

Analysis of Volatile Organic Compounds in the Ambient Air of a Paper Mill- A Case Study

Xin Tong, Zhenbin Zhang, Xiaoquan Chen, and Wenhao Shen*

In this work, volatile organic compounds (VOCs) in the ambient air of a secondary fiber paper mill were analyzed. For the sake of studying pollution comprehensively, four sites in the paper mill were analyzed and active sampling methods were used. Desorption was carried out with two solvents, carbon disulfide and dichloromethane. The compositions of VOCs were determined by gas chromatography-mass spectrometry (GC-MS) method. The main identified substances in the four sites were as follows: (1) waste paper sorting room: alkanes, phenols, and esters; (2) papermaking workshop: benzene series, alkanes, ethers, and phenols; (3) vacuum pump outlet: benzene series and phenols; and (4) office area: benzene series and phenols. Two main toxic substances in VOCs, the benzene series and phenols, were detected in the ambient air of the paper mill. The benzene series existed in three places along the main process of the paper mill and even existed in the office area, which was far away from the production line. Additionally, phenols were detected in all sampling locations in the paper mill.

Keywords: VOCs; Paper mill; Ambient air; Desorption; Benzene series; Phenols

*Contact information: State Key Laboratory of Pulp and Paper Engineering, South China University of Technology, Guangzhou, 510640, P.R. China; *Corresponding author: ppwhshen@scut.edu.cn*

INTRODUCTION

Due to the rapid growth of the Chinese population and urban development, the papermaking industry has been confronted with increasing demands. According to a survey report, in 2014 there were about 3000 paper mills in China, and their capacities and consumptions were 104.7 and 100.7 million tons, respectively. Additionally, the annual per capita consumption was 74 kg (1.368 billion people) (China Technical Association of Paper Industry 2015). China surpassed the United States and became the biggest paper and paperboard producer in the world in 2009, and its production accounts for 25% of the global total.

The pulp and papermaking industry is the world's sixth largest polluting industry (after the oil, cement, leather, textile, and steel industries), and emits large amounts of gas, liquid, and solid pollutants into the natural environment (Ali and Sreekrishnan 2001; Chandra and Sankhwar 2011). According to the U.S. Environmental Protection Agency (EPA), the main polluting gases from paper mills include highly toxic sulfides (TRS) and volatile organic compounds (VOCs). One paper mill can produce 400,000 pounds of dimethyl sulfide gas and 600,000 pounds of methanol per year (US Environmental Protection Agency 2013).

Analyzing VOCs in ambient air is becoming increasingly important due to continuous global industrialization. The World Health Organization defines VOCs as the organic compounds sampled on a solid sorbent and with a lower boiling point limit of 50

to 100 °C and an upper boiling point limit of 240 to 260 °C, which include alkanes (or paraffins), alkenes (or olefins), saturated and unsaturated alkyl halides, carbonyls, alcohols, and aromatic and halogenated aromatic hydrocarbons (Ince *et al.* 2011). Some investigations have revealed that many VOCs are either known or suspected carcinogens and some have toxic effects. Even low concentrations of VOCs (benzene, toluene, xylene, methyl ethyl ketone, acetone, isopropyl alcohol) have been associated with discomfort, irritation, and disease (Barro *et al.* 2009; Shin *et al.* 2015).

Measurements of VOCs are variously affected by different detecting purposes and sampling sites. For example, Fourier transform infrared spectrometry (FT-IR) is used successfully for measuring combustion gas (Dai *et al.* 2015). Proton transfer reaction-mass spectrometry (PTR-MS) is used for real time measurements of VOCs (Sahu and Saxena 2015). And cantilever enhanced photoacoustic spectroscopy (CEPAS) is a novel and promising technique in the analysis of VOCs (Hirschmann *et al.* 2013). As it is suitable for analysis of a complex mixture of compounds, the most commonly used method to detect VOCs in the ambient air is gas chromatography-mass spectrometry (GC-MS), where the sampling and preconcentration are always done in the first two steps because of the low concentration, and the solvent desorption is followed by the extraction of water-insoluble volatile fractions in the ambient gas (Ramirez *et al.* 2010; Lorenzo *et al.* 2014).

The pollutants from paper mills not only cause environmental pollution, but also threaten human health, when VOCs are major gaseous emissions (Tao *et al.* 2006). Despite the possible environmental and health risks, monitoring VOCs in paper mills has not been widely done, and little research has been reported. The results in the literature (Bordado and Gomes 2002; Avsar and Demirer 2008; Toda *et al.* 2010) revealed that methanethiol, hydrogen sulfide, sulfur dioxide, and ozone were detected by the on-site measurement of pulp and paper plants, and some malodorous gases were measured from the emissions of kraft pulp mills. These studies have focused on analyzing sulfides in atmospheric emissions from pulp mills, ambient air near the pulp and paper mills, or the compositions of pollutants based on the types of materials added during the production process. Until now, no systematic studies on VOCs in pulp and paper mills have been reported.

In this paper, field sampling of the ambient air was done in different locations in a secondary fiber paper mill, desorbing was done with carbon disulfide and dichloromethane, analyzing was done using GC-MS, and the compositions of VOCs during pulp- and papermaking were studied.

EXPERIMENTAL

Active Sampling

The air samples were collected by active sampling with a glass tube containing activated carbon granules that were micro-porous materials (Tianyue Instrument Co., China). The tube was composed of two activated carbon layers, a sampling layer and a breakthrough layer, which contained 100 mg and 50 mg activated carbon granules, respectively. An air sampler (TWA-300Z, Tianyue Instrument Co., China) was used to pump air samples at a height of 1.5 meters and a flow rate of 500 mL·min⁻¹ for 20 minutes, which meant that a total of 10 L of air was collected each time. After sampling, the tubes containing activated carbon were removed from the sampler and the two openings were tightly covered with special polytetrafluoroethylene (PTFE) caps. Then, the tubes were stored at -20 °C before further treatment.

Solvent Desorption

In this study, the substances in the activated carbon tubes were extracted with two kinds of solvents, carbon disulfide (I) and dichloromethane (II). The detailed operations were as follows: (1) all of the substances in the tubes were dissolved in 1 mL solvent by ultrasonication for 3 min; (2) the activated carbon granules were separated from the organic solvents by natural sedimentation for 30 minutes; (3) the supernatant solutions from step two were obtained, which were named sample I and sample II.

Chromatographic Conditions

Determination of the composition of sample I and sample II was performed with a gas chromatograph coupled with a mass spectrometer (Varian-4000, Varian, Inc., USA). The DB-5MS capillary column of 60 m × 0.25 mm ID with 0.25 μm film thickness (Agilent J&W Scientific, USA) was used as an analytical column. Electron impact spectra were obtained with electron energy of 70 eV, and mass spectral data were acquired over a mass range of 33-350 amu. The detailed detection conditions for GC-MS for samples I and II are presented in Table 1.

Table 1. GC-MS Parameters of Samples I and II

Parameters	Sample I	Sample II
Gas carrier	Helium	Helium
Gas flow	0.6 mL·min ⁻¹	1 mL·min ⁻¹
Injector	Splitless, 250 °C	Splitless, 250 °C
MS source temperature	200 °C	230 °C
Oven temperature	30 °C held 1 min; 10 °C·min ⁻¹ to 60 °C; 30 °C·min ⁻¹ to 150 °C, held 15 min	40 °C for 2 min; 10 °C·min ⁻¹ to 80 °C, held for 2 min; 25 °C·min ⁻¹ to 260 °C, held 5 min

Case Study

The ambient air samples were collected from Guangzhou Paper Mill, which is located in the southernmost part of Guangzhou (almost 50 kilometers from downtown).

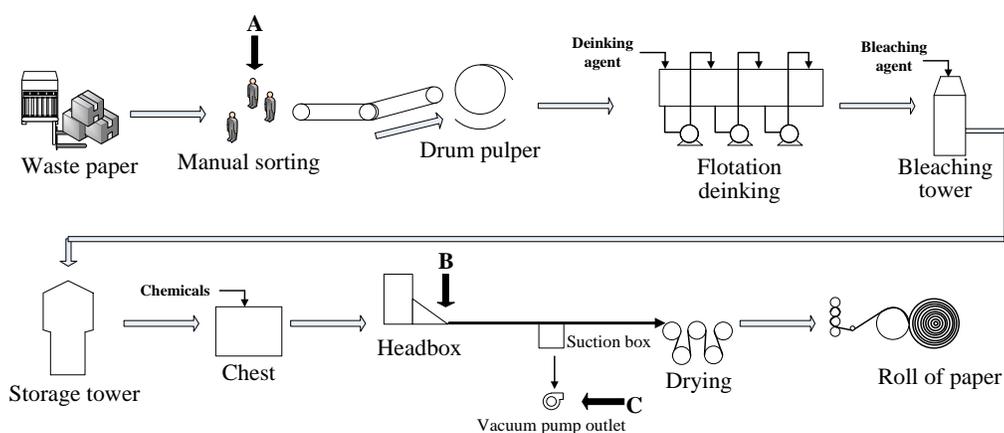


Fig. 1. The flow chart of Guangzhou Paper Mill and the sampling locations: (A) waste paper sorting room, (B) papermaking workshop, (C) vacuum pump outlet

The mill's main product is newsprint paper. Using secondary fiber as a raw material, its annual production capacity of paper is 557,800 tons, which accounts for about 17% of the national total (China Technical Association of Paper Industry 2015). A flow chart of Guangzhou Paper Mill is illustrated in Fig. 1.

The newsprint paper production process with secondary fiber starts in the waste paper sorting room where impurities are manually sorted and removed by a drum pulper. The next operations are ink and adhesive removals, which are performed in the flotation deinking process by a deink agent. The whiteness of deinked pulp (DIP) is improved by a bleaching agent in the bleaching tower. Before feeding into the papermaking process, the bleached DIP in the storage tower mixes with some chemicals in the chest to maintain the desired quality index of the final paper products. Then, well-dispersed pulp is jetted onto the forming fabric of the paper machine from the headbox. After dewatering with natural gravitational action on the Fourdrinier wire, further dewatering is achieved through a suction box at the end of the wire. During the dewatering action, all of the gaseous substances in the papermaking workshop will be discharged into the atmosphere through the vacuum pump simultaneously. Finally, the production of newsprint paper is completed after drying, calendaring, and rewinding processes.

In the study, four air samples in the paper mill were collected and analyzed from the following locations: (A) waste paper sorting room, (B) papermaking workshop, (C) vacuum pump outlet, and (D) office area. The overview of the plant and the sampling sites are indicated in Fig. 2. The office area is about 1 kilometer away from the production area, which is the reason why site (D) is absent in Fig. 1.



Fig. 2. Overview of the plant and the sampling locations: (A) waste paper sorting room, (B) papermaking workshop, (C) vacuum pump outlet, (D) office area

RESULTS AND DISCUSSION

With high surface area and pore volumes, activated carbons have been efficiently used in many gas pollution detection processes (Duan *et al.* 2006; Ramirez *et al.* 2010), and the detection of VOCs can be achieved with further desorption treatment. Carbon disulfide is a favorable solvent for desorption of nonpolar compounds due to its

physicochemical properties, such as low response value on the detector, and the fact that it can be eluted rapidly on GC columns (Harper 2000; Woolfenden 2010). Dichloromethane is another extraction solvent with different chemical polarity, which favors desorption of polar compounds (Lerner *et al.* 2012).

As described in the section “Solvent Desorption,” samples I and II with two desorption solvents were obtained, and their GC-MS analytical results are shown in Figs. 3 to 6 and Tables 2 to 5. In Figs. 3 to 6, when multiple peaks occurred in a single ion chromatogram, the components of interest were identified by the mass spectra with their molecular fragmentation, and the most representative compounds of each sample were marked.

Waste Paper Sorting Room

In this area, with the extraction of carbon disulfide, the analyzed results of sample I in Table 2 revealed that all the detected substances were alkane compounds with high boiling points. As illustrated in Fig. 3(A), the amount of those substances was relatively high compared with other sites (Fig. 4(A), 5(A), and 6(A)). It was surmised that during the collection and transportation process of waste paper, some compounds with high boiling points might attach to dust and particulate matter on the surface of the waste paper, thus they were absorbed on the activated carbon during the sampling process.

Table 2. GC-MS Analytical Results of Air Sample from Waste Paper Sorting Room

Carbon disulfide (Sample I)				Dichloromethane (Sample II)			
No.	Organic pollutant	Molecular formula	Matching degree (%)	No.	Organic pollutant	Molecular formula	Matching degree (%)
1	Nonadecane	C ₁₉ H ₄₀	95	1	2-Hydroxy-1-phenylethanone	C ₈ H ₈ O ₂	95
2	n-Heneicosane	C ₂₁ H ₄₄	75	2	cis-9-Heneicosene	C ₂₁ H ₄₂	77
3	Tetracosane	C ₂₄ H ₅₀	90	3	Methyl hexadecanoate	C ₁₇ H ₃₄ O ₂	62
4	Octacosane	C ₂₈ H ₅₈	85	4	13-Methyl Pentadecanoic acid*methyl ester	C ₁₇ H ₃₄ O ₂	60
				5	2,2'-Methylenebis(6-tert-butyl-4-methylphenol)	C ₂₃ H ₃₂ O ₂	71

Figure 3(B) is the chromatograph of sample II extracted by the dichloromethane desorption procedure. With the extraction of the weak polar solvent, the information in Table 2 shows that the following substances were detected in the waste paper sorting room and their relative quantities were: phenols (57%), esters (38%), alkanes (3%), and ketones (2%).

The analyzed results of the two samples revealed that there were mainly alkane compounds, phenols, and esters with high boiling points in the waste paper sorting room of the paper mill.

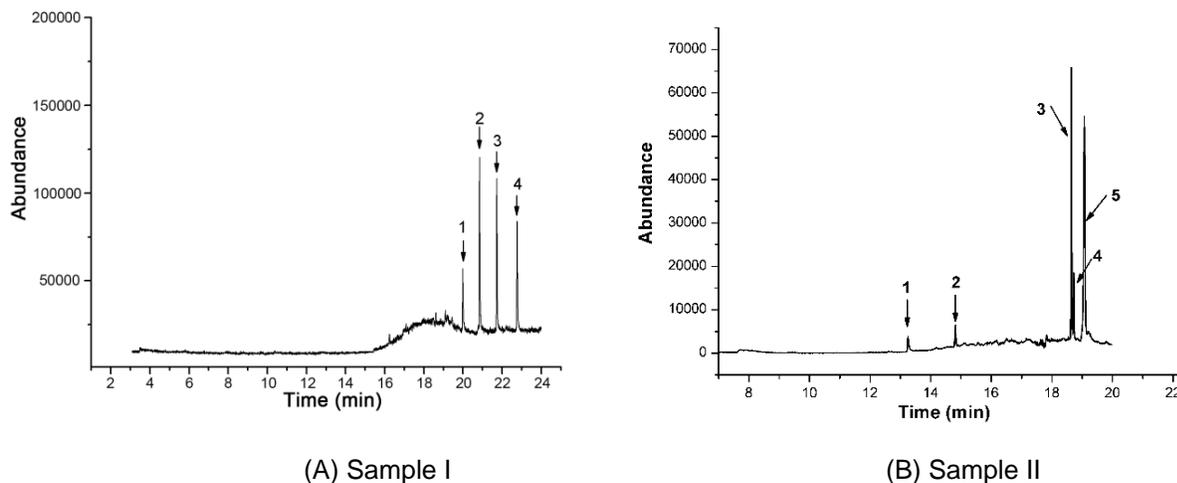


Fig. 3. Total ion chromatograms of air samples from waste paper sorting room

Papermaking Workshop

The total ion chromatograms and the mass spectra results of air samples from the papermaking workshop with two extraction solvents are shown in Fig. 4 and Table 3. They show that sample I with carbon disulfide solvent contained benzene, methylbenzene, p-xylene, ethylbenzene, 2-ethyltoluene, decane, and dodecane, *etc.*, while sample II with dichloromethane solvent included ketones (5%), ethers (12%), alkane compounds (64%), esters (6%), and phenols (13%).

The identified results demonstrate that some hazardous substances were present in the papermaking workshop, which include the benzene series, alkane compounds, phenols, ethers, *etc.* As gray marks indicate in Table 3, the degrees of these substances are so high that it is an indisputable fact that they are present in the ambient air of this area.

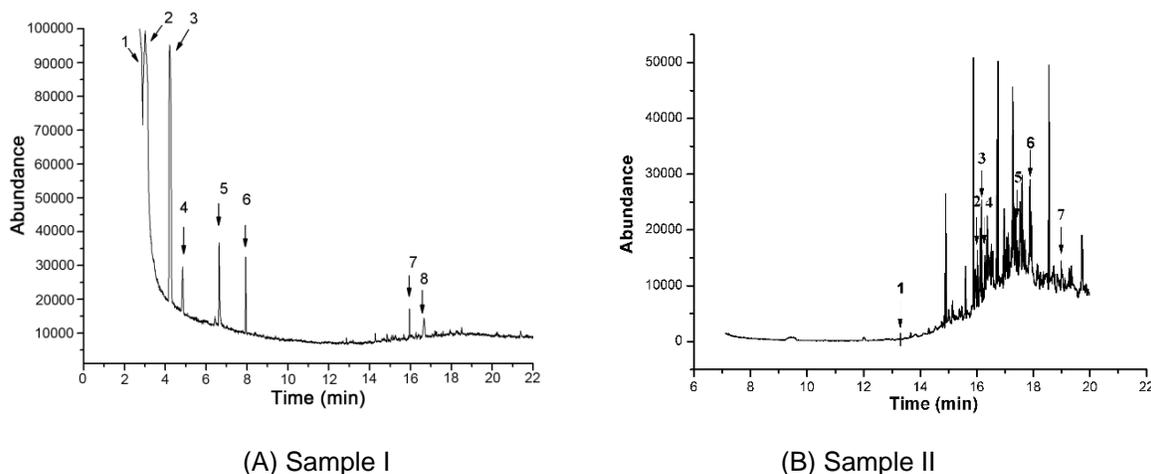


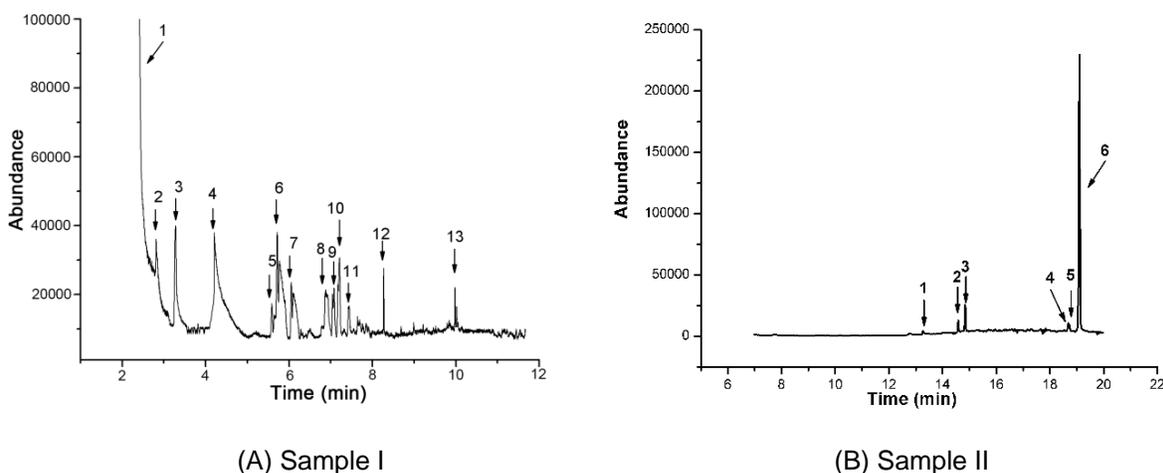
Fig. 4. Total ion chromatograms of air samples from papermaking workshop

Table 3. GC-MS Analytical Results of Air Sample from Papermaking Workshop

Carbon disulfide (Sample I)				Dichloromethane (Sample II)			
No.	Organic pollutant	Molecular formula	Matching degree (%)	No.	Organic pollutant	Molecular formula	Matching degree (%)
1	Carbon disulfide	CS ₂	97	1	2-Hydroxy-1-phenylethanone	C ₈ H ₈ O ₂	95
2	Benzene	C ₆ H ₆	94	2	Butylated hydroxyanisole	C ₁₁ H ₁₆ O ₂	71
3	Methylbenzene	C ₇ H ₈	93	3	Heptadecane	C ₁₇ H ₃₆	82
4	p-xylene	C ₈ H ₁₀	88	4	2,4,5-triisopropylphenol	C ₁₅ H ₂₄ O	65
5	Ethylbenzene	C ₈ H ₁₀	70	5	9-Acridone	C ₁₃ H ₉ NO	81
6	2-Ethyltoluene	C ₉ H ₁₂	90	6	Ditolylmethane	C ₁₅ H ₁₆	77
7	Decane	C ₁₀ H ₂₂	41	7	Methyl pentadecanoate	C ₁₆ H ₃₂ O ₂	66
8	Dodecane	C ₁₂ H ₂₆	39				

Vacuum Pump Outlet

According to the process technology, the gas in the papermaking workshop is collected by an exhaust hood and discharged into the atmosphere through a vacuum pump. As illustrated in Fig. 5(A), sample I with carbon disulfide in the air of the vacuum pump outlet contained more abundant substances than other sites (Fig. 3(A), 4(A), and 6(A)). The direct interpretations of the mass spectra obtained by GC-MS analysis are listed in Table 4. It clearly states that all the analyzed substances were benzene series. Furthermore, more benzene series (1,3-dimethyl benzene, 2,5-dimethyl-2-amino propyl benzene, 1-allyl mercaptan, heptane) were found in the vacuum pump outlet.

**Fig. 5.** Total ion chromatograms of air samples from vacuum pump outlet

Unlike the results of sample I (Table 4), sample II with dichloromethane solvent in this location had a different composition, which included phenols, esters, ketones, and olefins, and the abundance of the 6th peak in Fig. 5(B) shows that phenols (88%) were the main substances at this site. Meanwhile, it can be observed that the peak areas of the compounds detected in the outlet of vacuum pump were significantly higher than elsewhere, most likely the result of high temperatures at this site. Therefore, VOCs in the hot ambient air are more likely to volatilize and absorbed by the activated carbon.

To summarize, different results were obtained in the vacuum pump outlet. Although there were more substances detected in this area than other sites, the main composition was made up of benzene series and phenols in large quantities.

Table 4. GC-MS Analytical Results of Air Sample from Vacuum Pump Outlet

Carbon disulfide (Sample I)				Dichloromethane (Sample II)			
No.	Organic pollutant	Molecular formula	Matching degree (%)	No.	Organic pollutant	Molecular formula	Matching degree (%)
1	Carbon disulfide	CS ₂	98	1	2,2-Dimethylpropiophenone	C ₁₁ H ₁₄ O	79
2	Benzene	C ₆ H ₆	95	2	Methyl nonadecanoate	C ₂₀ H ₄₀ O ₂	55
3	Methylbenzene	C ₇ H ₈	93	3	cis-9-Heneicosene	C ₂₁ H ₄₂	79
4	p-xylene	C ₈ H ₁₀	80	4	cis-11-Tetradecenyl acetate	C ₁₆ H ₃₀ O ₂	77
5	Ethylbenzene	C ₈ H ₁₀	63	5	Methyl 2-methyltetradecanoate	C ₁₆ H ₃₂ O ₂	67
6	o-xylene	C ₈ H ₁₀	50	6	2,2'-Methylenebis(6-tert-butyl-4-methylphenol)	C ₂₃ H ₃₂ O ₂	70
7	1, 3 - Dimethyl benzene	C ₈ H ₁₀	52				
8	2-Ethyltoluene	C ₉ H ₁₂	70				
9	Cumene	C ₉ H ₁₂	83				
10	1, 2, 3 - Trimethyl benzene	C ₉ H ₁₂	40				
11	2, 5 - Dimethyl - 2 - amino propyl benzene	C ₁₁ H ₁₇ N	60				
12	Allyl mercaptan	C ₃ H ₆ S	35				
13	Heptane	C ₇ H ₁₆	56				

Office Area

According to the GC-MS results of sample I in Fig. 6(A), in contrast to the former total ion chromatograms (Fig. 3(A), 4(A), and 5(A)), only two substances were observed: benzene and methylbenzene. Fig. 6(B) shows that several compounds were detected in sample II by desorption with dichloromethane at this site and phenols (93%) were the main substance. All of the interpretations of the mass spectra are listed in Table 5.

It should be noted that the range of different kinds of VOCs in the office area was much narrower than that in the other three sites. Only two main substances were revealed: benzene series and phenols. The reason for this could be that the substances in the ambient

air of the production area gradually reduced after one kilometer's diffusion process. Although the detected substances do not amount to much, it is astonishing that there are still hazardous substances one kilometer away from the production area.

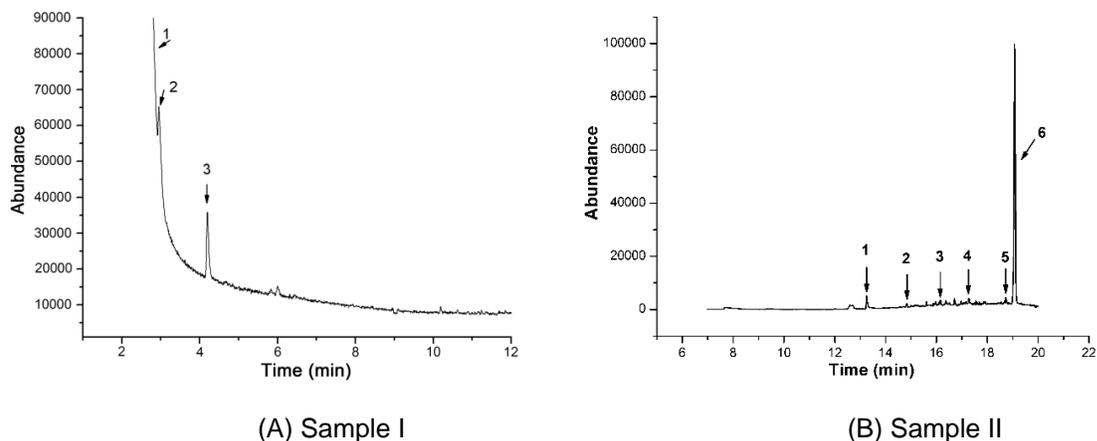


Fig. 6. Total ion chromatograms of air samples from office area

Table 5. GC-MS Analytical Results of Air Sample from Office Area

Carbon disulfide (Sample I)				Dichloromethane (Sample II)			
No.	Organic pollutant	Molecular formula	Matching degree (%)	No.	Organic pollutant	Molecular formula	Matching degree (%)
1	Carbon disulfide	CS ₂	97	1	2-Hydroxy-1-phenylethanone	C ₈ H ₈ O ₂	96
2	benzene	C ₆ H ₆	86	2	cis-9-Heneicosene	C ₂₁ H ₄₂	82
3	methylbenzene	C ₇ H ₈	90	3	2,4,6-Trimethyldecane	C ₁₃ H ₂₈	88
				4	7-Methylpentadecane	C ₁₆ H ₃₄	78
				5	Methyl hexadecanoate	C ₁₇ H ₃₄ O ₂	61
				6	2,2'-Methylenebis(6-tert-butyl-4-methylphenol)	C ₂₃ H ₃₂ O ₂	72

CONCLUSIONS

- The volatile organic compounds (VOCs) in the ambient air of a secondary fiber paper mill were analyzed in this work. Considering four sites in the paper mill, active sampling methods were used first, followed by desorption operations with two solvents, carbon disulfide and dichloromethane, respectively. The compositions of VOCs were determined by GC-MS.
- The identified compositions in these four sites were as follows:
 - waste paper sorting room: alkane compounds, phenols, and esters
 - papermaking workshop: benzene series, alkanes, ethers, and phenols
 - vacuum pump outlet: benzene series and phenols
 - office area: benzene series and phenols

3. Two main toxic substances in VOCs, benzene series and phenols, were detected as the main substances in the ambient air of the secondary fiber paper mill. The benzene series existed in three detected sites in the main process of papermaking, and even in the office area, which is far away from the production line. Additionally, phenols were detected in all sampling locations.
4. Considering these remarkable results, for the sake of pollution control, determining the source of pollution during the pulp and papermaking process is necessary and of interest for future research.

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