Sigma J Eng & Nat Sci 38 (3), 2020, 1179-1189

Sigma Journal of Engineering and Natural Sciences
Sigma Mühendislik ve Fen Bilimleri Dergisi

Research Article

COMPARISON OF OZONATION, ADSORPTION AND AIR STRIPPING PROCESS FOR AMMONIA NITROGEN REMOVAL FROM REAL TEXTILE WASTEWATER

Yalcin GUNES[1], Ferhat BARUT[2], Gul KAYKIOGLU*[3], Ali Riza DINCER[4]

[1]*Tekirdag Namık Kemal University, Department of Environmental Engineering, TEKİRDAG;*
ORCID: 0000-0001-8697-3345
[2]*Tekirdag Namık Kemal University, Department of Environmental Engineering, TEKİRDAG;*
ORCID: 0000-0002-1297-8781
[3]*Tekirdag Namık Kemal University, Department of Environmental Engineering, TEKİRDAG;*
ORCID: 0000-0003-3271-211X
[4]*Tekirdag Namık Kemal University, Department of Environmental Engineering, TEKİRDAG;*
ORCID: 0000-0002-9294-0643

Received: 10.03.2020 Revised: 01.04.2020 Accepted: 02.04.2020

ABSTRACT

In this study, the NH_4-N, organic nitrogen, color and COD removal were investigated in raw wastewaters (WW1, WW2, WW3) of a textile industry that performs cotton and polyester dyeing and printing processes. For this purpose, ozonation at neutral pH (pH 7.5) and at high pH (pH 10), powder activated carbon adsorption at pH 7 and air stripping process at high pH (pH 10) were used. The maximum removal efficiency for WW1, WW2 and WW3 in terms of NH_4-N was obtained by ozonation (99%) at high pH and by adsorption (96%). However, the maximum removal efficiency for NH_4-N obtained by the air stripping process was determined as 56%. Due to the difficulty of chemical hydrolysis of the organic nitrogen, the removal efficiency by ozonation and air stripping process was low.
As a result, ozonation of the raw wastewater at high pH values can be regarded as the most suitable method due to the high pH value of the industrial raw wastewater and higher removal efficiency and easy application according to other processes applied.
Keywords: Adsorption, aeration, ammonia nitrogen, ozonation, wastewater treatment.

1. INTRODUCTION

Conventional methods such as biological activated sludge and chemical treatment systems are generally used for treatment of wastewater of textile industry. The application and design stages of conventional treatment methods generally take into account organic carbon removal. However, in the textile industry, which carries out the printing process and especially uses a large amount of urea, nitrogen removal may not be sufficient by existing conventional treatment methods [1].

* Corresponding Author: e-mail: gkaykioglu@nku.edu.tr, tel: (282) 250 23 69
**This manuscript was presented at the 4th International Conference on Recycling and Reuse, 24-26 October, 2018.

In Turkey, NH_4-N discharge limit to receiving waters is 5 mg/L for textile industry according to Water Pollution Control Regulation. Nitrogen in discharged wastewater can cause serious environmental problems such as toxic algal blooms, fish deaths, decrease in oxygen level, decrease of biodiversity and decrease of aquatic plants and corals in receiving environments [2].

Nitrogen removal from wastewater is generally carried out by nitrification-denitrification, physico-chemical processes, ammonia stripping and ion exchange methods. The conventional nitrification-denitrification process is the most common process. However, not only long hydraulic retention time is needed but also high sludge age. This is due to the slow growth rate of microorganisms responsible for nitrogen removal. Excessive amounts of oxygen are consumed in nitrification.In denitrification, organic carbon is needed as electron donor and C / N ratio is important [3]. The operating conditions of this system (aerobic and anoxic) are difficult to maintain.

In chemical precipitation process, there is a need for different chemical substances and new pollutant forms are formed. The cost of selective organic resins used in the ion exchange method is very high. Due to these negative conditions in the removal of nitrogen from textile wastewaters, different nitrogen removal methods are needed. Depending on the characterization of wastewater, ozonation, adsorption and ammonia stripping can be used to remove nitrogen from textile wastewater.

The use of chemical oxidation with ozone application in the treatment of textile industry wastewater has started to attract a lot of interest in recent years. The application of ozone in the treatment of textile industry wastewater is usually done for two purposes. The first one is to provide the required color standard for the recovery of water which organic matter has been removed. The second is to make it suitable for biological treatment by oxidation of recalcitrant compounds in wastewater [3-6]. The oxidation rate of ammonia with ozone is quite slow and the reaction rate varies depending on pH. As the pH increases, the ammonia removal efficiency is also increased. The oxidation reaction of ammonia with ozone is as follows [7].

$$4O_3 + NH_3 \rightarrow NO_3^- + 4O_2 + H_3O^+ \quad (1)$$

Ammonia oxidation with ozone has previously been applied to various wastewater and successful results have been obtained. NH_4-N removal efficiency from aqueous solutions was determined as 85.2% by the application of catalytic ozone using magnesium oxide and cobalt oxides [8]. Luo et al. (2015)[9] studied to remove ammonia nitrogen by using two stage ozonation process from wastewaters with ammonia nitrogen concentration 100 mg/L. In the first stage of the application of 1 L/min ozone flow, the initial pH decreased from 11 to 6.63 and the NH_4-N removal efficiency was determined as 59.32%. In the second stage, over 85% efficiency was obtained with ozone application.

The adsorption is the process of collecting the dissolved materials in solution on a suitable interface, and it is possible to use for the removal of many pollutants (heavy metals, color, phenol, ammonia etc.). NH_4^+ removal with two different types of zeolite was evaluated. The best removal efficiencies were obtained for both zeolite in pH 5-6 (81-87.5 %), qm values were 13.3 mg/g and 16.2 mg/g [10]. In another study, Serezli and Tabak (2013)[11] studied the adsorption of NH_4^+ on to bentonite from the aqueous solution in laboratory conditions and stated that bentonite was used successfully in ammonium removal. Yunnen et al. (2016) [2]evaluated NH_4-N adsorption using activated sludge modified with iron hydroxide and observed that NH_4-N concentration decreased from 110 mg/L to 11 mg/L (qmax 32.7 mg/g, pH 7.8).

For the removal of NH_4-N from the dyeing process wastewater, adsorption experiments were performed on the column using granular activated carbon and zeolite and 60, 82% removal efficiency was obtained [12]. For the removal of ammonia nitrogen from solid waste landfill leachate, experiments were carried out in zeolite columns with different grain sizes and over 90% removal efficiencies were obtained.

Ammonia stripping is a process used for r emoval of some volatile materials from water and wastewater by aeration. The process for removal of NH_4^+ can be expressed as follows.

$$NH_4^+ \leftrightarrow NH_3 + H^+ \quad (2)$$

Ammonium ions in the wastewater are in equilibrium with ammonia and hydrogen ions. In equation (2) pKa is approximately 9.5. When the pH is increased, the reaction changes to the right side and a significant amount of non-ionized ammonia occurs. This occurs when the pH value is greater than the pKa value. Ammonia removal is carried out in two stages in ammonia stripping process. 1: the conversion of ammonium nitrogen to non-ionized ammonia by increasing the pH value and 2: separation of ammonia from the liquid according to Henry's Law [7].
More than 90% efficiency has achieved in various studies on the removal of ammonia from leachate and synthetic solutions (over pH 10) by ammonia stripping process [13-17].

In textile industry especially printing wastewater contains very high nitrogen concentrations (ammonia and organic nitrogen) due to the use of ammonia and urea as a hydrotropic agent in pigment printing pastes [18]. It may not possible to reduce the concentration of high ammonia nitrogen in the textile industry wastewater, to the limit values that can be discharged by conventional biological treatment methods. In this study, ozonation (pH 7.5 and 10), powder activated carbon adsorption and air stripping process (pH 10) were used to removal of NH_4-N from raw wastewater of cotton and polyester printing and dyeing industry. In addition to NH_4-N removal, organic nitrogen, color and COD removal efficiencies were determined and compared.

2. MATERIALS AND METHODS

2.1. Wastewater characteristics

In the study, untreated wastewater (WW1, WW2 and WW3) taken at different times from equalization tank of the wastewater treatment plant were used. The industry, where the wastewater used in the study is taken, carries out fabric printing and dyeing operations. In experimental study, NH_4-N, org-N, COD and color were measured according to Standard Methods [19]. The characterization of wastewater samples was given in Table 1.

As can be seen from Table 1, the pH value of the raw wastewater is ≈ 11. The NH_4-N (56.6 mg/L) and Org-N (750.9 mg/L) values for WW2 are higher than all wastewater. The color (2356 Pt-Co) and COD (1277 mg/L) parameters for WW3 are higher than all wastewater.

Table 1. Characteristics of wastewater samples

Parameters	Raw wastewater		
	WW1	WW2	WW3
pH	10.58	11.24	11.95
NH_4-N, mg/L	15.7	56.6	11.5
Org-N, mg/L	35.1	750.9	328.1
Color, Pt-Co	771	773	2356
COD, mg/L	640	894	1277

2.2. Ozone Oxidation

An ozone generator manufactured by Degremont with production rate of 2 g O_3 per hour was used to supply ozone. Ozone was produced from air and the capacity of the air pump was 10 L/min. Ozone was supplied to the reactor using a diffuser. Ozonation system was operated in semi-continuous type, i.e., continuous with respect to the gas flow and batch with respect to solution at room temperature (25 ^{O}C). 3 L wastewater was filled into 4 L stainless steel reactor. Samples were taken at 30, 60, 90 min for WW1, WW2 and WW3. Excess ozone gas (off gas)

passed out through the top of the reactor into gas-washing bottles containing KI solution to trap excessive ozone.

2.3. Adsorption

Adsorption experiments were carried out in batch conditions at 25°C using an orbital shaker at constant agitating speed of 250 rpm. The pH was 7 during experiments. The effect of contact time (0, 30, 60 and 90 min) on NH_4-N, org-N, COD and color removal were studied. Adsorbent dose was 20 g/L. At the end of the contact period, samples were taken from the supernatant and centrifuged at 3500 rpm for 5 minutes. Then the determined parameters were measured. The pH of the solution was adjusted using either 0.1 N NaOH or 0.1 N HCl. Powder activated carbon (PAC) was acquired from a commercial company and BET surface area was 686.52 m^2/g [20].

2.4. Ammonia Stripping

Ammonia stripping was carried out using 3 L sample volume in 5 L cylindrical reactor. The air capacity of the air pump was 10 L/min. and the air was given to the reactor using a diffuser. The pH was adjusted to 10 using 6 N NaOH. Samples were taken at 0, 30, 60 and 90 min. and parameters were measured.

2.5 Analytical Methods

Samples taken during wastewater characterization and treatment experiments; pH, NH_4-N, org-N and COD analyzes were performed according to Standard Methods. During the experiments, WTW pH 315 i brand pH meter was used for pH measurements. Color measurements (436, 525 and 620 nm) were performed using a PerkinElmer spectrophotometer [21]. All chemicals were of analytical grade.

The removal rate (R) was calculated by Equation (3) as follows:

$$R = \frac{C_o - C}{C_o} * 100 \quad (3)$$

where Co is the initial concentration and C is the concentration at reaction time t (min).

3. RESULTS AND DISCUSSION

3.1. Ozone Oxidation

Ozone oxidation was performed at neutral pH (pH 7.5) and high pH (pH 10) conditions. The results obtained and the removal rates are given in Table 2 and Figure 1 for WW1, WW2 and WW3.

In ozone application, the removal efficiencies obtained were higher at higher pH than at neutral pH for all parameters. In high pH conditions, ozone forms the free hydroxyl radical as a result of the following equations [9].

$$O_3 + OH^- \rightarrow HO_2^- + O_2 \quad (4)$$

$$O_3 + HO_2^- \rightarrow HO\bullet + O_2^- + O_2 \quad (5)$$

Table 2. Experimental results of ozonation

Time (min.)	NH_4-N (mg/L)	Org-N (mg/L)	Color (Pt-Co)	COD (mg/L)	NH_4-N (mg/L)	Org-N (mg/L)	Color (Pt-Co)	COD (mg/L)
WW1		**pH=7.5, Ozonation**				**pH=10, Ozonation**		
0	15.7	35.1	771	640	15.7	35.1	771	640
30	10.6	33.1	376	595	1.3	34.2	310	555
60	6.7	32.4	246	590	0.86	33.9	190	507
90	4.1	30.2	192	575	0.18	29.6	114	450
WW2		**pH=7.5, Ozonation**				**pH=10, Ozonation**		
0	56.6	751	773	894	56.6	751	773	894
30	35.7	693	378	692	24.9	692	300	779
60	25.6	692	314	692	16.7	687	220	639
90	19	675	276	675	7.4	660	127	560
WW3		**pH=7.5, Ozonation**				**pH=10, Ozonation**		
0	11.5	328	2356	1277	11.5	328	2356	1277
30	9.2	321	1245	1212	7.2	318	1042	1164
60	9.1	313	940	1100	5.6	309	503	1133
90	8.7	302	643	1100	3.3	292	250	988

The oxidation potential of HO• (2.80 v) is higher than ozone (2.07 v) [22]. Both ozone gas and OH radicals are effective in the oxidation process (Equations (6)–(13)) [9, 23].

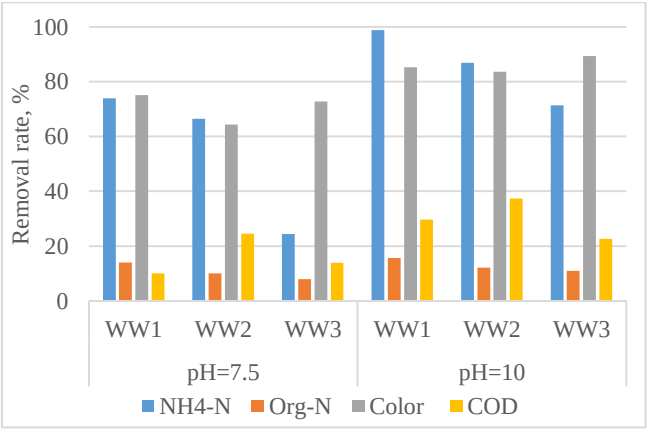

Figure 1. Comparison of removal efficiency for WW1, WW2 and WW3 by ozonation (pH=7.5 and pH=10)

$$3O_3 + NH_4^+ \rightarrow NO_2^- + 2H^+ + H_2O + 3O_2 \quad (6)$$

$$NO_2^- + O_3 \rightarrow NO_3^- + O_2 \quad (7)$$

$$4O_3 + NH_4^+ \rightarrow NO_3^- + 2H^+ + H_2O + 4O_2 \quad (8)$$

$$3O_3 + NH_3 \rightarrow NO_2^- + H+ + H_2O + 3O_2 \quad (9)$$

$$4O_3 + 3NH_3 \rightarrow NO_3^- + H^+ + H_2O + 4O_2 \quad (10)$$

$$6\ HO\bullet + NH_3 \rightarrow NO_2^- + H^+ + 4H_2O \quad (11)$$

$$NO_2^- + 2\ HO\bullet \rightarrow NO_3^- + H_2O \quad (12)$$

$$NH_3 + 8\ HO\bullet \rightarrow NO_3^- + H^+ + 5H_2O \quad (13)$$

Direct oxidation of ozone gas is dominant in acidic conditions, whereas oxidation with hydroxyl radical is dominant in high pH [9].

For WW1, WW2 and WW3, the NH_4-N removal efficiencies obtained as a result of ozone treatment for 90 minutes at neutral pH (pH 7.5) were 74%, 66% and 24% and at high pH were 99%, 87% and 71%, respectively. The highest ammonia nitrogen removal efficiency was obtained for WW1. The COD and the organic nitrogen concentration of WW1 are lower than WW2 and WW3. Considering that the organic nitrogen can be converted to ammonia nitrogen during oxidation, it can be said that ozone is used more efficiently in WW1 for ammonia oxidation. The organic nitrogen concentrations of WW2 and WW3 are much higher than WW1 hence, ammonia removal efficiencies were lower than WW1 due to the organic nitrogen which was converted to ammonia in the oxidation process. NH_4-N removal efficiencies for WW3 were lower than other wastewater. This may be attributed to the fact that the WW3 contained higher color (2356 Pt-Co) and COD (1277 mg/L) than other wastewater. It can be concluded that the ozone used for this wastewater (WW3) may be used for COD removal rather than ammonia oxidation.

By applying the ozone pre-treatment, the refractory materials become more biodegradable, thus improving the performance of biological treatment [24-27]. In this study, considering the ozonation process as a pre-treatment may increase the COD and nitrogen removal efficiency of the existing biological treatment plant.

The NH_4-N results obtained by high pH (pH 10) application were found to be high for WW2 only and were limited at 7.4 mg/L. This can be attributed to the fact that the initial NH_4-N concentration of WW2 (56.6 mg/L) is about 4 times higher than that of other wastewaters. As expected, in ozone application, at high pH, the color and COD removal efficiencies are also higher than the neutral pH.

3.2. Adsorption

In the adsorption study, when the NH_4-N removal is considered, the equilibrium state is reached at contact time of 90 minutes (pH 7). The results obtained and the removal rates are given in Table 3 and Figure 2 for WW1, WW2 and WW3.

Table 3. Experimental results of adsorption

Time (min)	NH_4-N (mg/L)	Org-N (mg/L)	Color (Pt-Co)	COD (mg/L)
WW1		pH=7.0, Adsorption		
0	15.7	35.1	771	640
30	6.2	25.5	369.3	350
60	0.74	24.4	239	275
90	0.56	23.7	239	180
WW2		pH=7.0, Adsorption		
0	56.6	751	773	894
30	25.1	564	404	326.6
60	11.2	549	146	223.3
90	9.7	529	77	140
WW3		pH=7.0, Adsorption		
0	11.5	328	2356	1277
30	8.2	265	1084	643
60	4.2	253	923	590
90	4.1	249	630.4	577

Ammonia nitrogen removal efficiencies are generally high and were determined as 96%, 83% and 64% for WW1, WW2 and WW3, respectively. The concentration of ammonia nitrogen in treated wastewaters with adsorption were 0.56 mg/L, 9.7 mg/L and 4.1mg/L for WW1, WW2 and WW3, respectively. The organic nitrogen removal efficiency for WW1, WW2 and WW3 was determined as 32-24%, the color removal efficiency was 90-69% and the COD removal efficiency was 84-55%.

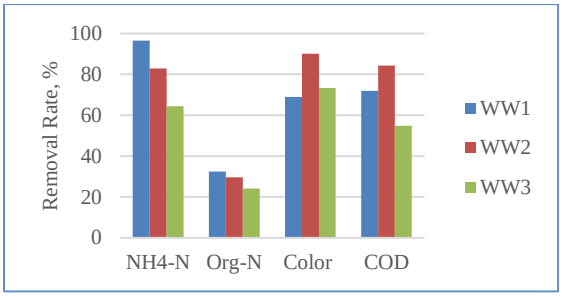

Figure 2. Comparison of removal efficiency for WW1, WW2 and WW3 by adsorption (pH=7)

For WW1, WW2 and WW3 the COD values decreased to 180, 140 and 577 mg/L respectively in adsorption. Therefore, it can be said that the as a pre-treatment process adsorption will affect the performance of biological treatment positively.

3.3. Ammonia Stripping

In high pH conditions (pH 10), ammonia stripping experiments were carried out for 90 minutes. The experimental results obtained and the removal rates are given in Table 4 and Figure 3 for WW1, WW2 and WW3, respectively.

In ammonia stripping method, particularly NH_4-N, organic nitrogen and COD parameters were decreased. However, no color removal was detected. NH_4-N removal efficiencies were obtained 52%, 56% and 30% for WW1, WW2 and WW3. The removal efficiencies obtained for the organic nitrogen parameter were determined in the range of 6-7% and it is estimated that organic nitrogen is hydrolysed to ammonia nitrogen during the process. The COD removal efficiency is about 10% depending on the removal of volatile organic substances.

Table 4. Experimental results of ammonia stripping

Time (min)	NH_4-N (mg/L)	Org-N (mg/L)	Color (Pt-Co)	COD (mg/L)
WW1	pH=10, Ammonia Stripping			
0	15.7	35.1	771	640
30	11.76	34.5	771	640
60	10.4	33.5	771	580
90	7.5	32.7	771	575
WW2	pH=10, Ammonia Stripping			
0	56.6	750.9	773	894
30	47.5	750.6	773	854
60	30.8	721.7	773	840
90	25.1	708.1	773	812
WW3	pH=10, Ammonia Stripping			
0	11.5	328.1	2356	1277
30	9.4	318	2356	1207
60	8.2	311	2356	1195
90	8	305	2356	1160

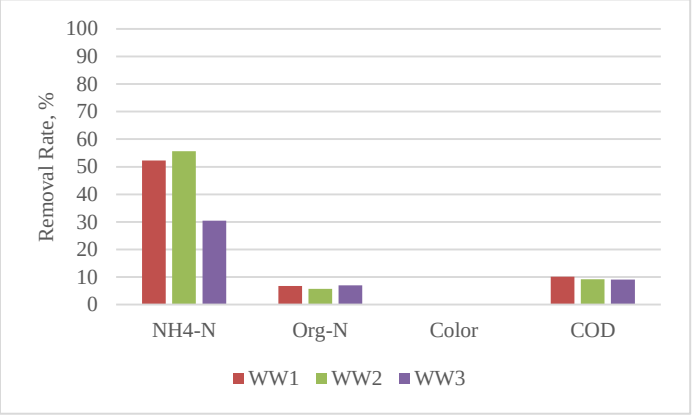

Figure 3. Comparison of removal efficiency for WW1, WW2 and WW3 by ammonia stripping (pH=10)

3.4. Comparison of treatment methods

The comparison of NH_4-N removal efficiency obtained by ozone, adsorption and ammonia stripping process is shown in the Figure 4. The maximum NH_4-N removal efficiencies were obtained by ozonation at pH 10 (99%) and adsorption (96%) while the maximum removal

efficiency obtained with the ammonia stripping application remained at 56%. Although an equivalent amount of air is given, the difference in removal efficiency between ozonation and ammonia stripping processes at high pH can be attributed to the oxidation power of ozone.

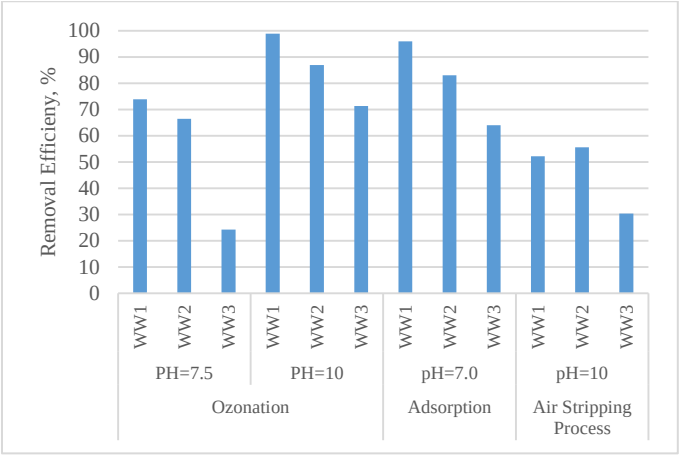

Figure 4. Comparison of NH_4-N removal efficiency obtained by ozonation, adsorption and ammonia stripping process

For all the parameters examined high removal efficiencies were obtained by ozonation at high pH conditions. pH values of the raw textile industry wastewater used in the study are in the range of about 11. Since no additional chemical consumption is required for pH adjustment, ozone application is advantageous for influent wastewater. In addition, as a pre-treatment of ozone, refractory materials become more biodegradable, thus improving the performance of the biological process.

When all parameters are taken into consideration, higher removal efficiencies have been obtained in the application of adsorption than ammonia stripping. However, in general, operating costs and the need for removal or regenerate of waste activated carbon are disadvantages of the application of adsorption.

Most of the organic nitrogen compounds coming into the system are formed by the urea used in the printing process, the other part is considered to be organic nitrogen compounds resulting from dyes and organic nitrogen from a small amount of domestic wastewater. Although it depends on the structure of organic nitrogen compounds, it is known that chemical hydrolysis of organic nitrogen is difficult. Therefore, organic nitrogen removal efficiency is higher in the adsorption process compared to ozonation and air stripping.

Except for the ammonia stripping at high pH, the color removal efficiency of more than 50% was obtained for all treatment processes. The highest COD removal efficiency was obtained by ozonation at high pH.

4. CONCLUSION

Textile industry wastewater may contain high concentrations of nitrogen and biological treatment systems sometimes do not meet the standards. For this reason, in some cases a pre-treatment may be required.

Ozonation of the raw wastewater at high pH values can be regarded as the most suitable method due to the additional advantages such as high pH value of the industrial raw wastewater, high removal efficiency and not producing additional waste.

In ozone oxidation, refractory materials become more biodegradable, thus improving the performance of the biological process. In addition, substances such as sulfur and sulphide, which can be found in textile wastewater and have a toxic effect on biomass, may also be oxidized by ozonation.

As the characterization of textile wastewater varies, long-term pilot scale studies should be performed before full scale applications and optimum conditions should be determined again.

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