



Aromatic amine contents, component distributions and risk assessment in sludge from 10 textile-dyeing plants



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HIGHLIGHTS

- Fourteen carcinogenic AAs were detected in 10 textile-dyeing sludge.
- Monocyclic anilines were the predominant AAs in textile-dyeing sludge.
- AAs may pose a high risk to the soil ecosystem after being discarded or in a landfill.

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ABSTRACT

Aromatic amines (AAs), which are components of synthetic dyes, are recalcitrant to the wastewater treatment process and can accumulate in sludge produced by textile-dyeing, which may pose a threat to the environment. A comprehensive investigation of 10 textile-dyeing plants was undertaken in Guangdong Province in China. The contents and component distributions of AAs were evaluated in this study, and a risk assessment was performed. The total concentrations of 14 AAs ($\Sigma 14$ AAs) varied from $11 \mu\text{g g}^{-1}$ dw to $82.5 \mu\text{g g}^{-1}$ dw, with a mean value of $25 \mu\text{g g}^{-1}$ dw. The component distributions of AAs were characterized by monocyclic anilines, of which 2-methoxy-5-methylaniline and 5-nitro-o-toluidine were the most dominant components. The risk quotient (RQ) value was used to numerically evaluate the ecological risk of 14 AAs in the environment. The result showed that the 14 AAs contents in textile-dyeing sludge may pose a high risk to the soil ecosystem after being discarded on soil or in a landfill.

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1. Introduction

With the expansion of the textile-dyeing industry in China, the wastes produced by textile-dyeing plants (TDPs) are causing serious environmental problems (Liang et al., 2013; Vanhulle et al., 2008). According to the China Environment Statistical Yearbook, approximately 2.15 billion tons of textile-dyeing wastewater and 5.38 million tons of textile-dyeing sludge (80% moisture content) were produced in 2013. Zhejiang, Jiangsu, Guangdong, Shandong and Fujian Province produces approximately 4.35 billion tons of textile-dyeing sludge, account for 81% of total textile-dyeing sludge output in China. And Guangdong produced about 2.37 billion tons of textile-dyeing sludge in 2012. Textile-dyeing sludge is a very complex mixture, and the effluents of textile-dyeing plants are extremely difficult to treat due to their high content of pollutants such as polycyclic aromatic hydrocarbons, heavy metal

ions, surfactants, dyes, solvents, detergents and recalcitrant compounds (Vanhulle et al., 2008; Ning et al., 2014; Liang et al., 2013; Luo et al., 2013; Körbahti and Tanyolac, 2008). Pollutants can accumulate in sludge due to their refractory characteristics. Aromatic amines (AAs) in particular should be given more attention because AAs are essential precursors to and metabolites of azo dyes, the largest family of commercial synthetic dyes used today (Freeman, 2013). The removal of AAs from wastewater in textile-dyeing plants occurs through multiple physical, chemical and biological treatment techniques (van der Zee and Villaverde, 2005; Robinson et al., 2001). According to van der Zee and Villaverde, the removal rates of AAs in azo dye-containing wastewater generally ranged from 35% to 100% through sequential anaerobic-aerobic reactor systems. However, it is clear that AAs cannot be completely removed from wastewater due to the sorption of AAs to sediment and soil via physical and chemical processes (Vasudevan et al., 2013; Weber et al., 2001; Colón et al., 2002; Li et al., 2001). Twenty-two AAs have been prohibited by Regulation (European Union) 1907/2006, and 24 have been banned in China (GB/T 17592-2011) due to their toxic, carcinogenic and

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mutagenic characteristics (Chen, 2006; Porru et al., 2014; Chen et al., 2009; Xue et al., 2014; Ferraz et al., 2012; Rodricks, 1992).

Incineration and landfill are the two main disposal methods for textile-dyeing sludge in China. From a regulatory and environmental perspective, incineration is an effective method of stabilizing and reducing textile-dyeing sludge (Werther and Ogada, 1999), but harmful gases can be produced and released into the atmosphere during this process. Landfill also poses a threat to the terrestrial environment due to the AAs in textile-dyeing sludge. AAs in soil and sediment undergo irreversible binding through oxidative radical coupling and nucleophilic addition (Weber et al., 2001; Colón et al., 2002). Some ecotoxicological studies have shown that AAs might be cytotoxic (Chen, 2006; Wang et al., 2003). Thus, the textile-dyeing sludge should be pretreated to degrade the toxic organic contaminants (e.g. AAs) before disposal. However, there is a lack of detailed research on the contents and risks of organic contaminants to guide textile-dyeing sludge treatment and disposal.

In recent years, AAs have attracted increasing attention due to their adverse impact on the environment. Although the environmental content of AAs has been widely studied (Shabbir et al., 2015; Erdemir et al., 2009; Li et al., 2013; Jurado-Sanchez et al., 2012, 2013; Akyüz, 2008; Saha et al., 2009), investigations of the unintentional release of AAs from potential sources are not sufficient to determine the total emission of AAs. Guangdong Province is one of the most highly industrialized and urbanized provinces in China. Textile-dyeing is an important economic pillar industry, and TDPs are densely distributed there. Thus, large quantities of textile-dyeing sludge are produced, and a variety of toxic chemicals, including AAs, are released into the environment during sludge disposal. Therefore, textile-dyeing sludge has been listed as a Strict Control Waste in Guangdong Province. However, to date, the contents, component distributions and risk assessment of AAs in textile-dyeing sludge have not been investigated, even though the textile-dyeing sludge has seriously polluted the environment.

To address this general knowledge gap regarding this form of industrial waste, AAs in sludge samples from 10 TDPs were analyzed to investigate their contents and component distributions. A risk assessment of AAs in textile-dyeing sludge was also conducted, which will be very helpful for future studies. To our knowledge, exclusively, this is the first effort to comprehensively assess the risk of AAs in textile-dyeing sludge in China.

2. Materials and methods

2.1. Sampling

Ten sludge samples were collected from different representative TDPs in Guangdong Province, China (Fig. 1), from July to August 2014. Basic information on the TDPs in terms of textile material (chemical fiber or cotton), dyes (reactive dyes, disperse dyes, acid dyes or ionic dyes) and the wastewater treatment process (physical-chemical biological treatment or biological physical-chemical treatment) utilized is given in Table 1. Three textile-dyeing sludge sub-samples taken after dewatering were collected from the final storage container of each TDP over three days and mixed thoroughly to form one composite sample. All samples were obtained when the textile-dyeing wastewater treatment plant ran normally. The samples were wrapped with aluminum foil, packed in a plastic bag and then stored in a refrigerator at $-20\text{ }^{\circ}\text{C}$ until analysis.

2.2. Standards and chemicals

All reagents including methanol and methyl tert-butyl ether were HPLC grade and supplied by Fisher Scientific (USA).

Twenty-four AAs were analyzed, including o-toluidine (o-T), 2,4-dimethylaniline (2,4-DMA)/2,6-dimethylaniline (2,6-DMA), 2-aminoanisole (MOA), p-chloroaniline (PCA), 2-methoxy-5-methylaniline (MMA), 2,4,5-trimethylaniline (TMA), 4-chloro-o-toluidine (CT), 2,4-toluenediamine (TDA), 2,4-diaminoanisole (DAA), 2-naphthylamine (NA), 5-nitro-o-toluidine (NT), 4-aminobiphenyl (ABP), 4-aminoazobenzol (AAB), 4,4'-diamino diphenyl ether (DDE), benzidine (BNZ), 4,4'-diamino diphenyl methane (DDM), o-aminoazotoluene (OAAT), 3'-dimethyl-4,4'-diaminobiphenylmethane (DMDAB), 3,3'-dimethyl benzidine (DMB), 4,4'-thiobisbenzenamine (TOA), 3,3-dichloro benzidine (DCB), 4,4'-methylene-bis(2-chloroaniline) (MBCA) and 3,3'-dimethoxybenzidine (DMOB), each at the concentration of $300\text{ }\mu\text{g/mL}$. All chemical standards were obtained from O2si Smart Solutions (Charleston, SC, USA).

2.3. Sample pretreatment

Microwave-assisted extraction was used to analyze the AAs in the textile-dyeing sludge because it enables the rapid extraction of solutes from solid matrices and has a higher extraction efficiency than that of traditional techniques (Camel, 2000).

Approximately 1.00 g dry weight (dw) of each sample was extracted with methanol at $100\text{ }^{\circ}\text{C}$ for 10 min using a microwave-assisted extractor (MARS, USA). After extraction, the methanol extract was filtered and concentrated to approximately 1 mL by rotary evaporation, redissolved in 16 mL 0.6 M citrate buffer solution at $\text{pH}\sim 6$ and then subjected to cleanup using an Extrelut liquid-liquid extraction column (18 g, 60 mL) and stayed in the column for 15 min. The column was eluted with $4\times 20\text{ mL}$ methyl tert-butyl ether, and the eluate was concentrated to approximately 1 mL by rotary vacuum evaporation. And then the eluate was concentrated to 0.2 mL under a gentle flow of nitrogen with an N-EVAP 112 (Organomation, USA). The final extract was injected into the GC-MS for quantification.

2.4. Instrumental analysis

Instrumental determination of contents of AAs was performed using an Agilent 7890A gas chromatograph-5975C mass spectrometer (GC-MS, Agilent, USA). Chromatographic separation was achieved by a HP-5MS column ($30\text{ m}\times 0.25\text{ mm}$, $0.25\text{ }\mu\text{m}$ film thickness, Agilent, USA) with constant flow rate of 1.0 mL/min . The operating conditions for GC were as follows: oven temperature program initiated at $60\text{ }^{\circ}\text{C}$ (held for 1 min), linearly increased to $220\text{ }^{\circ}\text{C}$ at $8\text{ }^{\circ}\text{C/min}$ (held for 4 min) and further increased to $280\text{ }^{\circ}\text{C}$ at $12\text{ }^{\circ}\text{C/min}$ (held for 5 min). The sample injection was done in the splitless mode (1:10 ratio), using an injection volume of $1\text{ }\mu\text{L}$ and the time for solvent delay was set to 3 min. The mass spectrometer was used in the following conditions: ion source temperature, $230\text{ }^{\circ}\text{C}$; transfer line temperature, $250\text{ }^{\circ}\text{C}$; electron impact ionization mode, 70 eV . The MS was set in full scan mode ($35\text{--}350\text{ amu}$) for identification purposes. For quantification (SIM mode) the base peak ion was selected and two additional qualifiers ions were monitored for confirmation.

2.5. Quality assurance and quality control (QA/QC)

Quality assurance and control measures were strictly performed to ensure precise identification and quantification. A chromatogram of the AAs in the standard solution is shown in Fig. 2, and the ions and retention times are shown in Table S1. The obtained data are almost identical to those of previous studies (SN/T 3045-2011).

Procedural blanks were routinely analyzed for every batch of samples, and no analyte was detected in any instance. A

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