> (min <sup>-1</sup> )	<i>r</i> <sup>2</sup>	<i>k</i> (min <sup>-1</sup> )	<i>r</i> <sup>2</sup>
Without Cl <sup>-</sup>	0.0177	0.9975	0.0128	0.9818	0.0341	0.9725
1000 mg/l Cl <sup>-</sup>	0.0162	0.9984	0.0141	0.9809	0.0266	0.9977
10,000 mg/l Cl <sup>-</sup>	0.0167	0.9844	0.0161	0.9888	0.0262	0.9990

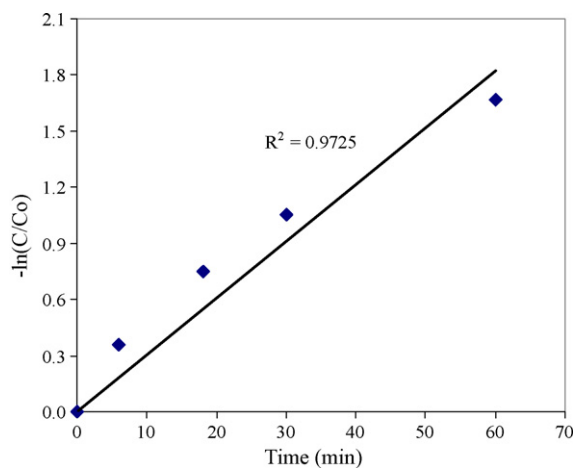


Fig. 4. Concentration vs. time dependency for first order degradation of EDTA (without  $\text{Cl}^-$ , pH 10).

#### 4. Conclusion

This study reveals the role played by various chloride ion concentrations in the AOX formation at different pHs during UV/ $\text{H}_2\text{O}_2$  oxidation. Regarding the experimental studies, during batch treatment of EDTA, the maximum AOX formation rate was observed with 10,000 mg/l  $\text{Cl}^-$  at pH 10, and approximately 95% TOC removal efficiency was obtained. The maximum first-order reaction rate constant,  $k$ , was  $0.0291 \text{ min}^{-1}$  and determined at pH 10.

According to other studies, pH is an important parameter for oxidation processes, but in our work, results showed that the  $\text{Cl}^-$  concentration in the water and the chemical structure of the substances are more important than the pH of the water for AOX<sub>de novo</sub> production. During the batch experiments, a de novo synthesis of AOX was observed very intensively due to the high chloride content of the wastewaters. This may imply that OH-radicals oxidize some chloride-ions to form chlorine, which further reacts with organic compounds so that AOX<sub>de novo</sub> is produced; eventually, these AOX compounds are destroyed.

The advantages of the UV-FSR process as an oxidative pretreatment step over other photochemical treatment processes include reduced reaction times for complete mineralization, economics, efficiency, especially if aromatic compounds are to be

destroyed, easy handling method, because no specific technical equipment is necessary, and harmless process products, and no dirty coating on the surface of UV radiator.

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