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Removal of volatile organic compounds (VOCs) emitted from a textile dyeing wastewater treatment plant and the attenuation of respiratory health risks using a pilot-scale biofilter

Zhishu Liang, Jijun Wang, Yuna Zhang, Cheng Han, Shengtao Ma, Jiangyao Chen, Guiying Li*, Taicheng An

Guangdong Key Laboratory of Environmental Catalysis and Health Risk Control, Guangzhou Key Laboratory of Environmental Catalysis and Pollution Control, School of Environmental Science and Engineering, Institute of Environmental Health and Pollution Control, Guangdong University of Technology, Guangzhou, 510006, China

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ABSTRACT

The textile dyeing is a significant worldwide economic pillar of the industry mainstay of the region's industrial economy. Therefore, significant attention should be paid to its emitted volatile organic compounds (VOCs) regarding their potential adverse effects on ecosystems and human health. In this study, a spray tower (ST) (preferential elimination of hydrosoluble VOCs) and biofilter (primary removal of particle-free VOCs) were used in combination. Three techniques, including gas chromatography-mass spectrometer (GC-MS), e-nose, and Proton transfer reaction time-of-flight mass spectrometry (PTR-TOF-MS) were used to accurately identify the associated VOCs profile. In total, 50 types of VOCs, with total concentrations ranging from 1.26 to 2.79 mg m^{-3}, were detected from the outlet of the TDWTP over a 90-day treatment period. The highest level occurred for nitrogen- and oxygen-containing compounds (NAOCCs), followed by aliphatic hydrocarbons (AIHs), aromatic hydrocarbons (AHs), and halogenated hydrocarbons (HHs). Average removal efficiencies (REs) of the four studied VOC groups (NAOCCs, AIHs, AHs, and HHs) were 66.7%, 67.9%, 11.7%, and 52.1%, respectively. Proteobacteria dominated the biofilter, followed by Actinobacteria, Firmicutes, and Bacteroidetes. A positive correlation was observed between the relative abundance of Proteobacteria and the RE of NAOCCs, with RE significantly increased from 38.1% (day 1) to 83.2% (day 90). The degradation of these organic pollutants, such as NAOCCs, may be mainly performed by the dominant genus, such as *Acidithiobacillus* and *Metallibacterium*, as predicted by PICRUSt. A respiratory health risk evaluation demonstrated that the cancer and non-cancer risks of typical VOCs were dramatically reduced after the above-mentioned purification. The combined results indicate that the ST-biofilter is an efficient approach for the end-of-pipe treatment of exhausted gas in TDWTP and helps attenuate human health risk.

1. Introduction

As a key industry in China, the textile industry plays a vital role in boosting the national economy. In 2017, the value of textile exports of China reached approximately 110 billion U.S. dollars, represents 37.2% of the global market share, followed by the European Union and India (Rovira and Domingo, 2019). As with other industrial activities, the textile industry discharges toxic substances to the environment, generating significant environmental pollution, particularly with respect to water pollution and emitted waste gases from wastewater. Based on the China Environment Statistical Yearbook, more than 1.8 billion metric tonnes of textile-dyeing wastewater was produced in 2015, containing numerous dyes, heavy metal ions, solvents, and other recalcitrant pollutants (Liang et al., 2017). Although, the wastewater can be purified using many technologies, including physicochemical, biochemical, and combined treatment processes (Esther et al., 2004; Khandegar and Saroha, 2013), volatile organic compounds (VOCs) such as benzene, xylene, toluene, and esters presented in the fabrics, surfactants, detergents, and solvents are emitted into the environmental

* Corresponding author.
E-mail address: ligy1999@gdut.edu.cn (G. Li).

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system during wastewater treatment in textile-dyeing plants (He et al., 2012; Ning et al., 2015). A great deal of these VOCs like benzene have carcinogenic, teratogenic, and mutagenic properties (An, 2004; Li et al., 2013), therefore raised significant concerns about the health of workers and the communities living in areas adjacent to and surrounding textile-dyeing plants.

The evolution of public awareness, environmental protection policies, and managerial and engineering approaches are drivers for developing effective VOC treatment technologies (Barbusinski et al., 2017). Conventional chemical and physical technologies have the advantages of having a rapid startup period and efficient removal rates. However, they are relatively costly both in investment and operation in terms of energy, material and reagents consumption (Estrada et al., 2011), and could generate hazardous byproducts (Kennes and Veiga, 2010). Biological odor abatement technologies including biofilters, bioscrubbers, and other bioreactor types are robust and reliable alternative methods with low construction as well as operation costs (Dobslaw et al., 2017; Estrada et al., 2012). They have the potential to ultimately convert the pollutants into innocuous oxidation products (e.g., H_2O, CO_2, sulfate, and nitrate) by the versatile microorganisms (Barbusinski et al., 2017).

Given the significant benefits of biofiltration, including ecological cleanliness, easy operation, simple construction, high efficiency, and low investment, a great deal of attention has been recently paid to its application in eliminating gaseous pollutants (Barbusinski et al., 2017). For example, biofiltration has been shown to be an excellent way to effectively reduce the concentration of single compounds, such as styrene, toluene, and xylene, and also mixtures of different VOCs at the laboratory level, pilot-scale and industrial practice (Hazrati and Shayegan, 2016; Hazrati et al., 2015; Khoramfar et al., 2018). However, every single technology has disadvantages, and the biofilter has difficulty metabolizing hydrophobic VOCs like alkanes because of the massive transfer limitations (Cheng et al., 2016a).

To overcome this problem and enhance the bioavailability of compounds so that the required contact times and construction costs will be reduced, waste gases need to be pretreated before using this biofiltration method (Dobslaw et al., 2017; Yu et al., 2014). The spray tower (ST), using H_2O as a solvent, is an extensively used control strategy to humidify hydrophobic air. In this way, the potential of the hydrophobic VOCs to be degraded in the biofilter will be increased. It can also be used to remove particles and hydrophilic pollutants from industrial effluent gases, which can prevent the clogging of the packed bed. For example, Chen et al. demonstrated that ST could efficiently remove different sized particles and some water-soluble atmospheric pollutants, such as ethyl acetate, from industrial effluent gases (Chen et al., 2017; Liu et al., 2019). As such, integrating biofiltration with ST could be a better way to reduce the level of emitted VOCs and attenuate the corresponding health risk.

Typical application of biofilter is aimed to remove low level odorous amino-nitrogen and sulfur compounds, AIHs, AHs, and HHs emitted from publicly owned treatment facilities, biosolids composting (Iranpour et al., 2010) and organic waste treatment plants (Dobslaw et al., 2018). However, until now, no research has been conducted on the biological removal of VOCs from textile-dyeing wastewater treatment plant (TDWTP). There have also been few studies exploring the correlation between the microbial community structures in bioreactor and VOC mixture removal (He et al., 2012; Yu et al., 2014), even though microorganisms are the engine of this process (Liao et al., 2015). Moreover, the microbial community will shift as the variation of concentrations and compositions of the VOCs (Hu et al., 2016; Pérez et al., 2016). Thus, it is essential to gain insight about the response of microbial community to the elimination efficiencies of different VOCs.

Here, a field-scale integrated technique of ST with biofilter was specifically designed to control VOC emissions from a TDWTP and to attenuate the corresponding health risks. The first objective of this study was to analyze the components and levels of VOCs released from the TDWTP to provide the baseline data for the follow-up treatment. The second objective was to assess the VOC removal efficiency (RE) of the integrated technique, for over 90 days. The third objective was to examine the response of the bacterial community composition and diversity in the biofilter to the RE of different VOCs. In addition, the study also evaluated the health risk of the emitted VOCs on human beings before and after the treatment with the integrated ST-biofilter. The obtained data provide useful guidance related to managing VOC pollution and attenuating health risk in the textile industry.

2. Materials and methods

2.1. Experimental site and setup

The experimental site was selected at a textile company in Guangzhou city (Guangdong, China), which is mainly engaged in producing fabrics. The production cycle includes washing, scouring, bleaching, mercerizing, dyeing and finishing. These steps, especially washing and finishing, produce large amount of wastewater with high toxicity (Li et al., 2012; Ning et al., 2015). Tertiary treatment includes catalytic ozonation, zeolite biological aerated filters, and membrane filtration are used to remove the 5-day biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), suspended solids, and color from the dye wastewater (He et al., 2013). However, the contact of organic pollutants in surfactants, detergents, and solvents with the ambient air resulted in VOC emissions during wastewater treatment process.

To purify the emitted VOCs, pilot-scale experiments were carried out in a mid-scale custom-made integrated reactor (power: 33 kW, size (length (L) × width (W) × height (H)): 10 m × 8 m × 2.6 m, made by Guangdong Nanfang Environmental Protection Bio-technology Co., Ltd.) (Fig. 1). The reactor consists of two adjacent units: ST and biofilter. Waste gases emitted from TDWTP first flowed through the ST (L × W × H: 2 m × 8 m × 2.6 m) and then flowed to the biofilter (L × W × H: 8 m × 8 m × 2.6 m) with a rate of 22000 $m^3\ h^{-1}$ using a centrifugal fan mounted before the integrated reactor. To speed up biofilter start-up, the mixed microbial consortium obtained from Guangdong institute of microbiology was pre-enriched into nutrient broth under the condition of 30 °C and a pH range of 7.0–7.5. Then, the culture was sprayed from the top of biofilter with the inoculate rate of 1 L for every 1 m^3 packing material. The leachate was collected in a tank and was then continuously re-circulated for the next 24 h to guarantee microorganism attachment. After that, the water pump sprayed water in the biofilter unit at a flow rate of 10 $m^3\ h^{-1}$ and the watering frequency is 24 times a day each for 1 min. At the same time, waste gases with different VOCs were passed through the system for the acclimation of the microorganisms with the initial empty bed residence time (EBRT) of ST and biofilter of 5.9 and 15.2 s, respectively. The full experiment lasted for 90 days and the average pressure drop is approximately 800 pa, suggesting that no clogging or breakdown problems occurred during the 3-month period. More detail about the experimental setup can be found in the supporting information (SI).

2.2. Sample collection and analysis

2.2.1. Electronic-nose (e-nose)

A Portable Electronic Nose PEN3.5 (Win Muster Airsense (WMA) Analytics Inc; Schwerin, Germany) was used for the rapid on-line

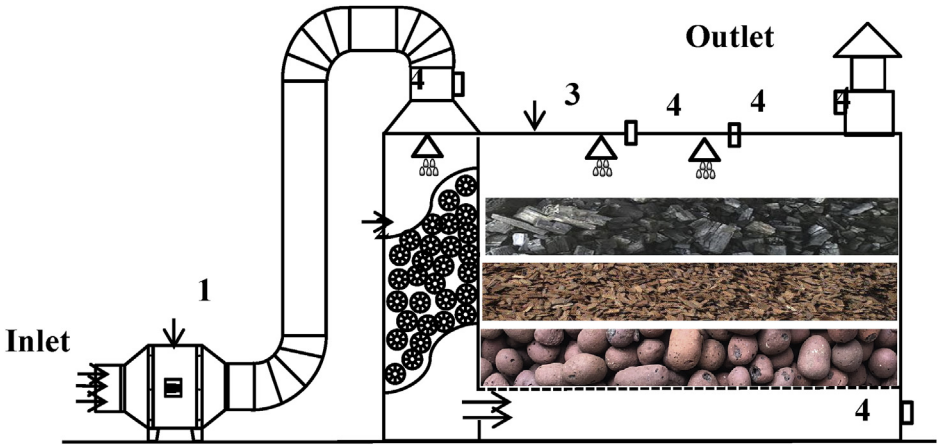

Fig. 1. Schematic diagram of integrated equipment of spray tower with biofilter. (1: centrifugal pump; 2: spray tower with multi-faceted hollow ball; 3: biofilter with packing materials (ceramic particles, wood chips, granular bamboo charcoal), 4: sampling points).

detection of odorous VOCs. The system consists of a detector unit containing the array of 10 different doped semi-conductive metaloxide gas sensors (MOS) (Table S1), a sampling apparatus, and a pattern recognition software (Baietto et al., 2010). The analysis procedure for the VOC mixtures was similar to previous studies (Baietto et al., 2010) and is summarized as follows. First, at the beginning of each run session, the instrument was pre-warmed for 10 min. Then, the samples were run based on the following procedure: sensor cleaning for 200 s and sampling run time for 60 s. All measurements were performed with air as the carrier gas and were run at a flow rate of 200 mL min^{-1}. The data from the sensor array were collected at 1-s intervals and statistically compared using normalized sensor outputs. Principal component analysis (PCA), which allowed the extraction of the most important information from the data was performed using Winmuster software to discriminate the VOCs before and after treatment (Benedetti et al., 2008).

2.2.2. Gas chromatography-mass spectrometer (GC-MS)

Further, an off-line GC-MS analysis was carried out for evaluation of the VOC levels in the TDWTP and the VOCs eliminating ability of the integrated technique. First, gaseous samples were collected on day 1 and 20 during summer, and on day 90 during winter (designed as 1st, 2nd, and 3rd) using 2.7-L Entech vacuum Summa canisters. Then, VOCs were analyzed using a 7200 preconcentrator (Entech Instruments Inc., CA, USA) equipped with Agilent GC (7890B)-MS (5977B) with US Environmental Protection Agency (USEPA) TO-15 method (USEPA, 1999). A control point at approximately 10 m outside of ST-biofilter was selected. Detailed sampling and analysis methods are provided in the SI and can be found in a previous study (He et al., 2012).

2.2.3. Proton transfer reaction time-of-flight mass spectrometry (PTR-TOF-MS)

To provide a complete pollution profile, VOCs were also analyzed with a commercially available Ionicon PTR-TOF-MS 1000 (Innsbruck, Austria). Detailed measurement principles are provided in previous publications (Cappellin et al., 2012b; Han et al., 2019; Huang et al., 2016). Briefly, the drift tube temperature was 80 °C, drift voltage was 603 V, drift pressure was 2.3 mbar, and the sampling time per channel was set to 1000 ms. The resulting E/N (N is the number density of a neutral gas and E is the electric field strength) was approximately 133 Townsend (Td, 1 Td = 10^{-17} V cm^2). The PTR-TOF-MS spectra were acquired at a frequency of 0.5 Hz in real time mode for different known mixing ratios using version 1.2.94 TofDaq data acquisition software (Tofwerk AG, Thun, Switzerland).

Together, the three technologies described above enabled the accurate detection and precise identification of odorous VOCs released from the TDWTP.

2.3. Performance evaluation of ST-biofilter reactor and health risk assessment

Health risk minimization (via ST-biofilter) and efficient end-of-pipe VOCs treatment both address odor nuisance management, especially when odorants have been produced and released from the wastewater. As such, it was urgent to evaluate the reactor performance in terms of RE (%), using the detailed equations provided in SI. Moreover, long-term exposure to the VOCs emitted is related to significant harmful effects on both public and occupational health (Zhang et al., 2018). Thus, health risks, including non-cancer and cancer VOCs exposure risks, were evaluated to provide a base line for protecting public health from air pollution, especially for workers (Payne-Sturges et al., 2004). Detailed calculation methods are also described in SI.

2.4. Characterization of bacterial community diversity by high-throughput sequencing

High-throughput sequencing was used to determine how bacterial communities in the biofilm of packing materials respond to the exposure of contaminants. Samples of the packing materials in the middle of the biofilter and circulating leachate were collected during treatment operations on day 1, 20, and 90, and were used to extract the genomic DNA using a Fast DNA Spin Kit (MP Biomedicals, USA) according to the manufacturer's instruction. The extracted DNA was amplified using degenerate primers (515 F: GTGCCAGCMGCCGCGGTAA and 806R: GGACTACHVGGGTWTCTAAT), which was targeted the V4 region of 16S rRNA gene. Sequencing was carried out using the Illumina PE 300 platform by Majorbio Bio-PharmTechnology Co., Ltd. Additional details, such as the PCR amplification and the data analysis pipeline, are summarized in the SI. The sequences obtained from high-throughput analysis were deposited in the NCBI Sequence Read Archive with the accession number SRP214393.

The above effective sequences were clustered into operational taxonomic units (OTUs) at a 0.03 (97% similarity) distance level.

Then, a bacterial taxonomic classification of each OTU was performed using the Mothur program against the SILVA 128 database. With RDP classifier, a taxonomic category was assigned to all OTUs at 0.5 confidence threshold (Zhai et al., 2017). For the alpha diversity analysis, samples were normalized to the lowest number of sequences reads obtained. The diversity of microbes was estimated using Simpson index and Shannon index; their richness was calculated using Chao1 and ACE index. Microbial community abundance at Phylum and Genus taxonomical levels were revealed using Heatmaps. Finally, statistical analysis and corresponding graphs were performed using the Statistical Analysis of Metagenomic Profiles (STAMP) software to assess the dissimilarity among the samples at different treatment times (Parks and Beiko, 2010). The metagenome content and the KEGG pathway were predicted using PICRUSt (Langille et al., 2013).

3. Results and discussion

3.1. The levels and composition of VOCs before and after treatment

Given many VOCs including ethyl alcohol (0–4.6 mg m^{-3}), methyl alcohol (0–979 mg m^{-3}), benzene (0–35.6 mg m^{-3}), methyl acetate (0–1351 mg m^{-3}), cyclohexane (0–51.2 mg m^{-3}), xylene (0–20.7 mg m^{-3}), and trichloroethylene (0–18.11 mg m^{-3}) were found in the atmosphere of chemical fiber wastewater plants (Yang et al., 2019). In this study, the fugitive emission of VOCs from TDWTP were collected into a pipe using negative pressure by centrifugal fan. These VOCs especially AIHs and HHs were powerfully identified and reliably quantified using GC-MS (Aprea et al., 2009; Cappellin et al., 2012a). Furthermore, since the newly available PTR-TOF-MS technique may complement GC-MS results by providing quantitative information on low molecular mass important metabolites, such as acetaldehyde, methanol, sulfur compounds, ethanol, and many others (Cappellin et al., 2012a), the samples were also measured using PTR-TOF-MS to identify compounds, which were difficult to be detected by a single GC-MS analysis because of their low volatility. As Table S2 shows, a total of 50 kinds of VOCs, grouped as AHs, AlHs, HHs and NAOCCs, were identified and quantified. The total concentrations of the emitted VOCs ranged from 1.26 to 2.79 mg m^{-3} on day 1, 20 and 90 (Fig. 2a). These levels were significantly higher than the atmospheric VOCs at the control site (Table S3), indicating that the atmosphere of TDWTP was highly polluted by VOCs and the air needs to be purified before direct emissions are allowed. Among the emissions, NAOCCs were present at the highest levels (85.1% in average), followed by AIHs (7.1%), AHs (6.3%), and HHs (1.5%) (Fig. 2b). These suggested that NAOCCs were the key pollutants in TDWTP that needed to be reduced. The significantly different VOC concentrations might be due to the widely use of surfactants, dyes, detergents, and solvents in the textile-dyeing industry (Liang et al., 2017).

The analysis of typical VOCs compositions volatilized from the TDWTP showed that the main AHs on day 1 and 20 were very similar. The most abundant component was toluene (45.4%, 62.1%), followed by benzene (16.0%, 5.6%), styrene (20.2%, 4.2%), and xylene (9.2%, 6.5%) (Fig. S1). This is consistent with a previous study, which found that toluene and xylenes were the predominant emitted AHs from six TDWTPs (Ning et al., 2015). For AIHs, n-nonane was the major component on day 1 and 20, representing 82.0% and 34.0%, respectively. However, the percentages of n-nonane and other VOCs on day 90 were remarkably different as compared with day 1 and 20, as evidenced by the even distribution of each portion (Fig. S2). A similar trend was seen for the other three VOC groups (Figs. S1, S3 and S4). This may be because the pollutants collected on day 1 and 20 (in summer) significantly differed from day 90 (in winter). That is, the higher temperature on day 1 and 20 contributed to the percent differences for these VOCs. Despite the fact that negligible changes were observed on day 1 and 20 after ST-biofilter unit treatment, probably because the biofilm was not yet completely finished at this period (An et al., 2010), a dramatic change was seen on day 90, as acetaldehyde decreased from 48.1% to 6.0%, while methylamine increased from 3.2% to 15.0% after treatment (Fig. S4).

3.2. Performance of pilot-scale ST-biofilter in treating TDWTP emissions

The PEN3 e-nose was able to differentiate and quantify volatiles emitted during Municipal Solid Waste-Legume Trimming (Delgado-Rodríguez et al., 2012) or released from different bacteria or patients, using discrimination power values from pair-wise PCA (Altomare et al., 2016). Therefore, the performance of the pilot scale

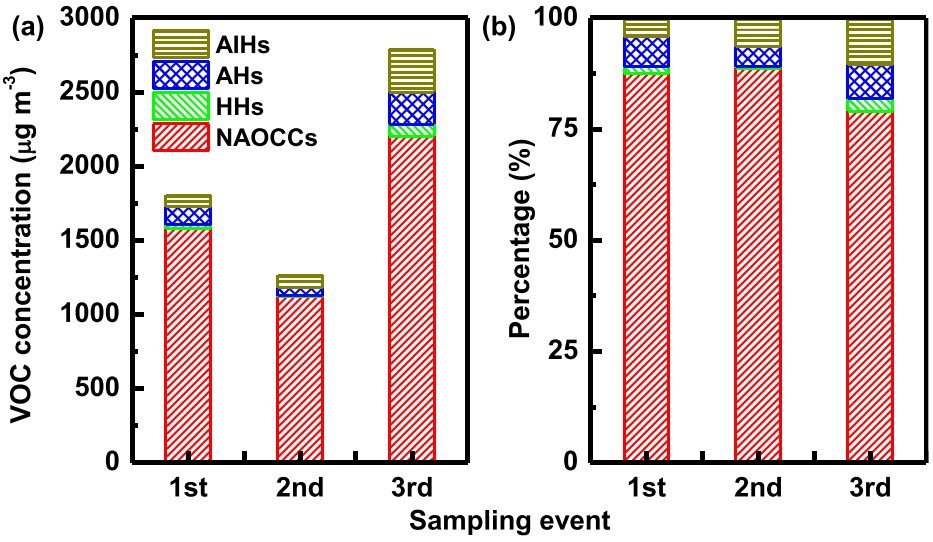

Fig. 2. Concentrations (a) and percentages (b) of VOCs emitted during textile-dyeing wastewater treating processes.

ST-biofilter was also evaluated on-line using e-nose and PCA analysis to compare odorant VOCs variation during treatment process. Fig. 3 illustrates that the odorous VOCs emitted from TDWTP (inlet) and ST-biofilter (outlet) were fairly well distinguished from the control environment atmosphere, suggesting there was a significant variation between the VOCs in the TDWTP and the environment. These results again confirmed that the TDWTP air was polluted with a high concentration of unpleasant VOCs that needed to be purified. In addition, a higher signal of sensors W1C, W5S, and W3C, which were used to discriminate aromatic benzene, oxides of nitrogen and amines, respectively, were observed in the inlet samples compared to other sampling sites, indicating that aromatic pollutants and oxides of nitrogen and ammonia contributed most to the odorous VOCs discharged from TDWTP. The inlet samples were clearly distinguished from the samples collected after ST-biofilter treatment (the outlets of biofilter), which suggested that the ST-biofilter has the potential to effectively remove these odorous VOCs emitted in the TDWTP.

Fig. 4 shows the detailed performance of the integrated ST-biofilter to treat the four groups of VOCs in the TDWTP. In general, the applied technology showed an excellent ability to remove the four groups of VOCs assessed, which is consistent with results obtained using e-nose. To be specific, after consecutive treatment periods of approximately 90 days, the average amount of residual NAOCCs, AIHs, AHs, and HHs significantly decreased from 1630 to 547, 149 to 48, 131 to 116, and 43.9 to 21.0 μg m^{-3}, with average REs of 66.7%, 67.9%, 11.7% and 52.1%, respectively (Fig. S5a). After treatment, even though the average percentage of AHs increased from 6.7% to 15.8%; significant decrease of NAOCCs from 83.4% to 74.7% was observed. This indicates that the combined technique showed a preferential ability to remove NAOCCs compared to AHs (Fig. S5b), which may result from the higher polar properties of NAOCCs compared to AHs (e.g., with the dielectric constants and Henry's law constant of methyl isobutyl ketone > styrene > toluene) (Table S4) (Chen et al., 2017). Similarly, ethyl acetate (one kind of NAOCCs) was more easily to be removed than AHs in the biofiltration system (He et al., 2012). In addition, further observations revealed that the RE of odorous VOCs in the TDWTP varied significantly at three sampling events, which might be due to the fluctuating inlet concentrations and different pollutant solubility properties (Liu et al., 2017). NAOCCs can be more effectively removed than the other three groups, reaching 83.2% RE at the third sampling event (Fig. S6).

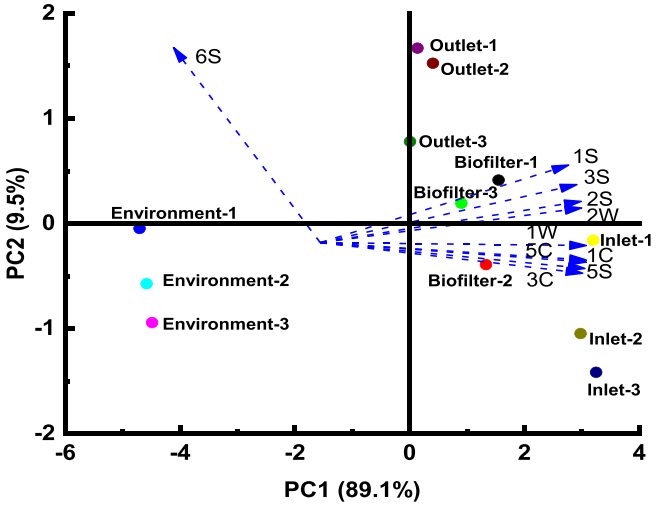

Fig. 3. Discrimination of VOCs in ST-biofilter reactor before and after treatment by principal component analysis (PCA) based on mean changes of VOC mixture composition in different sampling sites. PC1 and PC2 accounted for 89.1% and 9.5% of the variability, respectively. Volatiles of environmental samples were clustered in the lower left corner of the plot, while ST-biofilter treated samples were clustered in the upper right portions of the 2-dimensional PCA plot.

However, as noted previously (Liu et al., 2017) and Fig. 5 illustrated, the ST unit on its own can only decompose average 30.1% VOCs emitted from the TDWTP and most are relatively hydrophilic VOCs with high Henry law's constant due to the higher the constant, the easier be absorbed by water (Sander. 2015). Moreover, an increase of AH concentration was also observed after ST treatment, with RE of −67.4%. This may be attributed to easily release of AHs from ST reactor when emissions from the TDWYP (Chen et al., 2017) are treated owing to the low water solubility of the AHs. An increase of AHs and HHs concentration also occurred after the ST treatment of VOCs released from the e-waste dismantling process, due to different inlet concentrations and solubility properties (Liu et al., 2017). Comparatively, as shows in Fig. 6, much higher RE (46.4% in average) was achieved for TVOCS by the biofilter, and peaked at 56.5% for the 2nd sample event. Overall, an increase in RE of TVOC from 39.5% to 73.8% as treatment time increased from day 1−90 was obtained after the ST-biofilter treatment.

3.3. The response of bacterial community to VOCs discharged from TDWTP

Previous studies have found that bacterial communities would change in response to environmental variables (carbon source, pH, and temperature) (Russell et al., 2014) and waste pollutants (Xiong et al., 2017). Such shifts may often correspond to the unfolding of competitive dynamics when certain bacteria are under these pressures, particularly when exposed to different toxic compounds. Thus, to explore the influence of the complex contaminants on the community distribution at different stages of the pilot-scale biofilter, this study conducted 16S rRNA sequencing. Table S5 shows that the OTUs on the packing material of the biofilter collected on day 1 were higher compared to days 20 and 90, suggesting that the microbial community in biofilter developed differently when exposed to emitted VOCs during the experiment. Alpha diversity as an ecological method was also used to measure how many taxonomic groups presented within each sample and whether these groups abundance is evenly distributed (Lou, 2010). The results showed that the microbial diversity was reduced as the treatment proceeded from day 1−90, as evidenced by the decrease of ACE (from 26.5 to 21.0), Chao1 (from 26.0 to 21.0), and Shannon (from 1.5 to 0.8) index values. This implies that the unspecific inoculum was not able to deal with lignin in the woodchips or the VOC as carbon source and results in less biological diversity as the biofilter operation time continued.

By analyzing the microbial community composition and abundance at the phylum level following changes in the typical VOCs from the TDWTP, we found that Proteobacteria represented the most dominant phylum, comprising 65.5%, 65.4% and 80.5% of the microbial community on day 1, 20 and 90 samples, respectively. This indicated it was the main phyla responsible for eliminating odorous VOCs (Fig. 7). Moreover, increased REs of NAOCCs from 38.1% to 73.91% and to 83.2% as degradation proceeded from day 1−90 were also observed (Fig. S6), indicating that the Proteobacteria shift may be correlated with NAOCCs.

Fig. 8 shows that the elimination of NAOCCs (sulfur and nitrogen) corresponded to the increasing abundance of Firmicutes (from 4.4% on day 1−7.6% on day 90). The increased abundance of Actinobacteria (from 1.7% to 12.6% on day 1−20) may have been due to changes in the inlet VOCs, such as AHs (from 103 to 361 μg m^{-3}) and HHs (from 25.4 to 79.0 μg m^{-3}), as evidenced by their significant correlation with styrene, toluene, ethylbenzene, xylene,

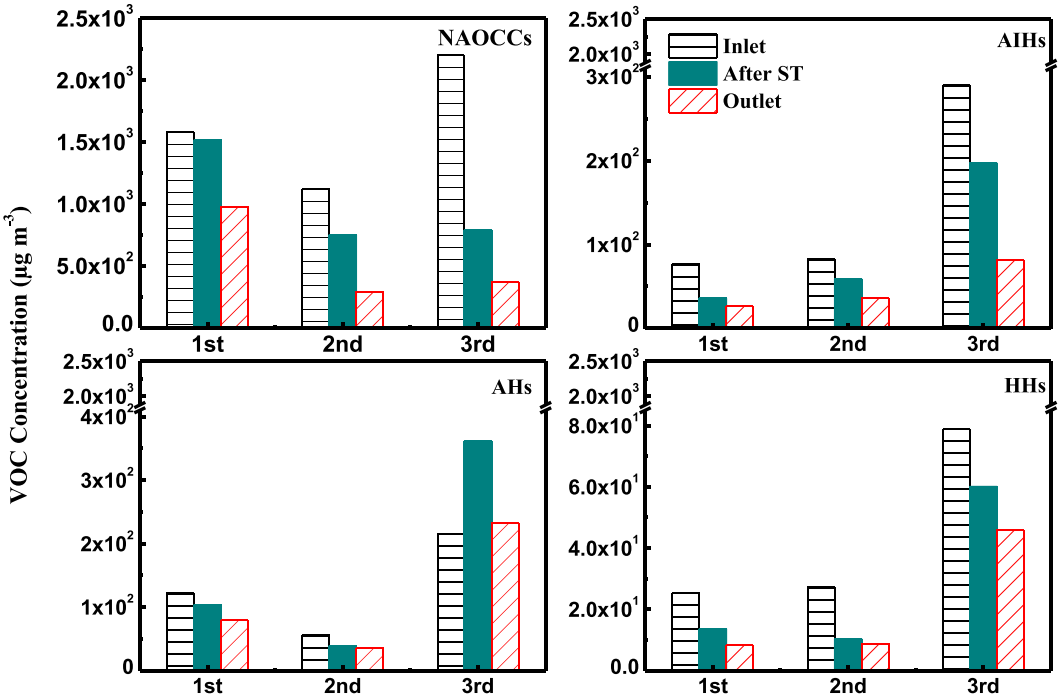

Fig. 4. Concentrations of NAOCCs, AlHs, AHs and HHs at different sampling events before and after treatment by the integrated technique.

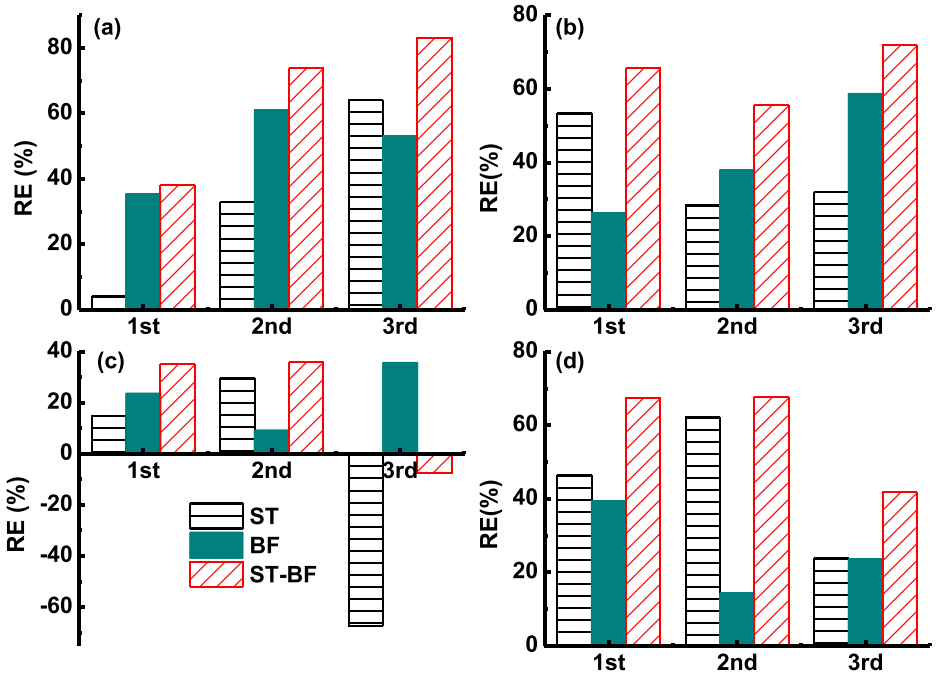

Fig. 5. RE of NAOCCs (a), AlHs (b), AHs (c) and HHs (d) by the integrated technique.

naphthalene, PAHs, and chloroalkane metabolism. This result aligns with a previous study, which showed that Actinobacteria has been used to remove ethylbenzene and chlorobenzene (Lu et al., 2018). However, the decreased relative abundance of Bacteroidetes from day 1 (7.5%) to day 90 (1.1%) suggested that phenolic compounds might inhibit bacterial growth. Reddivari et al. reported that the abundance of *Bacteroidetes* spp. was reduced after bisphenol-A exposure (Reddivari et al., 2015). In summary, the high consistency between functions of the predominant bacterial strains and the VOCs discharged from TDWTP indicated that the ST-biofilter provides effective growing conditions for these microorganisms, which attributed to good biofilter performance.

As described above, the composition of the microbial communities differed at the phyla level. Considering the microorganism diversity was closely related to contaminant content and this selective pressure may result in some genus becoming dominant among surviving strains (Cheng et al., 2016b), they also varied significantly at genus levels. For example, in this study, the most

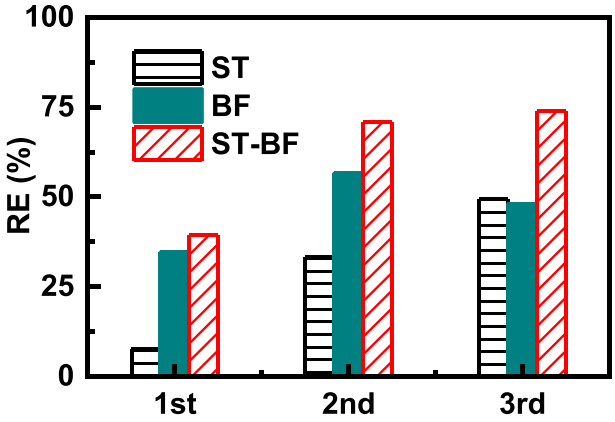

Fig. 6. RE of TVOCs by the integrated technique at different sample events.

abundant genus, *acidithiobacillus* from phyla Proteobacteria, increased from 25.0% (day 1) to 30.7% (day 20) and to 53.1% (day 90) (Fig. S7), as the RE of NAOCCs increased. Besides, the Proteobacteria contains many genera with xenobiotics degradation ability, such as *Metallibacterium* (7.2% in average), which plays an important role in simultaneously removing CS_2 and H_2S from waste gases (Xia et al., 2019); *Acidovorax* (2.2% in average), which degrades toluene, chlorobenzenes, and polycyclic aromatic hydrocarbons (PAHs) (Singleton et al., 2009; Yoshikawa et al., 2016); *Xanthomonadaceae* (1.9% in average), which contributes to the mineralization of alkane and aromatic hydrocarbons (Gutierrez, 2017; Song et al., 2016); and *Hyphomicrobium,* which uses trimethylamine as the carbon and energy source (Aguirre et al., 2016). The correlation analysis indicated that genus *Thiobacillus*, *Pseudarthrobacter*, *Novosphingobium*, *Rhizobiales*, *Hydrogenophilaceae*, *Methylobacteriaceae,* and *Planctomycetaceae* were positively correlated with the metabolism of three other groups (AHs, AIHs and HHs) (Fig. S8). Therefore, we concluded that the metabolism of the VOCs emitted from TDWTP was associated with biofilter performance and was related to the changes of bacterial abundance from day 1–90.

3.4. The attenuation of cancer and non-cancer risks of VOCs emitted from TDWTP

This study applied previously used methods (An et al., 2014) to assess the capabilities of the combined technique to reduce the non-cancer and cancer risks caused by exposure through inhalation to odorous VOCs emitted from TDWTP. Herein, six and ten kinds of typical VOCs were chosen to evaluate the cancer and non-cancer risk, respectively, based on how their measured concentrations compared with category thresholds computed from established unit risk estimates (carcinogens) and/or reference concentrations (noncarcinogens) in the USEPA Integrated Risk Information System (Sexton et al., 2007). Fig. 9 shows that before the treatment, the hazard ratios (HRs) of acetonitrile (2.49) on day 90, naphthalene, trichloroethylene, methyl isobutyl ketone and acetaldehyde on

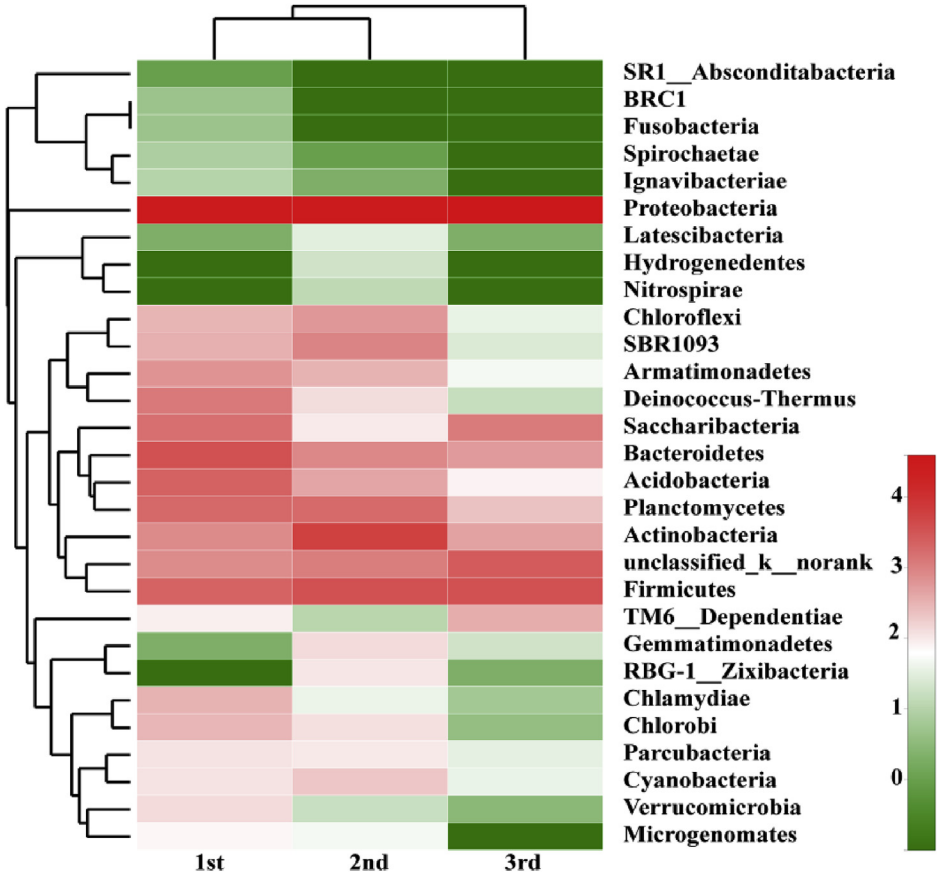

Fig. 7. Heatmaps of the relative abundance at the phylum level based on the classification of bacterial 16S rRNA gene of the specimens with the highest number of sequences. The color transition from dark green to dark red represents relative abundances from low to high. The left neighbor-joining tree was clustered in accordance with the similarity of bacterial abundance and different genera. The upper tree was clustered in accordance with the similarity of OTU composition. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

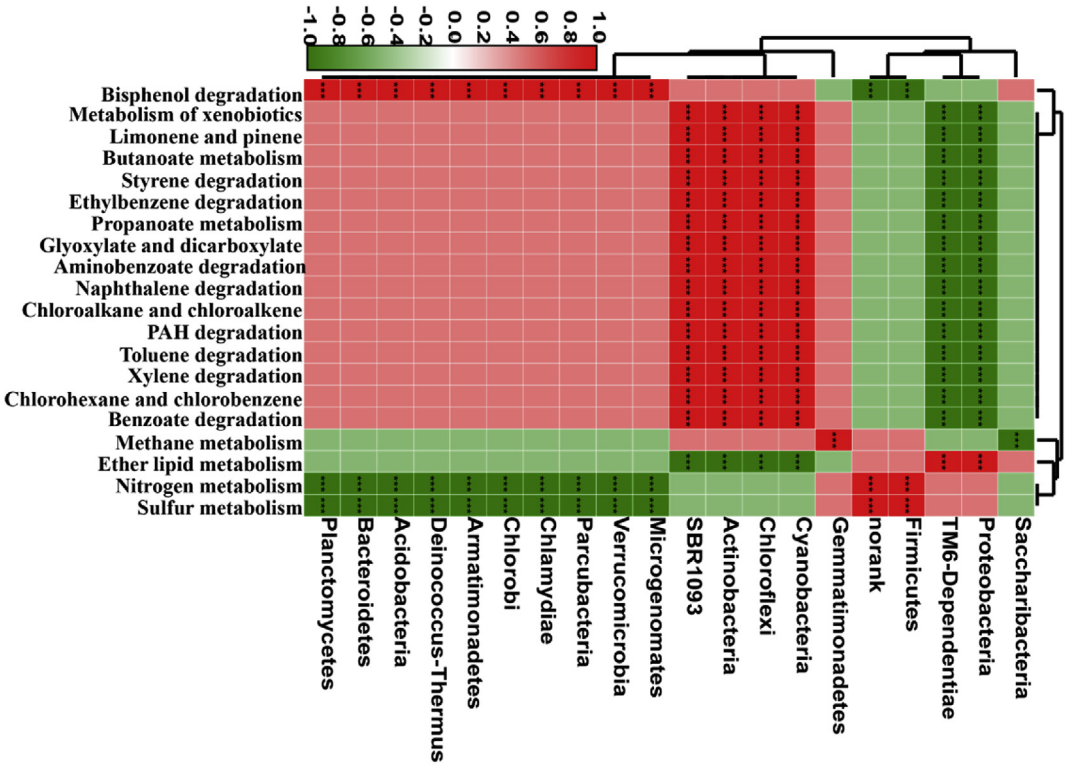

Fig. 8. Spearman correlations between microbiota at phyla level enriched in biofilter and PICRUSt-generated function of xenobiotics metabolism. The plot used asterisks as indicators for statistical significance (*). No symbol was used when the $p > 0.05$ (not significant). Two and one asterisk were used when $0.001 < P^{**} \leq 0.01$ (strong correlation), $0.01 < P^{*} \leq 0.05$ (relative strong correlation), respectively. The color transition from dark green to dark red represents relative abundances of the community from low to high. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

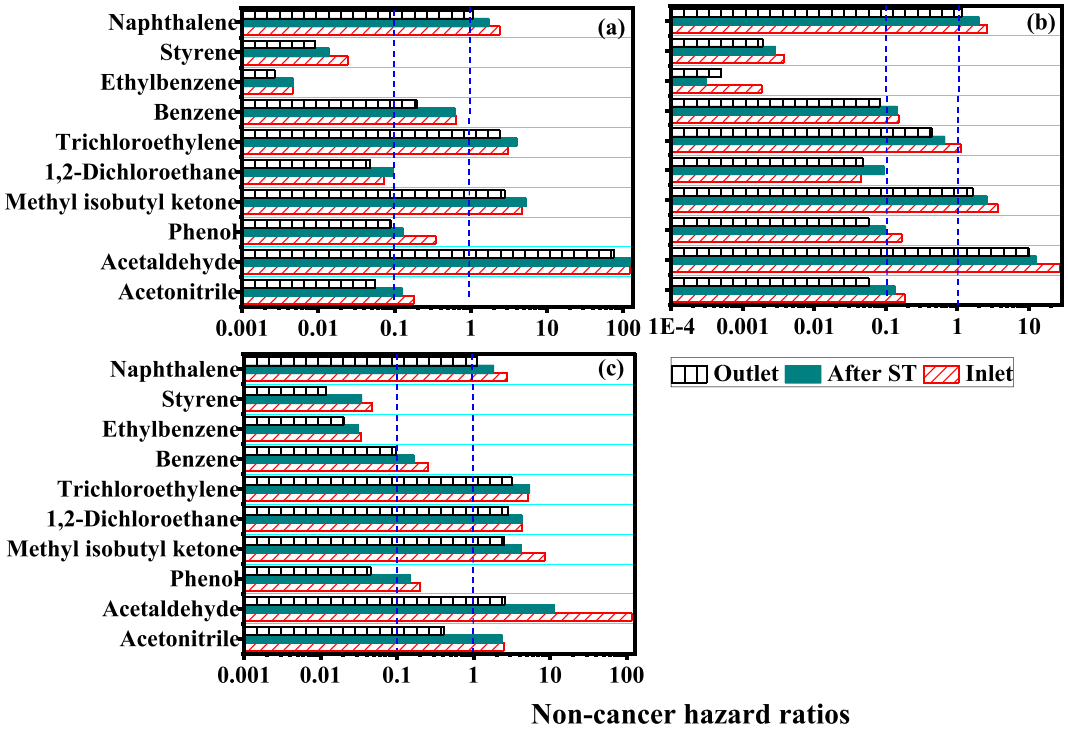

Fig. 9. Non-cancer risks for VOCs before and after treatment on day 1 (a), 20 (b) and 90 (c). Compounds with hazard ratios more than 1, between 0.1 and 1, and less than 0.1 were labeled as definite, probable or possible and negligible health risks, respectively.

days 1, 20 and 90 were all higher than 1. The highest non-cancer risk in TDWTP was seen for acetaldehyde, with a value up to 123.62 on day 1 and accounted for 70.4% of the total NAOCCs. This indicates that these VOCs discharged from TDWTP and their health risk was well above the concern levels (Ramírez et al., 2012). Other VOCs had HRs ranging from 0.1 to 1, including acetonitrile (0.18 and 0.18 on days 1 and 20, respectively), benzene (0.65, 0.15 and 0.26 on days 1, 20 and 90, respectively), and phenol (0.35, 0.17 and 0.20 on days 1, 20 and 90, respectively), suggesting a potential non-cancer risks to the workers (McCarthy Michael et al., 2009).

Fig. S9 shows the individual lifetime cancer risk (LCR) for six quantified VOCs, and the substance for which putative cancer risk was higher than 10^{-4}, between 10^{-4} and 10^{-5}, and lower than 10^{-5} was regarded as "definite risks", "probable risks", "possible risks", respectively (Sexton et al., 2007). The LCRs of acetaldehyde ($\geq 5.30 \times 10^{-4}$), styrene ($\geq 1.48 \times 10^{-4}$) and naphthalene ($\geq 2.55 \times 10^{-4}$) in the TDWTP for three sampling events all exceeded 10^{-4}, indicating a definite cancer risk at the current concentration. In addition, the LCRs of trichloroethylene and ethylbenzene on day 1 (2.49×10^{-5}, 1.16×10^{-5}) and 90 (4.24×10^{-5}, 8.47×10^{-5}), ranging from 10^{-4} to 10^{-5}, were regarded as probable cancer risks. Together, the results above showed that cancer and non-cancer risks attributable to VOC exposure in TDWTP are non-negligible and need to be paid significant attention.

Therefore, this study evaluated the capability of the ST-biofilter to diminish health risk. After ST unit treatment, the HRs and LCRs of some VOCs still exceeded 1 (naphthalene (1.71, 1.98 and 1.81 on day 1, 20 and 90), trichloroethylene (3.97 and 5.35 on day 1 and 90), methyl isobutyl ketone (5.24, 2.56 and 4.11 on day 1, 20 and 90) and acetaldehyde (122.15, 12.55 and 11.08 on day 1, 20 and 90)) (Fig. 9), and other VOCs were above 10^{-4} (acetaldehyde (2.42×10^{-3}, 2.49×10^{-4} and 2.19×10^{-4} on day 1, 20 and 90), benzene (1.43×10^{-4} on day 1), styrene (1.37×10^{-2}, 2.82×10^{-3} and 3.42×10^{-2} on day 1, 20 and 90), naphthalene (1.96×10^{-4}, 2.02×10^{-4} and 1.85×10^{-4} on day 1, 20 and 90)) (Fig. S9). It has to be mentioned that, after ST treatment, the HRs of some compounds increased, such as trichloroethylene and 1,2-dichloroethane due to their low Henry's law constant (Table S4). This indicated that the ST has a low ability to reduce risks of VOCs. Similarly, Liu et al. also found that the ST contributed little to the elimination of hydrophobic VOCs, and the maximum REs of HHs and AHs were only 31.2% and 17.3%, respectively (Liu et al., 2017).

In contrast, there was a very significant reduction in the non-cancer risks after biofilter treatment, especial for acetaldehyde (dropped from 123.62, 26.76 and 117.85 to 76.13, 10.01 and 2.56 on day 1, 20 and 90, respectively). In addition, a slight decrease of the non-cancer risks associated with naphthalene (from 2.60 to 1.11 in average), trichloroethylene (from 3.11 to 2.00 in average), and methyl isobutyl ketone (from 5.62 to 2.33 in average) were also found (Fig. 9). All these indicated that the biofilter has a dramatic enhancement in weakening the non-cancer risks of the released VOCs.

In summary, after ST-biofilter treatment, two trends were found concerning the cancer risk associated with VOCs. One trend was that the definite LCRs of acetaldehyde (5.86×10^{-4} in average), styrene (7.67×10^{-3} in average), and naphthalene (1.13×10^{-4} in average) after biofilter treatment was not decreased obviously, due to the notably enriched concentration (Liu et al., 2017). The second trend was that, consistent with the degradation results above, LCR attenuation was distinguished for acetaldehyde (from 2.33×10^{-3} to 5.07×10^{-5} on day 90), benzene (from 1.52×10^{-4} to 4.61×10^{-5} on day 1), and ethylbenzene (from 1.16×10^{-5} to 6.69×10^{-6} on day 1), which indicates that the SF-biofilter could also be used to efficiently reduce cancer risks at sites contaminated with odorous VOCs (Fig. S9). Combing the results above, we concluded that the ST-biofilter can be successfully used for end-of-pipe treatment of VOCs in TDWTP prior to discharge to the environment, and weakening the corresponding respiratory health risks. Nevertheless, to ensure that emitted VOCs meet the stringent standards (DB33/962–2015) imposed by regulatory bodies, it is important to couple source emission reductions with end-of-pipe treatment for controlling TDWTP emissions.

4. Conclusion

A pilot-scale integrated ST-biofilter was designed and applied for end-of-pipe treatment of odorous VOCs released from a TDWTP and attenuating the corresponding health risks. NAOCCs were identified as the predominant contaminants, followed by AHs, AlHs, and HHs. ST significantly reduced large sized particles and water-soluble VOCs, while the biofilter, which was pre-introduced VOCs on the packing material until saturated adsorption, effectively degraded other VOCs. When VOCs flowed through the ST-biofilter system, average REs of 66.6%, 67.9%, 11.7%, and 52.1% were achieved for NAOCCs, AlHs, AHs, and HHs, respectively, during the 90-day treatment period. Proteobacteria associated with ether lipid metabolism had the highest relative abundance in the biofilter (70.5% on average), followed by Actinobacteria, Firmicutes, and Bacteroidetes. The abundance of genus *acidithiobacillus* from phyla Proteobacteria increased when the RE of NAOCCs increased, as the ST-biofilter progressed, which indicated high consistency between microbial diversity and effective VOCs removal performance of the biofilter. Additionally, the ST-biofilter integrated technique significantly reduces cancer and non-cancer risks of the emitted VOCs, especially acetaldehyde and benzene. This result contributes to the ecological risk assessment and bioremediation of contaminated environments and reveals the response and adaptation mechanisms of microbiota to the complex VOC emissions from a TDWTP.

Author statement

Dr. Zhishu Liang: Conducted majority experiments, data analysis and paper drafting.
Mr. Jijun Wang: Conducted a portion of experiments.
Miss Yuna Zhang: Methodology, Data curation.
Mr. Cheng Han: Investigation and sampling.
Dr. Shengtao Ma: Validation.
Prof. Jiangyao Chen: Writing- Reviewing and Editing.
Prof. Guiying Li: Supervision, Writing- Reviewing and Editing.
Prof. Taicheng An: Conceptualization, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2020.120019.

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