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Toxicity evaluation of textile dyeing effluent and its possible relationship with chemical oxygen demand

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ABSTRACT

Textile dyeing wastewater was the focus of much research because of its adverse effect on aquatic biota. In the present research, textile dyeing influent and effluent samples were collected from four textile dyeing wastewater treatment plants (TDPs) in Guangdong province, China, and their conventional indicators and toxicity were examined to reveal relationships. The relationship between toxicity and chemical oxygen demand (COD) was clearly established at individual TDPs. Results indicated the highest removal efficiencies of 94.4%, 90.6%, 91.9%, 94.6%, 92.8% and 97.5% for TOC, mixed-liquor volatile suspended solids (MLVSS), COD, ammonia nitrogen (NH_3-N), total phosphorus (TP) and colour, respectively. The primary clarifier used in TDP3 and TDP4 was beneficial for removing macromolecular organic substances, and membrane filtration and sedimentation basin employed at TDP1 and TDP2, respectively, helped to remove toxic substances. Toxicity to *V. fischeri* or *D. subspicatus* was found to be related to certain conventional indicators such as TOC, COD, TP, colour, and MLVSS, and was positively correlated with COD in different textile dyeing effluents ($R^2 > 0.84$). It was recommended that the relationship between toxicity and COD in wastewater should be established individually at each plant. Therefore, this study could be useful in providing suggestions for guiding effluent management when no toxicity experiments were conducted.

1. Introduction

Approximately 1.84 billion tons of textile dyeing wastewater was generated in China during 2015, which made it the third highest industrial wastewater in China, according to the China Environment Statistical Yearbook (2015). Textile dyeing wastewater was released from various textile dyeing manufacturing processes of soaping and rinsing, in addition to bleaching during cleaning of the textile factory (Prigione et al., 2008). It thus contained many toxic substances, such as detergents solvents, surfactants, dyes, and other recalcitrant organic matter (Lotito et al., 2012; Li et al., 2012). Chemical oxygen demand (COD), total phosphorus (TP), ammonia nitrogen (NH_3-N), mixed-liquor volatile suspended solids (MLVSS) and colour were considered common effluent discharging indicators. However, reducing these indicators to levels below given regulatory limits did not necessarily eliminate any toxicity of the wastewater (Ra et al., 2007; Sánchez-Meza et al., 2007), as some more highly toxic products could be generated after biodegradation (Farré et al., 2008).

Most research focused on conducting toxicity evaluations on pharmaceutical wastewater (Ma et al., 2016; Yu et al., 2014), municipal wastewater (Katsoyiannis and Samara, 2007), landfill leachates (Fan et al., 2006) and kraft mill effluent (Raptis et al., 2014), some analytical and toxicological investigations of textile dyeing effluent have been conducted. For example, Zhang et al. (2012) used zebrafish to test genotoxicity and acute toxicity in textile dyeing effluent and found that more highly acute toxicity and genotoxicity was observed in the effluent than in the influent. It was therefore necessary to evaluate the toxicity level of textile dyeing effluent to assess the possible toxic effect of releasing the effluent into receiving waters.

Studies have shown that the toxic effect from all kinds of toxic substances used in textile dyeing wastewater could be ascertained using bioassays, and that these could also be used to determine the effect of unknown substances that produce adversarial, additive and synergistic effects (Yu et al., 2014; Ma et al., 2016). There existed some studies on the toxicity of textile effluents with various species. For example, Mansour et al. (2011) evaluated the toxicity of textile dyeing effluent by *Pseudomonas putida*; Khlifi et al. (2010) assessed the textile dyeing wastewater by laccase-mediator system; and Sharma et al. (2007) evaluated the textile dyeing effluent by fish (*Gambusia affinis*) and duckweed (*Lemna aequinoctialis*). A unicellular algal assay (for example

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the use of *Desmodesmus subspicatus (D. subspicatus)*), which was a significant primary food producer in aquatic ecosystems, had also been used in water pollution control research, as higher ecosystem levels were affected by interferences in algal dynamics (Silva et al., 2009). In addition, *D. subspicatus* was easy to cultivate and could be used to acquire highly repeatable results. *Vibrio fischeri (V. fischeri)* was a luminescent marine bacterium that had been widely applied in toxicity evaluations due to its many merits: its use delivered a comparatively simple and cheap operation that was quick and reliable (Mendonca et al., 2007). In this study, therefore, *V. fischeri* and *D. subspicatus* were chosen and used as test organisms in the acute and chronic toxicity experiments conducted.

Correlating conventional indicators with effluent toxicity responses could provide support for effluent management when toxicity testing was not available. For example, significant negative relationships were observed between fish reproduction and biochemical oxygen demand (BOD) in kraft mill effluent that had undergone biological treatment (Kovacs et al., 2011), and a negative relationship was found between Vibrio fischeri or Daphnia pulex and COD in mixed industrial effluent (Araújo et al., 2005; Sánchez-Meza et al., 2007). In addition, Raptis et al. (2014) investigated kraft mill effluent and reported a negative correlation between the growth of Pseudokirchneriella subcapitata and reproduction of Ceriodaphnia dubia with increasing COD and BOD concentrations, respectively. Furthermore, Ma et al. (2016) found that toxicity in pharmaceutical wastewater was significantly and positively correlated with total nitrogen, COD, and TP concentrations. It would thus be beneficial to identify correlations between these conventional indicators and toxic substances in textile dyeing effluent, as no correlations in textile dyeing wastewater have been reported to date.

In this research, textile dyeing influent and effluent were collected from four textile dyeing plants (TDPs) in Guangdong province, China, and the associated conventional indicators and toxicity were examined. Toxicity was evaluated using *D. subspicatus* and *V. fischeri*, and the relationships between wastewater toxicity and discharge indicators were uncovered. Furthermore, the relationship between toxicity and COD at different TDPs was clearly established over one month of sampling. To the authors' knowledge, this was the first study conducted to distinguish the relationship between toxicity and COD in different TDPs within China.

2. Materials and methods

2.1. Wastewater sampling

To gain an understanding of the differences between the wastewater effluent at various TDPs in Guangdong province, China, four typical plants in Zhongshan, Dongguan, and Guangzhou, were studied. Basic information, including operating capacity, main dyes employed, textile material used and wastewater treatment process conducted at each of the four TDPs is presented in Table 1. The textile dyeing influent and effluent samples used for analysis of conventional indicators and wastewater toxicity were collected on July 23, 2017. 100 mL wastewater was sampled using grab samples every two hours by factories' workers during normal operation to ensure that variability in substances was included in analysis. The subsamples were cooled at 4 °C. The wastewater composite was placed in empty 5-L bottles, which were then completely filled with textile dyeing influent or effluent. To establish the relationship between toxicity and COD, these four TDPs were followed and investigated for nearly a month (from July 24 to August 21, 2017). 100 mL textile dyeing effluent was sampled using grab samples by factories' workers every day during normal operation. After sampling, the bottles were sealed and transported to the laboratory at 4 °C and subsequently analysed.

Table 1
Basic information about the four TDPs and different textile dyeing wastewater treatments used.

TDP	Operating capacity (m^3 /d)	Main dyes	Textile material	Wastewater treatment process
1	13,000	Mix dyes	Cotton	AD-AB-MF
2	11,000	Ionic dye, acid dye	Chemical fiber	AD-AB-SB-SF
3	13,000	Disperse dyes	Cotton	PC-AD-AB-SF
4	14,000	Ionic dye, disperse dyes	Cotton	PC-AD-AB-BSB-ESB-BAF

PC: primary clarifier; AD: aeration digester; AB: aeration basin; SF: sand-filtration; BSB: biochemical sedimentation basin; ESB: end sedimentation; BAF: biological aerated-filtration; MF: membrane filtration; SB: sedimentation basin.

2.2. Analysis of conventional indicators of wastewater

Conventional indicators of textile dyeing wastewater, including COD, NH_3-N, colour and TP, were measured using the Standards Methods (Chinese NEPA, 2002). The potassium dichromate method was used to measure COD concentration; NH_3-N was determined by the salicylic acid-sodium hypochlorite method; TP was determined by ammonium molydbate-potassium antimonyl tartrate using a spectrophotometer (Cary 100, Agilent); colour was measured via the dilution method. That is, 100–150 mL wastewater sample was placed in the beaker and its colour type under the background with a white porcelain plate was described. Then, the wastewater sample was diluted to different multiples. 50 mL sample was separated and placed in the 50 mL colorimetric tube with a white porcelain plate at the bottom of the tube. The colour of the diluted water sample was observed from the top down, and compared with the distilled water until the colour was not found. The dilution multiple was recorded at this time. MLVSS measurements were conducted using the standard methods (APHA, 1998) and a TOC-VCPH analyser (Shimadzu, Japan) was used to determine TOC in sample filtrate. All experiments were conducted in triplicate, and the averaged data were presented with a standard deviation.

2.3. Analysis of organic chemicals in textile dyeing effluent

500 mL textile dyeing effluent was filtered through a 0.22 μm organic filter membrane followed by liquid-liquid extraction using 50 mL dichloromethane at pH 2, 7, 10, respectively. The extract was collected and completely dried using a gentle stream of high-purity nitrogen. The sample was redissolved in 1.0 mL *n*-hexane and then injected into a gas chromatograph-mass spectrometer (GC-MS) for the direct determination of organic chemicals.

Instrumental determination of the organic chemicals was performed using a 7890 A GC-5975C MS (Agilent, USA). Chromatographic separation was achieved via an HP-5MS column (30 × 0.25 mm, 0.25 μm film thickness; Agilent, USA) with a constant flow rate of 1.0 mL/min. The operating conditions for the GC were as follows: oven temperature program initiated at 60 °C (held for 2 min), linearly increased to 240 °C at 20 °C/min (held for 2 min). Sample injection was done in the splitless mode, using an injection volume of 0.2 μL, and the time for solvent delay was set to 3 min. The MS was used under the following conditions: ion source temperature of 280 °C, transfer line temperature of 280 °C, and electron impact ionization of 70 eV. The MS was set in full scan mode (15–500 amu) for identification purposes. The organic chemicals were identified via their mass spectrum using the national institute of standards and technology (NIST) database. The characteristic ions found via the GC-MS matched the spectrogram with fit values higher than 90% obtained from the NIST database.

2.4. V. fischeri inhibition test

Prior to determining the acute toxicity of textile dyeing wastewater, luminescent bacteria *V. fischeri* were exposed to the textile dyeing