

## Removal of Congo red from textile wastewater by ozonation

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**ABSTRACT:** Congo red, which has a complex molecular structure with various diazo aromatic groups, is widely used in textile industry as an anionic dye. The purpose of this study was to investigate the degradation of Congo red in laboratory solution which had the chemical properties of the rinse waters of textile manufacturing dye-houses and the samples with Congo red alone wastewater by ozonation and to optimize the reaction parameters such as pH and time which influence the efficiencies of total organic carbon, total kjeldahl nitrogen and chemical oxygen demand removal. Ozonation of Congo red dye were carried out in a semi-batch reactor with constant ozone flow rate and concentration of 23 mL/sec and 13.6 mg/L, respectively. Decolorization was complete within a few minutes of ozonation possibly due to the cleavage of chromophore groups. It was observed that its structural destruction occurs predominantly at higher pHs. The reduction of chemical oxygen demand and destruction of the dye was more than 60 % and 42 %, respectively. Total kjeldahl nitrogen removal was accompanied by slight changes in nitrogen oxides. It can be deduced from the experimental results that: (a) the mineralization is very weak; (b) the reaction follows the indirect mechanism; i.e., the interaction of hydroxyl radicals with the dye and (c) the nitrification is rather predominant. Biological oxygen demand is declined in simulated alkalic and neutral samples respectively. At 13.6 mg O<sub>3</sub>/L, the biological oxygen demand levels were significantly enhanced. This might be attributable to the enhancement of its biodegradation at alkaline pHs.

**Key words:** Biological factor, total organic carbon, chemical oxygen demand, azo dyes, decolorization, effluents

### INTRODUCTION

Textile industry is one of the most important industries in last few decades. It is broadcasted that more than 60 % of the dyes world production is consumed by textiles industries (Khadhraoui *et al.*, in press). Designated as water soluble, it was estimated that 10–20 % of dye was lost during the dyeing process and released as effluent (Zhiqiao *et al.*, 2007). The reagents used in textile industry are very diverse in chemical composition. The non-biodegradability of textile wastewater is due to the high content of dyestuffs, surfactants and other additives, which are generally organic compounds of complex structure (Muthukumar *et al.*, 2004). It is difficult to treat these wastewaters by conventional technologies (Faouzi *et al.*, 2006). Moreover, different papers report that in most cases, biochemical oxygen demand/chemical oxygen demand ratio of the composite textile wastewater is around 0.25 implying the non-biodegradability of the organic matter therein (Arsalan *et al.*, 2002; Ahmet *et al.*, 2003; Azbar *et al.*, 2004; Lopez *et al.*, 1998). Among the used dyes, azo-dyes are the most commonly utilized

reagents owing to the presence of the azo-group which confers to these chemicals a certain resistance to light, acids, bases and oxygen, the desired properties for clothes' makers (Hung Yee and Ming Chin, 2005; Jiangning and Tingwei, 2001). More than 53 % of these commonly used azo-dyes are identified as non-biodegradable compounds. As a consequence, wastewaters bearing such type of dyes are known to be highly resistant to the mostly widespread used conventional wastewater treatment method: the biological processes (Banat *et al.*, 1996; Vandavivire *et al.*, 1998; Razo-Flores *et al.*, 1997). In the past several decades, the wastewater discharges by dye manufacturing and textile finishing industries has been a major environmental concern (Koch and Yediler, 2002; Sheng, 2000). This is primarily due to their strong color with high dissolved solids in addition to the presence of elevated levels of aromatic ring containing organic compounds, the carcinogenic pollutants (Sarasa and Cortes, 2002). Congo red dye is one of important azo dyes. It is colored substances have complex chemical structures and high molecular weights. The chemical structure is the sodium salt of benzidinediazo-bis-1-

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naphtylamine-4-sulfonic acid. The dye's chemical structure and its main characteristics are shown in Fig. 1. It is highly soluble in water and persistent in the environment, once discharged into a natural environment. Thus, the study on Congo red is interesting not only for being possible pollutants of industrial effluents but also because it is a good model of complex pollutants (Tapalad *et al.*, 2008). As to ozone, which is considered one of the powerful oxidizing agents (2.07V) (Alaton and Balcioglu, 2002), its application as a pretreatment for the improvement of wastewater biodegradability is thoroughly investigated (Eremektar *et al.*, 2007; Gokcen and Ozbelge, 2005; Bila *et al.*, 2005). In the recent years, ozonation is one of the most attractive alternatives for decolorize dye wastewater. Ozone is an extremely strong oxidant and reacts rapidly with most of organic compounds (Olivia *et al.*, 2006). Ozone and hydroxyl radicals generated in aqueous solution are able to break the aromatic rings (Wei and Chi-Wai., 2000). The ozonation process left no chemical sludge in the effluent, combined color removal and COD reduction in one step and is easily operated. Moreover, the residual ozone naturally decomposed to oxygen (Jianging and Tingwei, 2001). It has been shown that ozone cleaves the conjugated bonds of azo-dyes chromophores, leading to color removal and enhancing the biodegradability of the treated wastewater (Eremektar *et al.*, 2007; Selcuk, 2005). In contrast, other studies (Yediler *et al.*, 2000) claim that short-term ozonation of a model aqueous azo-dye solution and dye-bath effluents from textile dyeing and finishing industry formed toxic compounds. These results were proved by testing the biodegradability of the effluent and bioluminescence tests (Yediler *et al.*, 2000; Wang *et al.*, 2002). Martins *et al.* (2006) have shown that in order to obtain high toxicity removal, longer ozonation periods with high ozone doses have to be applied. Zhang *et al.* (2004) reported that the major disadvantage of using ozone is the possible formation of toxic byproducts even from biodegradable substances. The limitations of conventional chemical oxidation technique can be

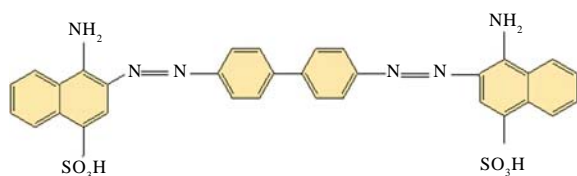


Fig. 1: Chemical structure of the studied molecule (molecular formula,  $C_{32}H_{22}N_6Na_2O_6S_2$ , molecular weight = 696.68 g)

overcome by the development of so-called advanced oxidation processes (AOPs) which involves the use of strong oxidizing agents ( $O_3$ ,  $H_2O_2$  or combined  $O_3/H_2O_2$ ) in the presence or absence of an irradiation source (Alaton and Balcioglu, 2002; Legini *et al.*, 1993). It is also possible to utilize natural processes such as flocculation to remove dissolved metals (Karbassi, *et al.*, 2008; Karbassi and Saeedi, 2008; Karbassi *et al.*, 2007). In principle, AOPs generate very powerful and non-selective oxidizing agent, the hydroxyl radical ( $OH^\bullet$ ) for the destruction of refractory and hazardous pollutants found in groundwater, surface water and industrial wastewaters, respectively. The treatment of reactive and direct dyes in aqueous solutions via different AOPs has been extensively studied (Balcioglu and Arslan, 1999; Chen *et al.*, 1999; Chun and Yizhong, 1999). However, the advanced oxidation of direct and reactive dye bath effluents is rather limited to a smaller number of investigations (Arslan *et al.*, 1999; Uygur and Kok, 1999). The present study focuses on the comparative analyses of the treatment of anionic direct dye in simple solutions and simulated exhausted dye bath liquors prepared from Congo red in different dyeing formulations. Thus this investigation is also aimed to obtain chemical and environmental data as COD, TKN, TOC or BOD for Congo red when its molecular deterioration is caused by ozone injection alone as an AOP technique. This research has been down at Islamic Azad University, Tabriz Branch on 2006.

## MATERIALS AND METHODS

### Materials

Congo red ( $C_{32}H_{22}N_6Na_2O_6S_2$ ; molecular weight: 696.66 g/mol) was purchased from Bayer, as labeled A200. Dye solutions were prepared by dissolving the dye in distilled water and concentration of 60 ppm to simulate loaded textile wastewater. The Congo red solution has the initial pH of 7. The pH was adjusted with hydrochloric chloride (HCl) and sodium hydroxide (NaOH). Solvents are purchased from BHD.

### Ozonation reactor

Ozone was produced by a discharge ozone generator model 500 from dry oxygen used as the feed gas provided by the Electronic Department of Baku National University. The ozone was produced by ozone generator with flow rate of 23 mL/s. and fed into the reactor through a porous glass diffuser located at the bottom of the reactor to produce fine bubbles. The oxygen was fed in constant flow rate and the input of ozone into the sample

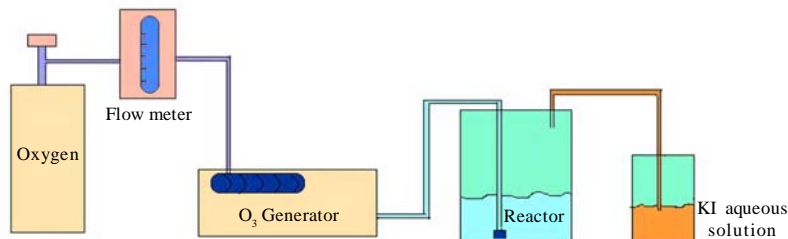


Fig. 2: Sketch of the experimental apparatus used for the ozonation

Table 1: Amount of the ozone absorbed at different time and oxygen flow rate.  $C(O_3) = 13.6 \text{ mg/L}$ ,  $C(\text{dye}) = 60 \text{ mg/L}$

Time (min.) (mL/sec)	0	5	10	15	30	45	$O_2$ flow rate
Absorbed $O_3$ (mg/L)	0	1.214	4.28	5.2	9.31	10.6	10
	0	1.371	3.628	6.121	10.52	11.8	16
	0	1.714	4.8	7.52	12.97	13.6	23
	0	1.714	4.6	6.85	12.1	13.2	28

solution was calibrated according to the time of injection. The excess ozone leaving the reactor was destroyed by sequential 20 % KI traps incorporated to the reactor set-up as shown in Fig. 2. In this study, during the ozonation times, any excess of ozone was not identified through the sequential process mentioned previously. All ozonation experiments were conducted at room temperature and no reactor cooling was provided. The calibration data are shown in Table 1 as well as in Fig. 3. The value in time axes of whole Figs. are presented in this work are taken from the first row of the above mentioned table; i.e., the contact time of ozone. It should be noticed that the amount of the ozone absorbed can be simply obtained through either the time of oxygen injected on the its into the pilot reactor.

*Simulated direct dye bath effluent*

The direct dye (anionic Congo red ) and dye assisting chemicals were kindly supplied by an integrated textile manufacturing plant. The recipe mixture and the direct dye were used upon considering the international market share of their direct dyestuff content and colors mostly applied to the cotton fabrics in the dyeing stage. Usually 90-100 ppm of dyestuff and 1000 ppm of all applied dye assisting chemical remain in the exhausted direct dye bath liquor. The synthetic dye bath effluent was prepared about 60 ppm. The Congo Red solutions in distilled water alone, referred as laboratory sample herein, was first dissolved in doubly distilled water and then kept in cool room for 12 h. prior to its use. The pH of solutions was adjusted with diluted sodium hydroxide or hydrochloric acid as when necessary. The pH values were selected in such a way as occurs in dyeing effluents of textile complexes; i.e.,  $7 < \text{pH} < 11$ .

*Chemical and biochemical measurements*

Oxidation advancement was followed by monitoring of chemical oxygen demand (COD), total organic carbon (TOC), total kjeldahl nitrogen (TKN) and pH. These parameters were analyzed according to the Standard methods described in the ASTM. Absorbance was determined using a Pharmacia Biotech Ultraspec 2000 UV/Vis spectrophotometer. Total organic carbon and total kjeldahl nitrogen were measured using a Skalar analyzer (Holland) and biological oxygen demand was measured using a BOD<sub>5</sub> Oxitop Box WTW (Germany) analyzer .

**RESULTS AND DISCUSSION**

*Experimental data*

Changes in pH were rather insignificant during any advanced oxidation of simulated dyehouse effluent. The pH was slightly declined in samples wherein the dye alone present since the solution was not highly buffered. In all treatment cases, the decline in the values of TOC, COD and UV –VIS absorbance is a general trend. The variation in  $A/A_0$  percentages with the

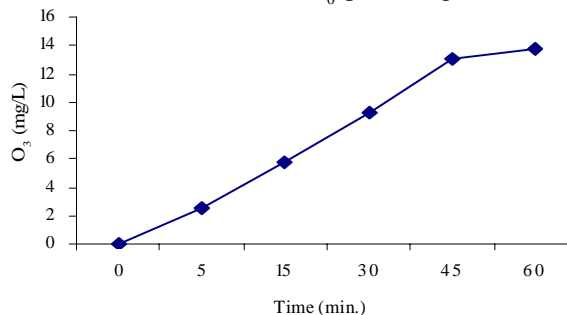


Fig. 3: The variation of ozone concentration versus the time of oxygen injection ( $O_3$  flow= 23 mL/S.)

duration of ozone injection is shown in Fig. 4 at different pHs of Congo red. The variation of COD/COD<sub>0</sub> % with the time of ozone injection is shown in Fig. 5 for the same samples. The TOC/TOC<sub>0</sub> % variation with the ozone doses (as calibrated to be 13.6 mg/L at the injection of 45 min.) is shown in Fig. 6 for the samples with different pH values. The variations of BOD5 and TKN with different injection time are shown in Tables 2 and 3 for samples with three different pH values, respectively.

*Analysis of data*

Ozone reacts with aromatic pollutants found in water and wastewater via two different pathways, namely direct molecular and indirect radical chain type reaction. The direct mechanism occurs mostly at neutral (and for less extend in acidic) chemical condition, whereas the indirect pathway is predominant at higher pHs (Shu and Huang, 1994). Hence, it can be deduced that the ozonation reaction pathway strongly depends on the characteristics of the sample to be treated, i.e. pH, concentration of initiators, promoters and scavengers in the reaction medium. The data show that the effect of pH on treatment efficiency. The simulated direct dye bath effluent subjected to ozonation at the specific O<sub>3</sub> doses had more or less the same consistency with respect to the pH of the samples. The same data was observed in the case of laboratory samples. From these data, it can be concluded that the OH<sup>0</sup> scavenging does not take place in all samples under study. To conduct the ozonation processes at higher pHs, the treatment performance of the ozonation should be further improved. In fact, Table 2 indicates that the biological efficiency increased as the doses of O<sub>3</sub> increased. This is mainly because of a biodegradable organic species produced following the destruction of the molecular structure of Congo red by ozonation treatment. Again, the best results can be observed with the samples of pH=11.0. All the cases mentioned previously are consistent with whole figures presented in the text. The removal percentages of dye at different chemical conditions in the terms of COD and TOC have been shown in Figs. 7 and 8, respectively.

**CONCLUSION**

In all treatment cases, decolorization of the samples was observed instantaneously in both simulated effluents and the samples contained the dye alone. This observation indicates that the color removal can be achieved in a simple process via both reaction pathways of ozone with Congo red. The higher molecular

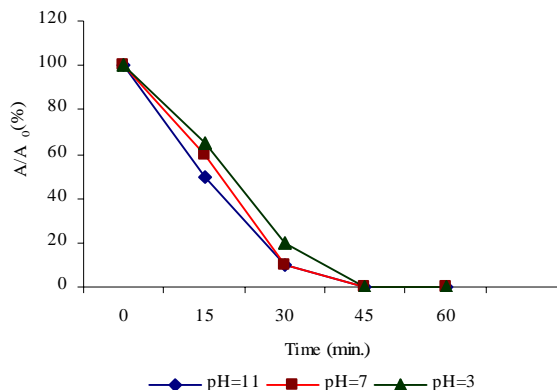


Fig. 4: Effect of pHs on percent UV absorption of Congo red at different ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

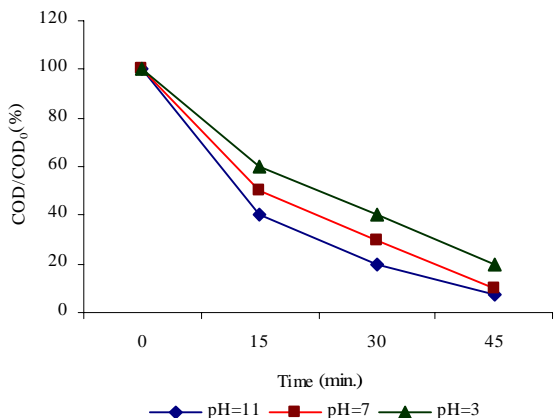


Fig. 5: Effect of pHs on percent COD removal of Congo red at different ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

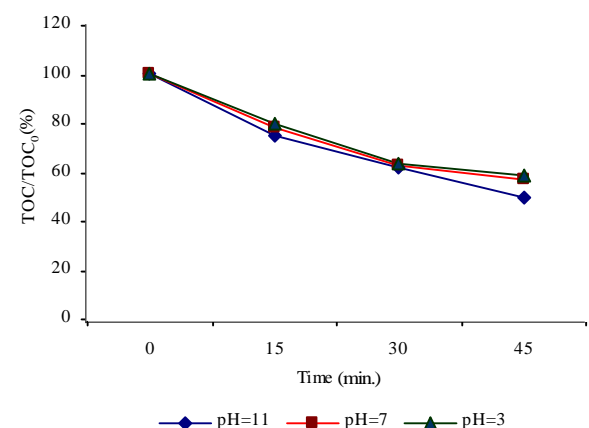


Fig. 6: Effect of pHs on percent TOC removal of Congo red at different ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

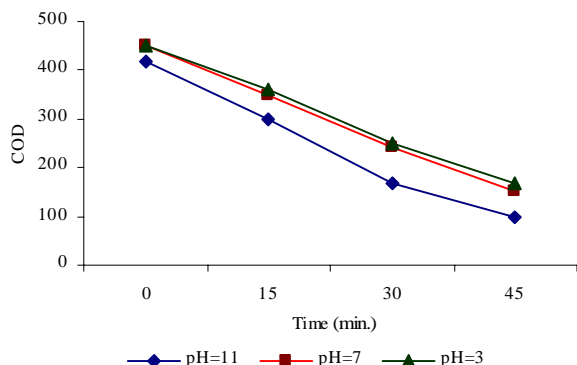


Fig. 7: Effect of pHs on COD removal of Congo red at different ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

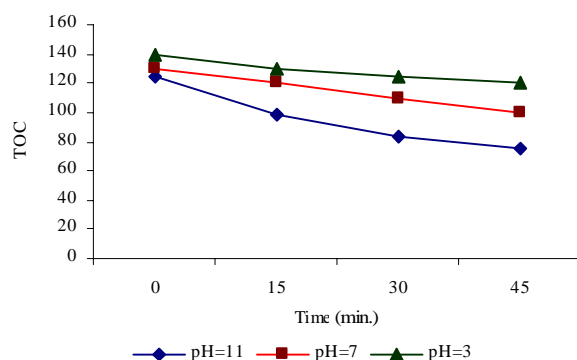


Fig. 8: Effect of pHs on TOC removal of Congo red at different ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

deterioration of Congo red takes place at alkaline condition, i.e., the indirect pathway of ozonation reaction. The ozonation process enhances the biodegradation efficiency due to the biodegradability of the products which were produced during the treatment. Therefore, it can be suggested that an optimal procedure for AOP<sub>s</sub> would be a pre-ozonation followed by a biodegradation process.

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Table 2: Effect of pHs on BOD value of Congo red at different ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

pH	BOD <sub>5</sub> /BOD <sub>0</sub>	BOD <sub>5</sub> /BOD <sub>15</sub>	BOD <sub>5</sub> /BOD <sub>30</sub>	BOD <sub>5</sub> /BOD <sub>45</sub>
3	156	110	80	40
7	160	170	210	140
11	180	195	220	120

Table 3: Effect of pHs on TKN value of Congo red before and after 45 min. ozonation time. C (O<sub>3</sub>) = 13.6 mg/L, C (dye) = 60 mg/L

pH	TKN <sub>0</sub>	TKN <sub>45</sub>	TKN <sub>45</sub> - TKN <sub>0</sub>
3	123	108	15
7	146	125	21
11	151	114	37

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