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Decolorization and detoxification of textile wastewater by ozonation and coagulation processes

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Abstract

The aim of this work is to evaluate and compare the performance of ferrous and aluminum sulfate coagulations and ozonation treatment techniques. The evaluation of treatment efficiency was made using the parameters of oxygen demand (COD), color absorbances at 436 nm, 525 nm and 620 nm and *Daphnia magna* toxicity test. Approximately 50–60% color, 60% COD and 70–80% toxicity were removed at 1000 mg L^{-1} and 1500 mg L^{-1} ferrous and aluminum sulfate, respectively. However, these required doses for optimum toxicity reduction are not economical due to the chemical sludge production. Ozonation was relatively effective in reducing color absorbances and toxic effects of textile effluents. Almost complete color absorbances (over 98%) were removed in 20 min ozone contact time, while COD removal (37%) was very low and almost stable in 30 min ozonation period. The toxicity of wastewater reduced after color degradation by 85% at the transferred ozone (TrO_3) concentration of 82.3 mg L^{-1}.

Keywords: Coagulation; Decolorization; Ozonation; Textile wastewater; Toxicity reduction

1. Introduction

The textile industry is one of the most complicated industry among manufacturing industries. Various toxic chemicals such as complexing, sizing, wetting, softening, anti-felting and finishing agents, biocides, carriers, halogenated benzenes, surfactants, phenols, pesticides, dyes and many other additives are used in wet processes which are mainly called washing, scouring, bleaching, mercerizing, dyeing, finishing processes. As a result, textile plants produce highly toxic wastewater [1–3].

Textile mill effluents are generally characterized by the parameters of biological oxygen demand (BOD), COD, pH, suspended solids and color. Most of these parameters are removed satisfactorily by conventional chemical coagulation and biological treatment methods except highly polymer structured color. Recently, many researchers found that textile wastewaters were toxic even after treatment [4,5].

Advanced oxidation processes including ozonation, UV/H_2O_2, TiO_2/UV, Fenton's regent, photo-Fenton and photoelectrocatalytic oxidation were tried for the purification of water and wastewater [6–12]. Among these advanced oxidation methods, ozone combinations are the most applied advanced oxidation methods used before biological treatment to enhance biodegradability and remove color in textile wastewaters. It is stated that an increase in the ratio of BOD/COD after ozonation results from an improved biodegradability of toxic substances [13–16]. However, the major disadvantage of using ozone is that it may form toxic byproducts even from biodegradable substances [16–18].

The aim of this work was to apply coagulation and ozonation treatment methods for decolorization and detoxification of wastewaters originated from synthetic–cotton textile mill. The evaluation of treatment efficiency

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was made using the parameters of COD, toxicity and color. Color was followed by the absorbances at 436 nm, 525 nm and 620 nm wave lengths (namely $color_{436}$, $color_{525}$ and $color_{620}$, respectively), which are referred in German wastewater discharge standards.

2. Materials and methods

2.1. Coagulation and flocculation

Aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$) and ferrous sulfate ($FeSO_4 \cdot 7H_2O$) of commercial grade were utilized for the experimental procedure. Aluminum sulfate and ferrous sulfate experiments were carried out at the optimum pH values of 7.0 and 9.5, respectively [19] at room temperature (20 ± 2 °C) using 500 ml sample. A series of Jar-test experiment applying 2 min rapid mixing at 100 rpm, 20 min slow mixing at 30 rpm and 1 h for settling was conducted on raw wastewater. Anionic polyelectrolyte (5 mg L^{-1}) (Henkel 23500) was used as flocculant.

2.2. Analytical methods

COD was measured according to Standard Methods [20]. Absorbance measurements were made using Pharmacia LKB-Novaspe II model spectrophotometer for color removal. The supernatants in each beaker were filtered by 0.45 μm Millipore membrane filter before measuring COD, and absorbance at 436, 525 and 620 nm wave lengths. $FeSO_4$ solution was prepared daily and all chemicals used were of analytical grade. Removal efficiency of COD and color were obtained according to the formula given below.

$$\text{Removal}\,(\%) = (C/C_0)/C_0 \qquad (1)$$

where C_0 and C are the initial and final absorbance or COD values of simulated wastewater, respectively.

2.3. Toxicity measurement

The toxicity of raw and treated textile wastewaters was tested using 24-h born *Daphnia magna* at 50% and 100% dilution as described in Standard Methods [20]. Daphnids were grown in the laboratory at 16 h light (under 3000 lx illumination) and 8 h dark periods. They were fed *Selenastrum capricornutum* (300.000 cell/ml) and baker's yeast (*Schizosaccharomyces cerevisiae*, 200.000 cell/ml). All solutions were prepared in bidistilled water at pH 8. Room temperature was kept at 20 ± 2 °C and minimum 6 mg L^{-1} of dissolved oxygen was supplied by air filtered through activated carbon. Toxicity experiments were carried out quadruplicate and five daphnids used in each test beaker with 50 ml of effective volume. Results were evaluated on the basis of immobilization percentage obtained by dividing the number of immobilized animals by total animals. The toxicity of wastewater samples was explained as toxic when the immobilization percent is higher than 50%.

2.4. Ozonation

Ozone was generated from air by ozone generator. A closed cylindrical Pyrex glass reactor with a diameter of 40 mm and height of 1100 mm was used in the ozonation and catalytic ozonation experiments. A tubular cylindrical porous diffuser was replaced at the bottom of the reactor to transfer input ozone gas into aqueous solution. Teflon tubing line was used for the connection between ozone generator and reactor. The appropriate ozone concentrations were adjusted by changing the electrical current of the ozone generator. All experiments were performed at room temperature (20 ± 2 °C) using 1 L sample. After ozonation, the sample was aerated for 5 min to remove possible residual ozone. Two bubblers containing 250 ml of 2% KI solution was used to trap the ozone in off gas (Fig. 1). Then, a sodium thiosulfate titration procedure was performed to measure the ozone concentration, trapped in the KI solutions. The transferred ozone (TrO_3) was calculated as follows:

$$TrO_3 = \text{Produced ozone} - (\text{Ozone in off gas}).$$

3. Results

3.1. Sampling and characterization of wastewater

Wastewater sample performed in this study was supplied from a cotton–synthetic textile factory located

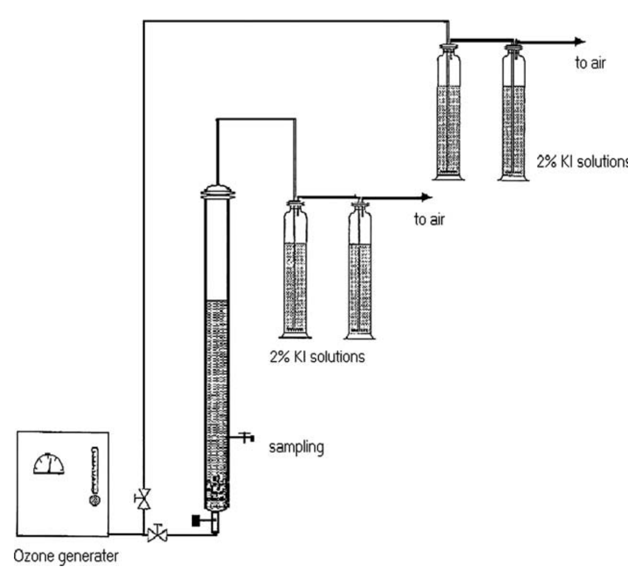

Fig. 1. Experimental set-ups for the ozonation experiments.

in Maslak, Istanbul, Turkey. The fibers used in the textile plant can be classified into two main types: cotton and synthetic fibers including polyamide, polyester and rayon. Washing, scouring, peroxide bleaching, dyeing and finishing processes are the major wastewater sources in the textile factory. According to the operation manager, various harmful chemicals such as complexing, sizing, wetting and finishing agents, biocides, carriers, halogenated benzenes and phenols are used in the processes. The operation in the textile plant varies from day to day even an hour. Currently, wastewater is treated by a conventional biological treatment method. In order to avoid the shock effect of harmful chemicals on the biologically activated sludge and to reduce the usage of high amount of acid and base for neutralization of wastewater, a basin with a 4 h retention time is used to balance influent wastewater. Table 1 shows the characterization of wastewater sample taken from balance tank. According to the textile waster groups [21], highly colored textile wastewater sample was medium strange wastewater with its COD level of 1150 mg L^{-1}. The results of *Daphnia magna* toxicity test showed that the wastewater sample was toxic even at 150% dilution.

3.2. Coagulation with ferrous sulfate

Basic equations occurring during the coagulation process for ferrous sulfate salts is given with Eqs. (2) and (3) as follows:

$$FeSO_4 + 2HCO_3^- \leftrightarrow Fe(OH)_2 + SO_4^{2-} + 2CO_2 \quad (2)$$

$$2FeCl_3 + 6HCO_3^- \leftrightarrow 2Fe(OH)_3 + 6Cl^- + 6CO_2 \quad (3)$$

Varying ferrous sulfate concentrations (up to 2000 mg L^{-1}) keeping pH constant (9.5) were applied for the treatment of raw wastewater. The raw wastewater was toxic at 100% dilution, thus after ferrous sulfate coagulation, *Daphnia magna* toxicity test was carried out at 100% dilution. As Table 2 presents, a ferrous sulfate concentration of 750 mg L^{-1} was necessary for an efficient COD removal and higher ferrous sulfate doses did not significantly change COD removal. On the other hand, the degradations of color absorbances were not very high at the ferrous sulfate concentrations $\leq$500 mg L^{-1}. They decreased with increasing ferrous sulfate concentration and above 75% removals were obtained for all measured color absorbances at the ferrous sulfate concentration of 1500 mg L^{-1}. The toxicity test was performed at 100% dilution and effective toxicity reduction was observed at the ferrous sulfate concentrations $\geq$1000 mg L^{-1}. Otherwise, it should be pointed out that the coagulated wastewater was not toxic at the COD concentrations $\leq$119 mg L^{-1} and at the color absorbances $\leq$0.11 cm^{-1} at 436 nm, 0.35 cm^{-1} at 525 nm and 0.10 cm^{-1} at 620 nm wave lengths. Approximately 50% color absorbances, 59% COD and 80% toxicity were removed from wastewater at the optimum ferrous sulfate dosage of 1000 mg L^{-1} that was found to be optimum for toxicity reduction. As can be seen in Table 1, a good correlation was observed between color degradation and toxicity reduction and toxicity reduced with the decreasing color absorbances. This finding may be attributed to the toxic effect of dyes as reported in the literature [5].

3.3. Coagulation with aluminum sulfate

Aluminum ion, Al^{+3}, behaves very much like Fe^{3+}. When aluminum sulfate is added to water or wastewater, $Al(OH)_3$ precipitation occurs as described by the following equation:

$$Al_2(SO_4)_3 + 6HCO_3^- \leftrightarrow 2Al(OH)_3 + 3SO_4^{2-} + 6CO_2 \quad (4)$$

Similar to ferrous sulfate experiment, varying aluminum sulfate concentrations, keeping pH at the optimum condition (7.0), were applied for the treatment of raw wastewater. Dilution (100%) was applied for the

Table 1
Characterization of influent and effluent wastewaters of the treatment plant

Parameter	Unit	Raw wastewater
COD	(mg L^{-1})	1150
Filtered COD	(mg L^{-1})	580
BOD_5	(mg L^{-1})	170
TSS	(mg L^{-1})	150
Chloride	(mg L^{-1})	1820
Sulfate	(mg L^{-1})	680
Total hardness	(mg $CaCO_3$ L^{-1})	80
Conductivity	(μMhos cm^{-1})	13 500
$Color_{436}$	m^{-1}	1.24
$Color_{525}$	m^{-1}	1.37
$Color_{620}$	m^{-1}	1.02
pH		10
Toxicity[a]	(%)	100
Toxicity[b]	(%)	70

[a] At 100% dilution.
[b] At 150% dilution.

Table 2
Treatment of raw wastewater with ferrous sulfate

$FeSO_4$ (mg L^{-1})	Absorbance (cm^{-1})			COD removal (%)	Toxicity reduction (%)
	436 nm	525 nm	620 nm		
0	1.06	1.37	0.77	0	0
250	0.83	1.29	0.68	15	0
500	0.78	1.13	0.43	38	0
750	0.30	0.81	0.25	55	20
1000	0.22	0.69	0.19	59	80
1500	0.19	0.34	0.13	62	100
2000	0.15	0.29	0.09	65	95

toxicity test. As can be seen in Table 3, the optimum aluminum sulfate concentrations for color absorbances, COD and toxicity removals were determined as 1500 mg L^{-1}, 1000 mg L^{-1} and 1500 mg L^{-1}, respectively. Color (60%), COD (56%) and toxicity (70%) were removed from raw wastewater at the 1500 mg L^{-1} aluminum sulfate, obtained as an optimum dose for color and toxicity reduction. Efficient toxicity reductions in aluminum sulfate experiment were gained at the COD concentrations ≤ 128 mg L^{-1} and at the absorbance values ≤ 0.12 cm^{-1} at 436 nm, 0.28 cm^{-1} at 525 nm and 0.11 cm^{-1} at 620 nm. The important point is that in both aluminum and ferrous sulfate experiments, COD and absorbance levels for the toxicity reduction were very close, however, the required optimum doses of aluminum sulfate are higher than that of ferrous sulfate.

Daphnia magna toxicity test results showed that wastewater was toxic at the COD levels >211 mg L^{-1}. Even though significant COD removal and toxicity reduction efficiencies were achieved in both aluminum and ferrous sulfate coagulation experiments, the coagulated wastewaters were toxic at the COD levels >120 mg L^{-1}. In the coagulation experiments, color degradation was observed after effective COD removal. Also, the sulfate concentration increases with increasing coagulant doses. On the other hand, some metal complexes may form during coagulation [11,22,23]. Previous studies have shown that sulfate, dyes and metal complexes may result in a toxicity effect [4,5,24]. Thus, toxicity effect of the lower COD levels during coagulation may be attributable to any or conjugated toxicity effect of metal complex formations, color and sulfate concentration in coagulation process.

3.4. Application of pre-ozonation

A first order kinetic reaction described by Eq. 6 has been extensively used in the literature to fit the degradation kinetics of various organic compounds in water [9,13,25]

$$-dC/dt = k_1[C]. \quad (5)$$

Textile wastewaters is very complex due to the organic chemicals such as many different dyes, carriers,

Table 3
Treatment of raw wastewater with aluminum sulfate

$Al_2(SO_4)_3$ (mg L^{-1})	Absorbance (cm^{-1})			COD removal (%)	Toxicity reduction (%)
	436 nm	525 nm	620 nm		
0	1.06	1.37	0.77	0	0
250	0.98	1.32	0.69	11	0
500	0.79	1.24	0.48	NA	0
750	0.50	1.05	0.31	49	0
1000	0.37	0.79	0.26	NA	45
1500	0.30	0.55	0.21	56	70
2000	0.24	0.49	0.16	59	100

biocides, bleaching agents, complexion agents, ionic and non-ionic surfactants, sizing agents, etc. As a result, it is hard to explain the overall degradation of the organic matter by ozone in textile wastewater individually. Thus, some global textile wastewater parameters such as color, COD and dissolved organic carbon are used for the degradation kinetic of organic matter by ozonation [13].

In general, it has been stated that ozone cleaves the conjugated bonds of dye molecule, resulting in color removal and enhancing biodegradability. The cleavage of dye bonds takes place in ozonation process by both direct ozonation and radical pathways as explained with HSB model in literature [26].

$$O_3 + OH^- \rightarrow HO_2^{\cdot} + O_2^{-\cdot} \; k = 70\ M^{-1}\,s^{-1} \quad (6)$$

$$O_3 + O_2^{-\cdot} \rightarrow O_2 + O_3^{-\cdot} \; k = 1.6 \times 10^9\ M^{-1}\,s^{-1} \quad (7)$$

$$O_3^{-\cdot} + H^+ \leftrightarrow HO_3^{\cdot} \; pK_a = 10.3 \quad (8)$$

$$HO_3^{\cdot} \rightarrow OH^{\cdot} + O_2 \; k = 1.1 \times 10^5\ M^{-1}\,s^{-1} \quad (9)$$

$$O_3 + OH^{\cdot} \rightarrow HO_4^{\cdot} \; k = 2.0 \times 10^9\ M^{-1}\,s^{-1} \quad (10)$$

$$HO_4^{\cdot} \rightarrow O_2 + HO_2^{\cdot} \; k = 2.8 \times 10^4\ M^{-1}\,s^{-1} \quad (11)$$

$$2HO_4^{\cdot} \rightarrow H_2O_2 + 2O_3 \; k = 5.0 \times 10^9\ M^{-1}\,s^{-1} \quad (12)$$

$$HO_4^{\cdot} + HO_3^{\cdot} \rightarrow H_2O_2 + O_2 + O_3 \; k = 5.0 \times 10^9\ M^{-1}\,s^{-1} \quad (13)$$

Radicals especially hydroxyl radical are much more powerful than molecular ozone to oxidize various organics such as dyes molecules in aqueous solution. Radical reactions increase with increasing pH value. Mostly, the pH values of the textile wastewater were higher than 9.5. Thus, ozonation of raw textile wastewater was performed at the original pH (10) applying 24 mg L^{-1} ozone dose to 1 L wastewater sample. Fig. 2 displays color and COD degradation during ozonation. Over 80% of all the observed colors were removed in 10 min ozone contact time. A first order reaction kinetic rate expression was applied for the color removal in raw wastewater. It was found that $color_{436}$, $color_{525}$ and $color_{620}$ degradations obey first order kinetic giving 0.18 min^{-1}, 0.23 min^{-1} and 0.59 min^{-1} kinetic constant (k), respectively (correlation constant, $R^2 > 0.92$). Fluctuating COD degradation was determined during ozonation. It increased over time and reached 37% in 5 min but it decreased again following ozonation. Similar results reported by Lin and Lin in 1993 for COD and total suspended solid. These findings reflect the fact that first ozone converts dissolved solids into suspended solids but subsequent ozonation destructs high suspended solids into very small molecules which increase COD level in the ozonated wastewater. On the other hand, no fluctuation was observed for toxicity reduction. The toxicity

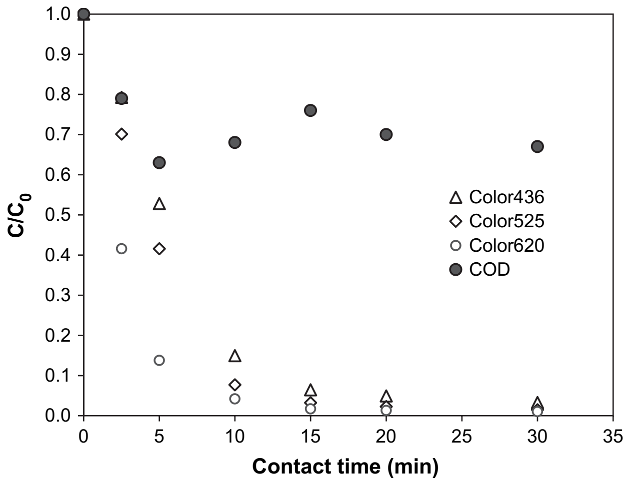

Fig. 2. Color and COD degradation as a function of ozonation time.

reduction was observed 10 min after color degradation and the ozonated wastewater was not toxic at 20 min (Fig. 2). As also reported in previous studies [16,18], these findings indicate that ozone was relatively effective in reducing the toxicity level of textile effluents.

Fig. 3 displays the amount of TrO_3 during ozonation experiment. Over 90% of all measured color absorbances were removed by the TrO_3 concentration of 53.7 mg L^{-1} in 10 min ozonation contact time, but the ozonated raw wastewater was still toxic up to the TrO_3 concentration of 71.4 mg L^{-1}. Toxicity reduction increased with increasing transferred ozone concentration and the ozonated wastewater was not toxic at TrO_3 concentration $\geq$ 82.3 mg L^{-1}. At this TrO_3 concentration, COD/TrO_3 ratio was about 7.0. The obtained different optimum TrO_3 concentrations for efficient color degradation and toxicity reduction reflect the fact that intermediate by-products which formed via color degradation during partial ozonation also have toxicity effects but their toxic effects were reduced at the prolonged ozonation time period [16–18].

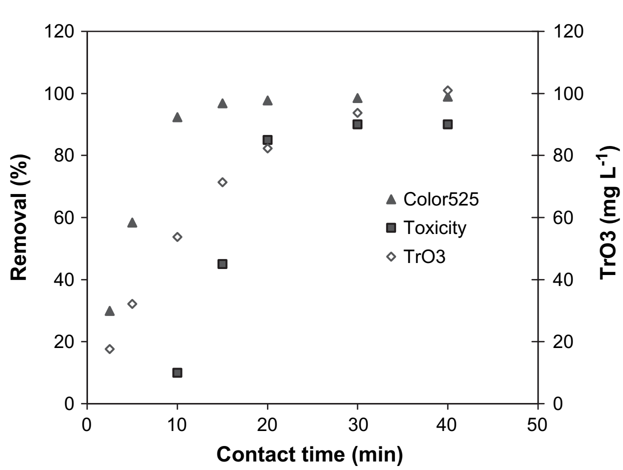

Fig. 3. TrO_3 during color and toxicity reduction.

Toxicity experiments were also carried out at different dilutions to evaluate ozone effect on the toxicity reduction. As can be seen in Fig. 4, the ozonation significantly reduced toxicity in the wastewater and the ozonated wastewater was not toxic at the dilutions $\geq$ 75% while untreated wastewater was found to be toxic up to 150% dilutions. On the other hand, both raw and ozonated wastewater were toxic at 50% dilution. The results indicate that COD level is one of the most important parameters to be considered for the toxicity control in textile wastewater. The raw and ozonated wastewater were toxic at the COD levels $<$211 mg L^{-1} and 232 mg L^{-1}, respectively. This result proved that ozone reduces the toxicity in the wastewater by not only reducing COD level but also by converting toxic chemicals such as dyes to non-toxic products.

4. Conclusions

Color removal (79%) was achieved using 1500 mg L^{-1} ferrous sulfate but due to the high chemical sludge production it seems coagulation was not applicable for color removal in the textile wastewater. On the other hand, optimum ferrous sulfate concentration of 1000 mg L^{-1} was obtained for toxicity removal and about 50% color absorbances, 59% COD and 80% toxicity were removed from raw wastewater.

The optimum aluminum sulfate concentrations for color absorbances, COD and toxicity removals were found to be 1500 mg L^{-1}, 1000 mg L^{-1} and 1500 mg L^{-1}, respectively. Once again, these optimum concentrations were not economical solutions due to the high sludge production, however, 60% color, 56% COD and 70% toxicity were removed from raw wastewater.

It is concluded that ozone is a very effective method for both color and toxicity reduction. In the ozonation experiments, the degradation of color at 436 nm, 525 nm and 620 nm wave lengths obeyed first order degradation kinetic model and almost complete color absorbances

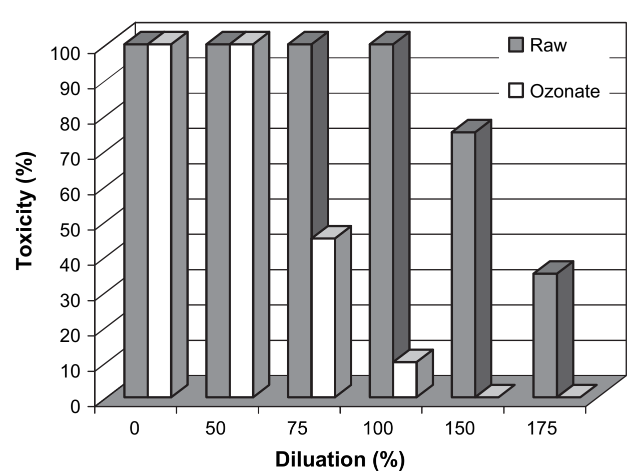

Fig. 4. Toxicity of raw and ozonated wastewater at different dilutions.

(over 98%) were removed in 20 min ozonation contact time period. The toxicity at 100% dilution reduced after color degradation. TrO_3 concentrations of 53.7 mg L^{-1} and 82.3 mg L^{-1} were determined for the optimum color degradation and toxicity reduction, respectively.

The ozonated wastewater was still toxic at 50% dilution due to the high COD level. The COD levels for toxicity removal were obtained as 211 mg L^{-1} in raw wastewater, around 120 mg L^{-1} in the coagulated wastewater and 232 mg L^{-1} in the ozonated wastewater. It is concluded that coagulation increases toxicity level of COD in the wastewater. However, toxicity reduced effectively at high coagulant doses due to the high DOC removal.

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