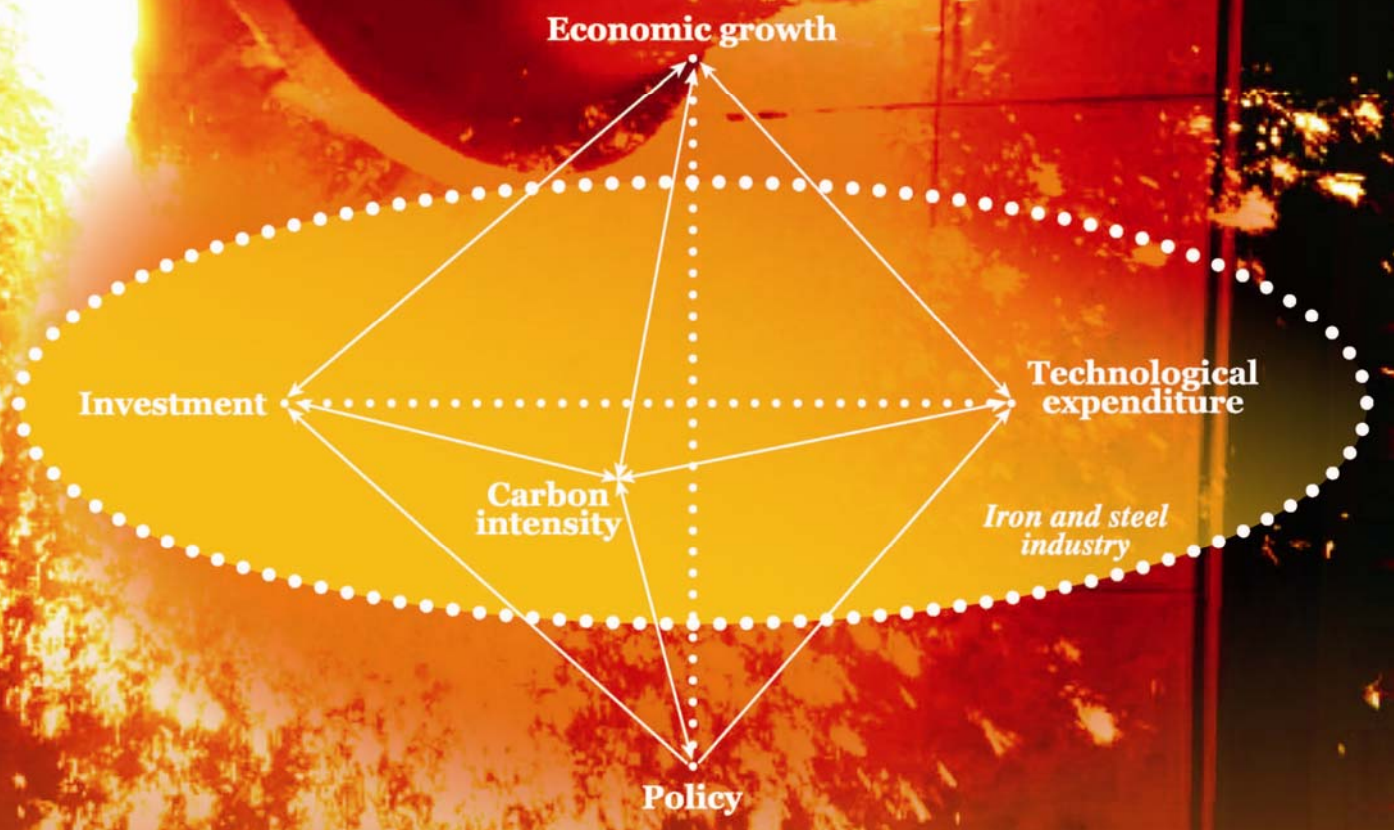
ISSN 1001-0742
CN 11-2629/X

JES

JOURNAL OF ENVIRONMENTAL SCIENCES

February 1, 2015 Volume 28
www.jesc.ac.cn



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Journal of Environmental Sciences Volume 28 2015

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Pollution levels and characteristics of phthalate esters in indoor air of offices

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ARTICLE INFO

Article history:
Received 26 March 2014
Revised 4 May 2014
Accepted 14 June 2014
Available online 18 December 2014

Keywords:
Phthalate esters
Pollution status
Particle size distribution
Indoor air
Office

ABSTRACT

The pollution status and characteristics of PAEs (phthalate esters) were investigated in indoor air of offices, and PAEs of both gas-phase and particulate-phase were detected in all the samples. The concentration (sum of the gas phase and the particulate phase) was 4748.24 ng/m^3, ranging between 3070.09 and 6700.14 ng/m^3. Diethyl phthalate, dibutyl phthalate, and di(2-ethylhexyl) phthalate were the most abundant compounds, together accounting for 70% of the ∑6PAEs. Dividing the particulate-phase PAEs into four size ranges (<2.5, 2.5–5, 5–10, >10 μm), the result indicated that PAEs in $PM_{2.5}$ were the most abundant, with the proportion of 72.64%. In addition, the PAE concentration in $PM_{2.5}$ correlated significantly with the total particulate-phase PAEs (R^2 = 0.85). Thus, the amount of PAEs in $PM_{2.5}$ can be estimated from the total amount of particulate-phase PAEs using this proportion. In a comparison between the offices and a newly decorated study room, it was found that pollution characteristics were similar between these two places. Thus, it is implied that the PAE concentration decreased by 50% 2 yr after decorating.

Introduction

Plasticizers are important promoters in material processing, and are widely used in building materials, packaging materials, electronics, medical parts and many other necessities of life (Zhang et al., 2012; Wang et al., 2010a, 2010b; Schripp et al., 2010; Afshari et al., 2004; Clausen et al., 1999; Sheldon et al., 1993; Pöhner et al., 1997). As the most widely used plasticizer, phthalate esters (PAEs) are ubiquitous in indoor environments, and have become emerging organic pollutants in indoor air (Bornehag et al., 2005; Koch et al., 2003; Wensing et al., 2005).

PAEs are considered to be endocrine-disrupting compounds that have functions similar to female sex hormones. These compounds can destroy testicles, inhibit the formation of sperm, affect reproductive functions, and damage the reproductive system of males (Hauser et al., 2006; Li et al., 2006; Swan et al., 2005; Silva et al., 2005; Foster et al., 2001). In addition, PAEs also have influences on females. They can cause precocious female puberty (Adibi et al., 2003; Colón et al., 2000) and increase the risk of breast cancer (Swan and Davis, 2003). What's worse, reports (Pei et al., 2013; Wang et al., 2010a, 2010b) indicated that PAEs may pose carcinogen risks to human health.

The respiratory system, digestive tract, and skin are the main pathways for PAEs entering the body; and concentrations of PAEs have been detected in human urine, blood, milk and many other bodily fluids (Hines et al., 2009a, 2009b; Mortensen et al., 2005). Compared with food and water, PAE exposure in the air is more common. So far, research on PAE pollution in indoor air has mainly focused on either the gas phase (Wensing et al., 2005) or particle phase (Wang et al., 2012; Lin et al., 2009; Kolarik et al.,

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http://dx.doi.org/10.1016/j.jes.2014.05.051
1001-0742/

Table 1 – Concentration of PAEs in indoor air of offices (ng/m^3). G: gas-phase; P: particulate-phase.

Site	DMP		DEP		DBP		BBP		DEHP		DnOP		∑PAEs		Total
	G	P	G	P	G	P	G	P	G	P	G	P	G	P	
1	320.08	180.04	597.07	332.07	711.65	340.18	571.97	299.62	720.66	690.75	nd	nd	2933.43	1842.67	4776.09
2	364.48	233.17	550.18	371.59	690.52	366.06	429.68	202.65	620.55	642.67	nd	nd	2655.37	1816.15	4471.52
3	566.06	311.62	858.66	559.41	1011.21	848.07	589.62	395.77	811.38	748.35	nd	nd	3836.92	2863.22	6700.14
4	510.65	245.97	708.68	417.18	606.15	414.66	263.66	189.97	564.52	504.89	nd	nd	2723.59	1846.35	4569.94
5	820.62	490.65	790.58	445.29	613.11	569.07	309.40	233.11	684.52	593.67	nd	nd	3218.22	2331.79	5550.01
6	598.12	310.66	805.98	417.65	617.56	478.43	287.65	239.45	536.99	467.78	nd	nd	2846.31	1913.97	4760.27
7	432.27	210.72	488.40	385.02	775.10	380.58	408.22	210.01	566.47	505.73	nd	nd	2670.45	1692.06	4362.51
8	663.73	374.88	746.92	389.68	675.74	452.62	379.32	204.24	611.46	507.87	nd	nd	3077.18	1929.28	5006.46
9	496.42	244.92	653.05	361.75	463.80	294.32	544.53	234.75	506.89	416.98	nd	nd	2662.70	1552.72	4215.41
10	537.73	230.75	380.06	168.28	422.96	260.31	342.13	173.92	290.78	263.16	nd	nd	1973.67	1096.43	3070.09
mean	532.22	283.33	657.96	384.79	658.78	440.42	419.61	245.72	591.22	534.18	nd	nd	2859.78	1888.46	4748.24

DEP: diethyl phthalate, DBP: dibutyl phthalate, BBP: butylbenzyl phthalate, DEHP: di(2-ethylhexyl) phthalate; DnOP: di-n-octyl phthalate; PAE: phthalate esters.

2008; Bornehag et al., 2005), but very few studies have investigated both gas-phase and particle-phase PAEs simultaneously in the indoor environment. Offices are important places for people to live and work; many people spend 8 hr a day or more in offices, which contain a lot of packaging materials and electronic parts. As a result, PAEs may be present at high levels in offices. However, there have been few studies on PAE pollution in offices, and studies on particle size distribution are even fewer. In this study, 10 offices decorated for over 2 yr were selected as sample sites, and the concentration level and pollution characteristics of PAEs (gas-phase and particulate) and size distribution of particulate PAEs in offices were investigated.

1. Materials and methods

1.1. Chemicals and materials

All chemicals and solvents used for extraction and gas chromatography (GC) analysis were of HPLC grade. Phthalate standard mixtures M-8060, including dimethyl phthalate (DMP), diethyl phthalate (DEP), dibutyl phthalate (DBP), butylbenzyl phthalate (BBP), di(2-ethylhexyl) phthalate (DEHP) and di-n-octyl phthalate(DnOP), were purchased from AccuStandard (New Haven, CT, USA) as stock solutions in isooctane. The concentration of each phthalate was 2.0 mg/mL.

1.2. Sample analysis

1.2.1. Sample collection

Indoor air samples were repeatedly collected from 10 offices in Hangzhou, from the spring to the fall in 2013. Before sampling, all the doors and windows were kept closed for 24 hr, and during our experiment, all the doors and the windows were kept closed, while normal residential activities were maintained in the offices. Sampling collections were divided into 2 parts.

In the 1st part, both gas-phase and particulate-phase PAEs were simultaneous determined, using a scientific and well-developed SVOC sampling method (Pei et al., 2013; Zhu et al., 1997): the samples were collected into a plexiglass sampling head with a glass fiber filter (37 mm in diameter, pore size of 0.45 μm, Whatman, England) and subsequently into a glass tube packed with 2 g XAD-2 adsorbent (Sigma-Aldrich, USA), using an electronically controlled air sampler (PC-A, Hengda, Zhejiang, China). The sampling device was set 1.5 m above the floor, with sampling times of 8 to 10 hr, and sampling flow of 1.0 L/min (air pump changes were in the range of less than 5% before and after sampling).

The 2nd part was particulate classification sampling. There are 4 sampling heads on the sampler, including a $PM_{2.5}$ sampling head, PM_5 sampling head, PM_{10} sampling head and TSP sampling head. When sampling $PM_{2.5}$, all the sampling heads were used successively; when sampling PM_5, the $PM_{2.5}$ sampling head was taken out; other fractions were collected similarly. The samples were collected with a glass fiber filter (90 mm in diameter, pore size of 0.45 μm, Whatman, England) using a medium volume sampler (Hengda, Zhejiang, China). The sampling device was set 1.5 m above the floor, with sampling times of 1–2 hr, and sampling flow of 60 L/min (air pump changes were in the range of less than 5% before and after sampling).

When sampling was finished, both ends of the sampling device were kept under seal, and samples were brought back to the laboratory for immediate processing. Temperature, humidity, and air pressure were synchronously recorded using an electronic temperature and humidity instrument (HTC-1, Boyang, Zhengzhou, China) and a digital air pressure monitor (BY-2003P, Taishi, Suzhou, China), respectively.

1.2.2. Sample pretreatment and analysis

Samples were extracted from XAD-2 and glass fibers filter using an ultrasonic cleaner (SK250HP, KUDOS, Shanghai, China) for 30 and 25 min, respectively, with a mixture of dichloromethane/acetone (1:1) as the extraction solvent. Then, half of the extraction liquid with 30 μL dimethyl sulfoxide added was condensed using a N-EVAP (MTN-2800W, Automatic Science, Tianjin, China). The sample was analyzed by a gas chromatograph (FULI 9790, Wenling, China) with a DB-5 capillary column (30 m × 0.25 mm ID × 0.25 μm, Agilent Technology Inc, America). The analysis was performed using the pulsed split-less mode, and the injection volume was 2 μL. High purity nitrogen was used as the carrier gas. The column condition was 60°C, held for 2 min, increased to 240°C with a rate of 15°C/min, followed by a rate of 5°C/min to 280°C, held for 10 min.

Table 2 – Size distribution of the particulate-phase PAEs (ng/m³).

Size (μm)	DMP (ng/m³)		DEP (ng/m³)		DBP (ng/m³)		BBP (ng/m³)		DEHP (ng/m³)		DnOP		∑PAEs	
	Mean	Range	Mean	Range	Mean	Range	Mean	Range	Mean	Range	Mean	Range	Mean	Range
<2.5	223.62	142.75–300.69	269.69	153.26–386.11	321.17	168.10–506.29	153.02	100.20–211.68	408.31	189.99–608.15	nd	nd	1375.81	774.64–2050.68
2.5–5	11.75	nd–23.25	17.15	12.14–23.19	34.33	23.74–47.61	20.20	nd–43.15	30.29	19.84–47.26	nd	nd	113.72	59.42–238.29
5–10	27.46	10.97–61.06	30.19	14.76–44.58	39.47	24.98–47.99	35.58	16.07–63.41	40.68	26.63–58.14	nd	nd	175.37	87.07–321.39
>10	20.49	13.47–28.93	67.77	nd–116.58	45.46	22.77–93.78	35.92	nd–108.70	53.90	7.23–94.37	nd	nd	223.56	90.18–393.46

1.2.3. Quality assurance

Quality assurance was implemented in the analysis of target compounds for the blank samples, detection limit, defined as three times the standard deviation of the lowest concentration of standard solution, and recovery rate. A field blank sample was taken on each sampling day. The detection limit values of the target compounds using GC-flame ionization detection were 26.48, 16.09, 11.72, 20.95, 14.00, and 20.70 pg (DnOP), respectively, while the recovery rate ranged from 91.75% to 115.10%.

2. Results and discussion

2.1. Concentration level

Concentration levels of PAEs in indoor air of offices are shown in Table 1. PAEs were detected in all samples; specifically, DMP, ?thyc=5?> DEP, DBP, BBP, and DEHP were all detected, and DnOP was not detected. The concentration of the detected PAEs ranged from 3070.09 to 6700.14 ng/m^3, and the mean concentration was 4748.24 ng/m^3, of which the gas concentration was 2859.78 ng/m^3, and the particulate-phase was 188.46 ng/m^3. The concentration of outdoor phthalates was reported to be approximately 97 ng/m^3, with a range of 43.8–159.6 ng/m^3 (Wang et al., 2008). The concentration of phthalates in the indoor air of offices in this study was about 49 times higher than that in the outdoors, which indicates that sources of phthalates mainly exist in the indoor environment.

The size distribution of the particulate-phase PAEs is shown in Table 2. Particulate-phase PAEs with size below 2.5 μm were the most abundant; the mean concentration was 1375.81 ng/m^3, with a range of 774.64–2050.68 ng/m^3. The mean concentration of particulate-phase PAEs with size above 10 μm, between 2.5–5, and between 5–10 μm was 223.56, 113.72, and 175.37 ng/m^3, respectively.

In addition, it was found that the mean concentration of each compound was higher than the median, which indicates that the mean concentration was greatly influenced by samples of high concentration. This finding applies to the investigation and analysis of all indoor pollutants.

2.2. Pollution characteristics

Of the 5 detected PAEs, DEHP was the most abundant compound, with a concentration of 1125.41 ng/m^3, about 1.02, 1.08, 1.38 and 1.69 times higher than DBP, DEP, DMP and BBP, respectively. DEHP, DBP, and DEP were the most abundant compounds, together accounting for nearly 70% of the ∑PAEs, seen in Fig. 1.

The gas-phase and particulate-phase concentration levels of the 5 detected PAEs in indoor air of offices were compared, as shown in Fig. 2. The results indicate that gas-phase PAEs had a higher level than particulate-phase, accounting for 60.23% of the total PAEs, 1.51 times the level of the particulate-phase, as seen in Fig. 3. DMP and DEP always exist in the gas phase in indoor air owing to their smaller molecular weight. The proportion of each PAE in the particulate phase increased as the molecular weight increased (except for BBP).

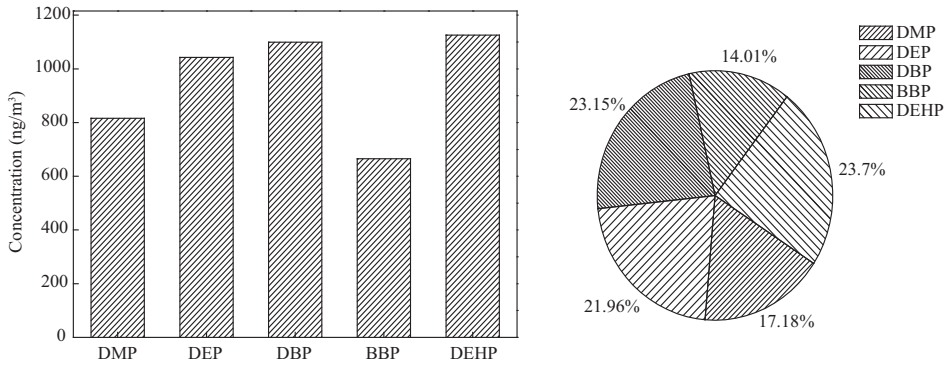

Fig. 1 – Concentration and the proportion of the 5 detected PAEs in offices.

The form and particle size distribution of ∑PAEs is demonstrated in Fig. 3. Fig. 4 shows the size distribution of each PAE. PAEs in $PM_{2.5}$ accounted for the largest amount; the proportion was 72.64%, ranging from 60.65% to 78.93%. PAEs in particles with size 2.5–5, 5–10, and above 10 μm were 5.7%, 9.18%, and 12.47% of ∑PAEs, respectively. As we all know, $PM_{2.5}$ can go directly into alveoli. As a result, these particles are more harmful to human health. Thus, great attention must be paid to this fraction. Correlation of the amount of PAEs in $PM_{2.5}$ and the total and gas-phase PAEs was analyzed.

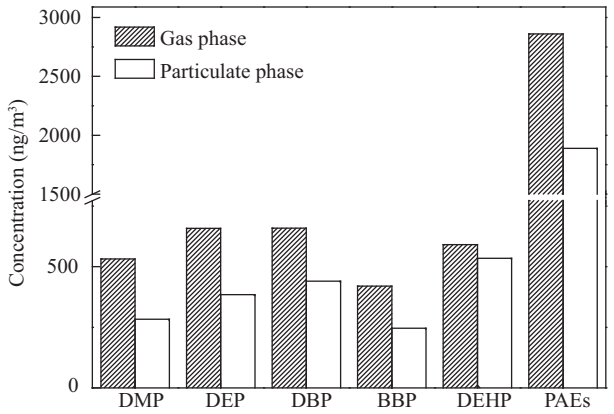

Fig. 2 – Comparison of the gas-phase PAEs and the particulate-phase PAEs in offices.

The result indicated that the amount of PAEs in $PM_{2.5}$ correlated significantly with the total particulate-phase PAEs and gas-phase PAEs, with R^2 values of 0.85 and 0.69, respectively. The sampling of $PM_{2.5}$ was very complicated, while that of the total particulate-phase was much easier. Thus, the amount of PAEs in $PM_{2.5}$ can be estimated from the total amount in particulates (TSP) using the proportion 72.64%.

Our previous research investigated the concentrations of PAEs in a study room (Pei et al., 2013). Thus, a comparison was carried out between the offices and the study room of a newly decorated house. As shown in Fig. 5, pollution characteristics were similar between offices and study rooms. The PAE concentration decreased by 48.55% (46.08% for gas-phase and 51.89% for particulate-phase) after 2 yr. The rate of decrease of PAEs had a positive relation with their initial values (except for BBP). Thus, we can estimate that PAE pollution can decrease by about 50% two years after decorating.

3. Conclusions

Based on our experimental results, the following conclusions can be drawn.

(1) PAE concentrations ranged from 3070.09 to 6700.14 ng/m^3, with a mean concentration of 4748.24 ng/m^3 in offices, of which the gas concentration accounted for 60%, and the

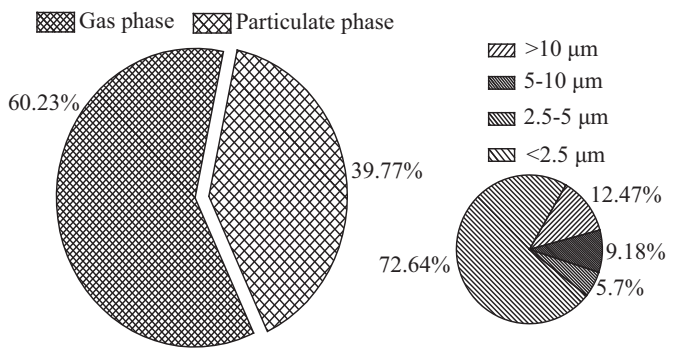

Fig. 3 – Form distribution and particle size distribution of PAEs.

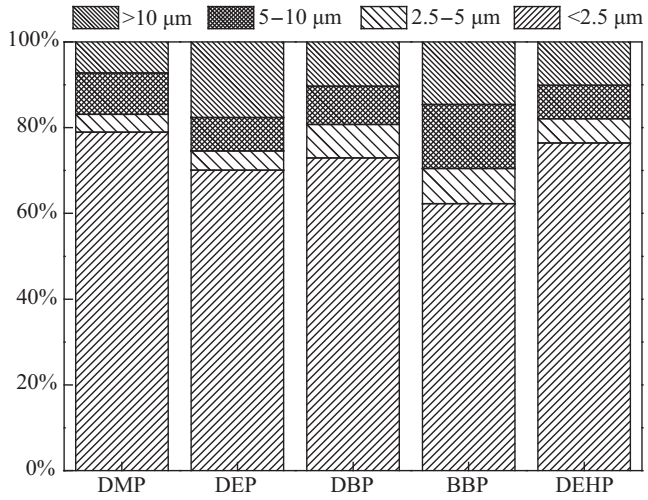

Fig. 4 – Particle size distribution of each PAE.

particulate phase accounted for 40%. In order to measure the pollution status precisely, both gas-phase and particle-phase values must be simultaneously determined. Among all the detected PAEs, DEHP, DBP, and DEP were the most abundant compounds, together accounting for 70% of the PAEs.

(2) As the amount of PAEs in $PM_{2.5}$ correlated significantly with the total particulate-phase PAEs ($R^2 = 0.85$), the amount of PAEs in $PM_{2.5}$ can be estimated from the amount of total particulate-phase PAEs using the proportion of 72.64%.

(3) Pollution characteristics of offices and study rooms are similar. Comparing newly decorated studying rooms and offices decorated for 2 yr, it is implied that PAEs in offices decreased by 50% 2 yr after decorating.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 20977075), the National High Technology Research and Development Program of China (863) (Nos. 2010AA064901, 2010AA064902). We thank all the people for their assistance with this study and their constructive advice.

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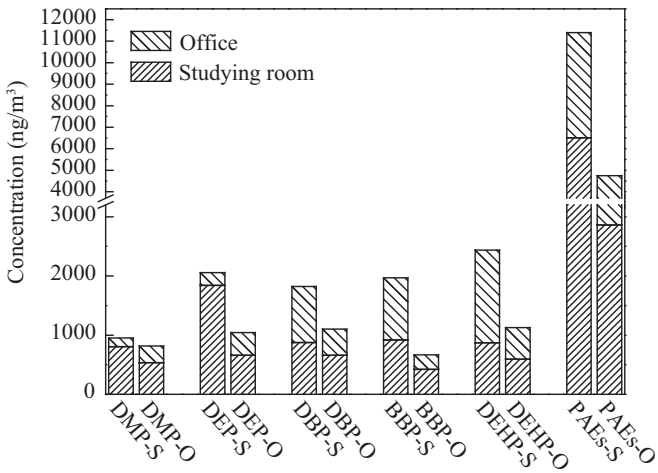

Fig. 5 – Comparison of gas-phase and particulate-phase PAEs between studying rooms and offices. O: offices, S: studying rooms.

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Journal of Environmental Sciences (Established in 1989)
Volume 28 2015

Supervised by	Chinese Academy of Sciences	**Published by**	Science Press, Beijing, China
Sponsored by	Research Center for Eco-Environmental		Elsevier Limited, The Netherlands
	Sciences, Chinese Academy of Sciences	**Distributed by**	
Edited by	Editorial Office of Journal of	Domestic	Science Press, 16 Donghuangchenggen
	Environmental Sciences		North Street, Beijing 100717, China
	P. O. Box 2871, Beijing 100085, China		Local Post Offices through China
	Tel: 86-10-62920553; http://www.jesc.ac.cn	Foreign	Elsevier Limited
	E-mail: jesc@rcees.ac.cn		http://www.elsevier.com/locate/jes
Editor-in-chief	X. Chris Le	**Printed by**	Beijing Beilin Printing House, 100083, China

CN 11-2629/X **Domestic postcode: 2-580** **Domestic price per issue RMB ¥ 110.00**

ISSN 1001-0742