Journal of the Taiwan Institute of Chemical Engineers 59 (2016) 359–364

Contents lists available at ScienceDirect

Journal of the Taiwan Institute of Chemical Engineers

journal homepage: www.elsevier.com/locate/jtice

Removal of fluorescent dissolved organic matter in biologically treated textile wastewater by ozonation-biological aerated filter

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ARTICLE INFO

Article history:
Received 7 January 2015
Revised 13 August 2015
Accepted 26 August 2015
Available online 29 September 2015

Keywords:
Textile wastewater
Ozone-biological aerated filter
Decoloration
Fluorescence excitation–emission matrix
High-performance liquid chromatography (HPLC)

ABSTRACT

Ozonation-biological aerated filter (O_3-BAF) has been proven effective for the biologically treated textile wastewater. However, little information on the behavior of dissolved organic matter in the process was available. In this study, a pilot scale O_3-BAF was set up to treat the biologically treated textile wastewater, and both fluorescent excitation–emission-matrix (EEM) and HPLC with multi-excitation/emission fluorescence scan were applied to study the removal characterization of fluorescent dissolved organic matter (DOM). The results showed that with the improvement of biodegradability by ozonation, the combination of ozonation and BAF could low the COD of the effluent below 50 mg/L, meeting the Chinese Discharge Standard. EEM analysis showed that the removal of the triple-excitation peaks at Em460 was the dominant mechanism of high removal efficiency of color and UV_{254} during ozonation process. In addition, during the ozonation process, the fluorescent DOM species were decomposed in the order of humic substances with Em at 460 nm (HS-Em460-Ex3), the hydrophobic aniline-like species, humic substances with Em at 430 nm (HS-Em430-Ex2) and the hydrophilic aniline-like species. This study provided better understanding on the decoloration mechanism of ozonation and helps to build automatic optimization systems in advanced treatment of textile wastewater.

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1. Introduction

Textile industry consumes substantial water and complex chemicals during various textile processing stages, and also generates a large quantity of hazardous wastewater that is high in color and toxic chemicals [1]. The direct discharge of such wastewater not only affects the aesthetic merits of the receiving water bodies, but also threatens the health of the aquatic systems and human beings by the possible toxicity and carcinogenicity [2–4]. Herein various techniques have been applied for the treatment of textile wastewater, such as biological degradation, adsorption, membrane separation, chemical coagulation and oxidation [2,5,6]. Due to its robustness, eco-friendliness and especially the lower costs, biological treatment is most widely used, in which the typical process is anoxic or anaerobic hydrolysis-acidification in combination with aerobic oxidation (A/O) [3,5,7]. And its removal mechanism has been clearly elucidated as anaerobic treatment for the reductive cleavage of azo bonds in dyestuffs with the following aerobic treatment for further degradation of the produced aromatic amines [3].

However, the textile wastewater contains different types of dyestuffs, which might have very low biodegradability and then result in the fluctuation of effluent quality [8,9]. Also, the biologically treated textile effluents containing large amounts of dissolved organic matter (DOM) still have deep color and high UV absorbance at 254 nm (UV_{254}), which can cause the light attenuation of the receiving water bodies [10,11]. Another important problem with the biological treatment is the residual aromatic amines in effluents, which could be more toxic and carcinogenic than their parent dyestuffs [12]. With the more stringent discharge standard, advanced treatment is quite necessary to achieve adequate level of decoloration and detoxification as well as chemical oxygen demand (COD) removal.

At present, advanced oxidation processes (AOPs) such as Fenton and ozonation seem to be the most desirable for the advanced treatment of biologically treated textile effluents [2,13]. As a kind of sludge-free AOPs, ozonation is increasingly promising because of the limited space and high expense for disposal of sludge [14]. Other merits of ozonation treatment are the improvement of biodegradability, reduction of aromatic compounds and also partially decrease of COD [15–17]. Typically, ozonation rarely causes complete mineralization of organic matter, but leads to partial oxidation products such as organic acids, aldehydes and ketones [14]. The biological aerated filter (BAF) is an alternative to the traditional activated sludge process,

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http://dx.doi.org/10.1016/j.jtice.2015.08.015
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Table 1
Characterization of textile wastewater after coagulation and sedimentation.

pH	Color (times)	UV_{254} (abs units)	COD (mg/L)	BOD (mg/L)
8.5–9	130–200	1.0–1.5	70–100	5–10

which is very effective in removing organic matter from wastewater that features relatively low levels of COD [18]. Ozonation in tandem with biological aerated filter (O_3-BAF) has been observed as one of the most economical and effective techniques for treatment of textile wastewater [18–21].

However, the previous works mainly use bulky characteristics like COD, biological oxygen demand (BOD), color, and UV_{254} to investigate the removal of DOM during ozonation treatment of textile wastewater [18–23]. Fluorescent excitation–emission-matrix (EEM) has been extensively applied for characterization of DOM in both natural and engineered water systems [24,25]. Recently, we firstly introduced HPLC/HPSEC with multi-excitation/emission fluorescence scan for DOM characterization and EEM interpretation [26,27]. For the textile wastewater, due to the electron-donating effect of amine groups, aromatic amines like derivatives of aniline or naphthylamine will exhibit strong fluorescence [28]; while ozone molecule preferentially attacks the unsaturated bonds of chromophores [15]. With the development of the field-portable fluorometer [29], it is very possible to realize the automatic optimization of ozone dosage. Herein it is meaningful to study the behavior of fluorescent DOM during the O_3-BAF treatment of textile wastewater, which can guide the selection of excitation and emission wavelengths for online monitoring.

In this work, the treatment efficiency of a pilot O_3-BAF for advanced treatment of textile effluents was studied. The EEM and HPLC with multi-excitation/emission fluorescence scan were further applied to investigate the behavior of fluorescent DOM during the O_3-BAF treatment. The results of this study help to understand the decoloration mechanism of ozonation and build automatic optimization systems in the advanced treatment of textile wastewater.

2. Material and methods

2.1. Chemicals and materials

Acetonitrile was of HPLC grade and purchased from Merck (Shanghai, China). Pure water from a Millipore system (Millipore, Bedford, MA, USA) was used in HPLC array. All other chemicals are of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). All reagents were used without further purification. Ceramsite balls are purchased from an environmental protection company in Henan Province, China.

2.2. Pilot O_3-BAF system

In this study, the pilot-scale O_3-BAF experiment was conducted in a textile mill located at Nantong city, Jiangsu Province, China. The textile mill engages in spinning, dyeing, weaving, finishing and so on. Many dyes, both azo dyes and non-azo dyes, such as reactive, disperse and acid dyes are utilized in the production process. The textile mill produces two streams of textile wastewaer. One is high-concentration wastwater with COD of 8000 mg/L and the flow of 1000 t/d and the other is low-concentration wastwater with COD of 800 mg/L and the flow 19000 t/d. High-concentration wastwater is mixed with low-concentration wastwater after up-flow anaerobic sludge bed (UASB) process. The mixed textile wastewater was treated by the typical A/O process in tandem with coagulation–sedimentation. The feeding stream for the pilot O_3-BAF system was from the effluent of coagulation–sedimentation. Table 1 lists the characteristics of the wastewater fed to O_3-BAF process.

The ozonation reactor consisted of ozonizer, ozone-online detector, contact reactor and off-gas absorption device**.** The ozonizer used compressed oxygen as the gas source with the gas flow rate of 0.6 m^3/h. Different ozone concentrations could be obtained by adjusting the voltage. Ozonation was performed in 6 series of polyvinyl chloride tubes (inner diameter 0.13 m, height 5 m), which is designed to improve utilization rate of ozone. After ozonation, the wastewater was aerated for about 10 min to eliminate residual ozone which might damage the microorganism in the BAF.

The upflow BAF reactor was made of carbon steel with an epoxy anticorrosive coat and the size of the reactor was 1.2 × 1.2 × 4.0 m^3 ($a \times b \times h$) with the volume of empty bed of 3.6 m^3. BAF reactor was packed with ceramsite balls ranging from 3 to 5 mm in diameter. Oxygen was introduced via compressed air through porous tubes located at the bottom of the BAF.

The BAF reactor was inoculated with activated sludge from the aerobic tank of wastewater treatment process in the textile mill. After a start-up stage of 20 days, the surface of ceramsite balls were attached with biofilm and microorganisms, and meanwhile, the COD removal increased to a steady level. Under the condition of hydraulic retention time 2.4 h and air–water ratio 5:1, the filter reactor was backwashed with gas–water once 2 weeks to remove the accumulated suspended solid and the excess biomass to keep the system operating efficiently and steadily. The schematic diagram and photograph of the pilot O_3-BAF is provided in Figs. S1 and S2 of the Supplementary Material (SM).

2.3. The kinetic experiment of ozonation

The kinetic experiment of ozonation was conducted in laboratory with sample from the same sedimentation tank. Ozone was generated from dry air using a laboratory model ozone generator with an ozone production of 2.5 mg/min. The ozonation was conducted in a 1 L cylindric glass reactor and samples were drawn at 0, 1, 2, 4, 6, 8, 10, 15, 20 min for analysis. The ozone dosage was determined by the reaction time. After ozonation, the wastewater was aerated for 5 min with air to eliminate the influences on the subsequent measurements from residual ozone.

2.4. Analytical methods

COD was analyzed by the standard method of potassium dichromate oxidation. Color was measured by the dilution multiple method and values are expressed as x° (0° indicates that the sample is as clear as pure water). The UV_{254} absorbance was measured with a Shimadzu UV-1800 ultraviolet−visible (UV–vis) spectrophotometer. Removal rate of color, UV_{254} and COD were calculated as:

$$\text{Removal rate}(\%) = \frac{C_0 - C}{C_0} \times 100\%$$

where C_0 and C are the initial and final values of color, UV_{254} and COD, respectively.

The EEM analysis was conducted on a Hitachi F-7000 spectrofluorometer. The slit widths of excitation and emission were both set at 5 nm. Excitation (Ex) wavelengths were conducted from 200 to 400 nm at 5 nm intervals and emission (Em) wavelengths were conducted every 1 nm from 280 to 550 nm with the scanning speed of 2400 nm/min.

After filtering with 0.45 μm membrane, samples were further analyzed by Agilent 1200 LC systems coupled with UV absorbance and fluorescence detectors (FLD) [28]. For the reverse-phase HPLC, an Eclipse XDB-C18 column (150 × 4.6 mm, 5 μm) was applied. The mobile phase was a mixture of acetonitrile and pure water, and the gradient elution procedure was set as shown in Table 2. According to results of EEM, FLD parameters were set as Em = 340/Ex = 220–300 or Em = 460/Ex = 220–400 for multi-excitation scan of protein-like

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