



PAEs occurrence and sources in road dust and soil in/around parks in May in Tianjin, China[☆]



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ABSTRACT

This is the first study reporting the presence of six phthalic acid esters (PAEs) in 45 composite soil and road dust samples collected in the urban zone of Tianjin, China. Three sample types (one soil and two road dust) were collected from the city parks. Soil samples (SI) were obtained from inside the park, road dust samples (RDI) were gathered from inside the park roads and the others (RDA) from roads surrounding parks. The range of concentrations of Σ_6 PAEs in SI, RDI and RDA were 0.07–0.92 $\mu\text{g g}^{-1}$, 0.42–6.32 $\mu\text{g g}^{-1}$ and 0.40–7.54 $\mu\text{g g}^{-1}$, respectively. The highest SI Σ_6 PAEs concentration (0.92 $\mu\text{g g}^{-1}$ in The People's Park) was 13 times higher than that of the lowest content (0.07 $\mu\text{g g}^{-1}$ in XiLiu Park). Furthermore, the spatial distribution of PAEs in RDI showed higher contents in the Nankai and Hexi districts. PAEs concentrations in different types of roads displayed significant differences ($P < 0.05$). The RDA PAEs distribution expressed decreasing order for different types of roads such as arterial road > sub-arterial road > branch road. The results of nonparametric tests on Σ_6 PAEs revealed significant differences between every two different sample types ($P < 0.05$). The analysis of the six PAEs types indicated DnBP and DEHP were the primary contaminating compounds in all sample types. The PCA results showed cosmetics and personal care products were important sources of PAEs in SI, and plasticizers were the key sources of PAEs in RDI and RDA.

1. Introduction

Phthalic acid esters (PAEs) define numerous chemical compounds sharing a common chemical structure such as dialkyl or alkyl/aryl esters of 1,2-benzenedicarboxylic acid (Latini, 2005). The alkyl chains contain from 1 to 13 carbons (Staples et al., 1997). According to the European Union definition on semi-volatile organic chemicals (SVOCs), the physical and chemical properties of PAEs correspond to the definition of SVOCs except for the dimethyl phthalate (DMP) which belongs to the VOCs class (Wang et al., 2008). PAEs are extensively used in the plastic industry as adhesives and plasticizers for polyvinyl chloride (PVC) materials to improve their flexibility and processing ability (Gómez-Hens, 2003; Tyler et al., 1998). They are also used in other non-plasticizers industries including the manufacturing of cosmetics, insect repellents, propellants, decorative inks and lubricating oils, etc. (Peakall, 1975; Tyler et al., 1998). Several million tons of PAEs are produced worldwide every year for the fabrication of soft PVC and

other plastics (Guo and Kannan, 2011). Plasticizers are also widely used in building materials, home furnishing, transportation, clothing, and to a limited extent in food (packaging) and medical products (Staples et al., 1997). Thus, PAEs occupy an important part of our lives as a result of their widespread usage. Since PAEs are physically incorporated (physically bound) to the polymeric matrices, up to 10–60% weight, they easily permeate into the ecosystem during manufacturing, storage, usage and after disposal (Bauer and Hermann, 1997; Clausen et al., 2012; Wams, 1987). They are extensively distributed in the environment, and can be detected in air, sediments, water, and soils (Di Gennaro et al., 2005; Škrbić et al., 2016).

The distribution of phthalates across different environments has been investigated in China. Several studies have analyzed phthalates in soil samples (SI) collected from numerous Chinese cities (Niu et al., 2014; Song et al., 2015; Teng et al., 2015; Xu et al., 2008; Zeng et al., 2008b). The levels of phthalates in water samples have also been monitored from several cities (He et al., 2013; Sha et al., 2007; Zeng

[☆] Capsule: Road dust exhibited higher PAEs concentrations and the PAEs contents in arterial roads are greater than those of sub-arterial and branch roads. The source of PAEs in road dust and soil is different.

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et al., 2008a, 2009a,b). Other studies of phthalates are related to particulate matter (e.g. PM_{2.5} and PM₁₀), settled dust (indoor/outdoor) and gas-phase (Langer et al., 2010; Rakkestad et al., 2007; Song et al., 2015; Wang et al., 2008, 2013; Zhang et al., 2014). The presence of phthalates in various types of environments represents a potential threat to human and biologic health. A large number of epidemiological and toxicological studies have demonstrated the noxious effect of long-term exposure to phthalates. The adverse effects of phthalates have been confirmed on animals in short and long-term studies on rodents. Phthalates can lead to liver, kidney and testicular diseases and cause fetal death and malformations (Heudorf et al., 2007; Latini, 2005). The application of these toxicity studies to humans remains controversial and incompletely characterized (Latini, 2005).

There are several studies concerning phthalates originating from China. However, researches related to phthalates both in road dust (in and around parks) and soils (within parks) have not been conducted in China. In this study, six PAEs classified as priority environmental pollutants by the United States Environmental Protection Agency (Kong et al., 2012) were analyzed. These included dimethyl phthalate (DMP), diethyl phthalate (DEP), di-*n*-butyl phthalate (DnBP), benzyl butyl phthalate (BBP), di-2-ethylhexyl phthalate (DEHP) and di-*n*-butyl phthalate (DnOP), respectively. A brief description of their properties is given in Table 1, with the increase in molecular weights, decrease in vapor pressures and water solubility. The low molecular weights of some phthalates help their rapid diffusion in the atmospheric environment relative to the high molecular weight phthalates. The study proposes: 1) present the concentration levels of six PAEs contained in road dust (in and around parks) and soil (in the parks) samples collected in the city of Tianjin, China, 2) analyze the characteristics and distribution of sample contamination and, 3) use the principal component analysis (PCA) to identify the source of PAEs in different sample types.

2. Materials and methods

2.1. Sample sites and methods of collection

In May 2015, 45 samples were collected under stable weather conditions from 15 parks located in the city of Tianjin. Three types of samples (one soil and two road dust) were collected from each park. The soil samples (SI) were taken from each park, one of the road dust samples (RDI) was collected from a road located inside each park and the other (RDA) was sampled from a road surrounding the park. The roads located around each park can be divided into three types: arterial road, sub-arterial road and branch road. The Hongqiao North Street, Hongqi Road, Hongqi South Road, Qiongzhou Road, Youyi south Road and Beijing-Tianjin Road all belong to arterial roads. The sub-arterial roads include Chenglin Road, Zhujiang Road and Hongxing Road. Guanghua Road, No. five Wang Chuanchang Road, Water Park North Road, Huanhu Middle Road and Yingbo Road form branch roads. During the sampling procedures, the longitude and latitude of each park were recorded by a GPS device and presented in Fig. 1.

SI were collected with a cleaned stainless steel scoop from a depth of 0–10 cm below the surface layer. RDI and RDA were collected from the

pavement road surface using a brush containing no plastic. The samples were then put into aluminum foil envelopes having removed any stones, cigarette butts, branches and residual roots. The envelopes were stored in a temperature-controlled box during transport to the laboratory. To get rid of moisture, the RDI and RDA were placed in a desiccator for 3 days and SI were dried naturally. All samples were sieved using a stainless steel 60 mesh sieve. They were then kept in aluminum foil envelopes at -4°C until pretreatment.

2.2. Sample pretreatment and analysis

Detailed information about pretreatment and analytical methods is described by Kong et al. (2012) who investigated PAE concentrations in suburban soil samples in Tianjin. During the pretreatment process, 5 g of soil/road dust was used for sample extraction. The internal standard substances (50 mg/L benzyl benzoate, dissolved in hexane) and standard mixtures (2000 mg/L DMP, DEP, DnBP, BBP, DEHP, DnOP dissolved in iso-octane) were supplied by Dr. Ehrenstorfer (Augsburg, Germany). Analysis of PAEs was carried out using an Agilent 6890 N/5975B (Agilent Technologies, Avondale, PA, USA) gas chromatograph-mass spectrometer (GC-MS) with a HP-5 MS capillary column (30 m \times 0.25 mm, 0.25 μm film thickness) run in a selective ion monitoring mode. The detailed operational procedures of the GC-MS were described by Kong et al. (2012). The applied quantitative method is the internal standard method based on a five-point calibration curve.

2.3. Quality assurance and quality control

During the sample pretreatment procedure, the chemical reagents and solvents employed were of analytical grade and no plastic material was used. All glassware was soaked in pickler (a mixture of potassium dichromate, sulfuric acid and deionized water) for 12 h, and thoroughly washed in Milli-Q water (generated through a Millipore Ultrapure water system, USA), then baked at 60°C until desiccation and finally sealed with aluminum foil (to avoid contamination by laboratory air). This process is necessary to eliminate any phthalates residues in the glassware.

During the analytical process, five blank samples were analyzed to calculate the Method detection limits (MDLs) and to guarantee the quality of the analysis. The instruments were calibrated daily with calibration standards. The MDLs correspond to three times the standard deviation of the blank samples. The DMP, DEP, DnBP, BBP, DEHP and DnOP MDLs were 0.0002, 0.0033, 0.0056, 0.0003, 0.0042, 0.0003 $\mu\text{g g}^{-1}$, respectively. The recovery rates of all the analyzed phthalates were calculated using a certain amount of standard mixture having a concentration similar to that of the samples. The standard mixture was considered as a real sample undergoing the same pretreatment and analysis. The recoveries for DMP, DEP, DnBP, BBP, DEHP and DnOP were 105.06%, 95.95%, 113.96%, 88.64%, 79.78% and 82.19%, respectively.

2.4. Data analysis method

Nonparametric tests was used to identify the difference of PAEs

Table 1
A brief introduction of six PAEs.

Abbreviation	CAS-No	Molecular formula ¹	Molecular weight ¹	Vapor pressure (mm Hg) ²	Water solubility (mg/L) ²
DMP	131–11–3	C ₁₀ H ₁₀ O ₄	194.18	2.0×10^{-3}	4200
DEP	84–66–2	C ₁₂ H ₁₄ O ₄	222.24	1.0×10^{-3}	1100
DBP	84–74–2	C ₁₆ H ₂₂ O ₄	278.34	2.7×10^{-5}	11.2
BBP	85–68–7	C ₁₉ H ₂₀ O ₄	312.36	5.0×10^{-6}	2.7
DEHP	117–81–7	C ₂₄ H ₃₈ O ₄	390.56	1.0×10^{-7}	0.003
DOP	117–84–0	C ₂₄ H ₃₈ O ₄	390.56	1.0×10^{-7}	0.0005

Note:1: Data from Chemical Abstracts Service(CAS);2: Recommended values by Staples et al. (1997).

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