



Temporal Variation of Phthalic Acid Esters (PAEs) in Ambient Atmosphere of Delhi

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Received: 9 March 2018 / Accepted: 4 April 2018 / Published online: 26 April 2018
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Abstract

Phthalic acid esters (PAEs) are a group of chemical species, ubiquitously present in the environment and pose a serious risk to humans. In the present study, the average concentrations of PAEs in PM₁₀ (particulate matter ≤ 10 μm) are reported at a densely populated site in Delhi. The average concentration of PAEs was reported to be 703.1 ± 36.2 ng m⁻³ with slightly higher concentrations in winter than in summer; suggesting that sources are relatively stable over the whole year. The average concentration of PAEs was 35.7 ± 30.5 ng m⁻³ in winter, 35.4 ± 27.0 ng m⁻³ in summer, 3.4 ± 1.5 ng m⁻³ in monsoon and 7.5 ± 5.2 ng m⁻³ in post-monsoon. Principal component analysis was performed, which suggested that emissions were mainly due to plasticizers, cosmetics and personal care products, municipal solid waste, thermal power stations, industrial wastewater, cement plants and coke ovens.

Keywords Phthalic acid esters · Particulate matter · Seasonal variation · Principal component analysis · Urban pollution

The rapid increase in use of polymeric substances in new building materials, food and beverage containers and other consumer products over the past 50 years has resulted in a corresponding increase of new chemicals in living environment (Li and Wang 2015). Most of these chemicals have mutagenic and carcinogenic properties and are suspected as endocrine disrupting chemicals (EDCs) (Rudel and Perovich 2009). Phthalic acid esters (PAEs) are an important category of EDCs, which are widespread in the environment. PAEs are predominantly used as plasticizers of polymeric materials and as non-plasticizers in the manufacturing of wallpaper, toys, cosmetics, medical products, electronics, and auto-parts (Duty et al. 2004). Due to weak intermolecular forces between PAEs and their polymer products, they can easily be leached during manufacturing, storage and usage of plastic materials; making them ubiquitous environmental pollutants (Ji et al. 2014).

The most extensively used phthalates are diethyl phthalate (DEP), di-*n*-butyl phthalate (DBP), butyl benzyl phthalate

(BBP), di-(2-ethylhexyl) phthalate (DEHP), di-*n*-octyl phthalate (DOP), di-iso-decyl phthalate (DIDP) and di-isooctyl phthalate (DINP). Because of their carcinogenic and endocrine disruptive effects, the European Union placed an emergency ban on usage of six of the phthalates like DBP, BBP, DEHP, DOP, DINP, DIDP in soft toys and children's products (Xie et al. 2005). Ingestion of phthalates can be through direct exposure or by indirect sources like leaching and general environmental contamination; in case of less volatile PAEs like DEHP, inhalation is not the major route (Wormuth et al. 2006). Some scientific investigations have assessed the composition, human exposure and source profiles of PAEs in indoor as well as in ambient atmosphere (Teil et al. 2006; Srivastava et al. 2009; Fu et al. 2010; Giri et al. 2013; Kong et al. 2013a; Li and Wang 2015; Zhu et al. 2016). Zhu et al. (2016) estimated coefficient of divergence between PM₁₀ and PM_{2.5}, inhalation exposure and cancer risks for human due to PAEs in ambient air of Tianjin, China. Li and Wang (2015) studied concentrations, composition and size distribution of phthalates in mainland China and its surrounding regions. Kong et al. (2013a) explored the spatial and temporal variations of PAE concentration levels of PM₁₀ and PM_{2.5} in ambient air of Tianjin, China and performed principal component analysis (PCA) for source identification. They mainly reported correlations between PAE concentrations and temperature.

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The literature reveals that there are scattered studies on phthalates in atmospheric particulate matter (PM), in different regions of India but for Delhi, the present study is first of its kind. Although, Das et al. (2014) collected and analyzed food, water and dust samples for phthalates at Jawaharlal Nehru University (JNU) campus, Delhi and Okhla industrial Area, Delhi, but no scientific study has been reported for composition and source apportionment of PAEs in atmospheric PM₁₀ at an urban site, Delhi. Giri et al. (2013) reported phthalates ranging in concentrations from 2 to 926 ng m⁻³ with highest levels during winter in aerosol of Raipur, India. Additional plasticizers found in most of their samples were di-octyl-adipate and tri-phenyl phosphate, which were also high in winter. On the contrary, plasticizers in ambient air of Madras, India varied from 175 to 857 ng m⁻³, with high values during summer (Fu et al. 2010). The present study evaluated seven PAEs in atmospheric PM₁₀ samples: DEP, DBP, BBP, DEHP, DOP, DINP, and DIDP. The objectives of the study are to (i) determine the concentrations of PAEs in atmospheric PM₁₀ at a densely populated site, Indira Gandhi Delhi Technical University For Women (IGDTUW) campus, (ii) trace the possible source profiles of PAEs in ambient air by PCA and (iii) estimate inhalation exposure to PAEs in ambient air of Delhi. This study is important in aspect of source apportionment of non-polar molecular markers. For a highly polluted city like Delhi, mitigation strategies could be developed only if proper identification of source profiles could be developed.

Materials and Methods

The demography and climate of sampling site has been discussed in details in Gupta et al. (2017). Delhi (28°38'N and 77°20'E) the National Capital Territory (NCT), being second most populous agglomeration in India with a population of 18.2 million, is one of the most polluted cities of the world and is burdened with raised levels of atmospheric PM. The sampling site, IGDTUW campus, is located in the northern part of Delhi (Fig. 1), which have huge transport network, i.e. Inter State Bus Terminus (ISBT), Kashmere Gate and the heavy vehicular traffic road in the vicinity. Particulate matter (PM₁₀) was collected on Quartz fiber filters (QFFs, Pallflex) using a Respirable Dust Sampler APM 460 (Envirotech Instruments) at the flow rate of 1.35 m³ min⁻¹ at the IGDTUW campus during January 2015–December 2015. Two to three samples (24 h) were collected every week during the period (n = 69).

The seasonal distribution of NCT Delhi follows winter, summer, monsoon and post-monsoon (spring). The average rainfall in Delhi is 714 mm, three-fourth of which falls in July, August and September (Sharma and Dikshit 2016), but the monsoon season was delayed in year 2015 and originated

from south than the usual north-west trend (Press release, Indian Meteorological Department 2015). This climate array coupled with the El-Nino effect showed enormous increase in temperature of Asian Countries. Total 69 PM₁₀ samples were collected during winter (January–February and November–December, n = 20), summer (March–June, n = 25), monsoon (July–September, n = 16) and post-monsoon (October, n = 8) seasons to have a better understanding of seasonal variations.

The temperature ranges experienced in the summer and winter were 41–46 and 4–6°C, respectively. It remained hot until October and year 2015 has had the highest recorded temperatures since 1995 (Gopalaswami 2016). The sampling, analysis and quantification of PAEs were performed according to the procedure followed for organic markers (Gupta et al. 2017). Prior to sampling; the QFFs were prebaked at 550°C for 10 h. Before and after sampling, the filters were kept in a Secador desiccator (Tarsons) under controlled temperature (25–30°C; with automatic controller) and 35%–40% relative humidity for at least 24 h to prevent hydration of the filter surface. 1 cm² filter paper was cut into strips and extracted with 15 mL of HPLC grade Dichloromethane (DCM) using ultrasonic agitation for 15 min and the same repeated thrice. The extract was concentrated by using a rotary evaporator at temperature 30–40°C under gentle vacuum and filtered through a membrane filter (PVDF 0.45 µm micro syringe). The extract was further reduced up to 2 mL, under a gentle stream of nitrogen.

Detection and quantification of phthalates were carried out with Gas Chromatograph-Mass Spectrometer (Model GCMSQP2010 Plus, Shimadzu, Japan) system equipped with a fused silica capillary Rxi-5Sil MS (Restek, Bellefonte, PA, USA) of 30 m length, 0.25 mm id, and 0.25 µm d_f. Helium (99.999% pure) was used as a carrier gas. All samples were run according to a standardized temperature program (59 min/sample) as reported by Gupta et al. (2018). A distinctive peak fragmentation pattern for different phthalates was detected by MS detector in Full Scan mode. Phthalate esters were identified on the basis of retention time (Rt) of the sample peaks in chromatogram with the Rt of external phthalate standard (4S8231 - SS EPA Phthalates Esters Mix, Supelco). Library database [National Institute of Standards and Technology library (NIST05) and Wiley 8] was used to confirm the identification of peaks by comparing them with the corresponding standard mass spectra. Three point calibrations were done by plotting the mean peak area with the concentrations to make calibration curve for each component present in the standard mixture. Three different volumes of standards (0.4, 0.8, 1.0 ng µL⁻¹) were spiked on the blank quartz fiber filters, extracted and analyzed by following the same procedure as used for the analyte samples. Recovery

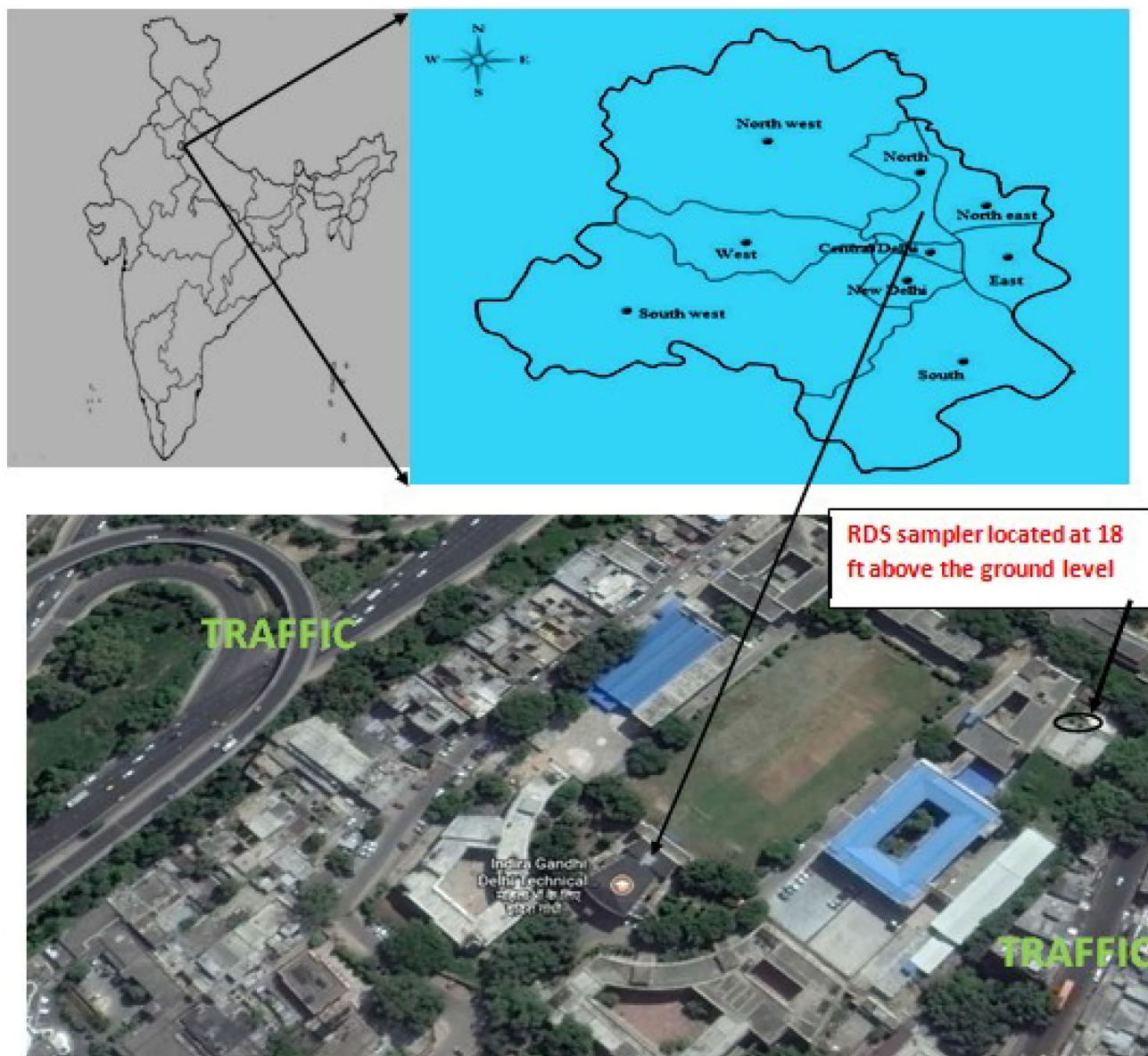


Fig. 1 Sampling site – Indra Gandhi Delhi Technical University for Women, Delhi

experiments were performed and overall recoveries of each analyte were in the range of 85.74%–112.41%. The Limit of Detection (LOD) and Limit of Quantification (LOQ) of the method were quantified from the standard deviation of field blank samples. The values for LOD for DEP, DBP, BBP, DEHP, DOP, DINP, and DIDP were 0.05, 0.05, 0.07, 0.08, 0.04, 0.05, 0.01 ng m^{-3} , respectively and values for LOQ were 0.13, 0.15, 0.21, 0.22, 0.12, 0.13, 0.01 ng m^{-3} , respectively. Reproducibility of representative samples and standard solutions was ascertained by processing same analysis thrice. The results were reproducible with an error less than $\pm 5\%$.

Results and Discussion

The average concentration of total PAEs ($703.1 \pm 36.2 \text{ ng m}^{-3}$) showed a complex seasonal pattern with slightly higher concentrations in winter than in summer, suggesting that sources are relatively stable over the whole year. The seasonal average concentration of PAEs was $35.7 \pm 30.5 \text{ ng m}^{-3}$ in winter, $35.4 \pm 27.0 \text{ ng m}^{-3}$ in summer, $3.4 \pm 1.5 \text{ ng m}^{-3}$ in monsoon and $7.5 \pm 5.2 \text{ ng m}^{-3}$ in post-monsoon (Fig. 2). Due to semi-volatile nature of phthalates, the gas/particle partitioning of phthalates is temperature dependent. Appreciable concentrations in winter are due to higher amount of combustion activities with calm weather

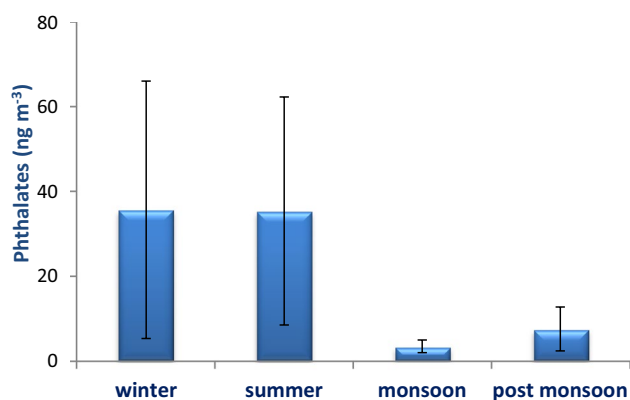


Fig. 2 Seasonal average concentrations of phthalates in PM₁₀ aerosols

conditions and a low boundary layer which does not favor atmospheric dispersion. In winter, long-range transport from origin of sources to other parts may influence the urban ambient atmosphere.

Although the sampling site is a traffic hotspot, but other potential sources in the surroundings of the sampling site include municipal solid waste (MSW) burning, construction activities, soil and road dust and fly ash suspension. A significant contribution from consistent sources was additionally observed during all the seasons like construction and demolition activities. In summer season, appreciable concentration of phthalates is probably due to convection re-suspension of dust by Loo-episodes of dry winds originating from Pakistan and Northwest India (Mishra and Shibata 2012). The crop residue burning (CRB) effect also increases the atmospheric PM and organic compounds in urban atmosphere of Delhi unexpectedly. The particles emitted from the CRB (practice prevalent in Punjab and Haryana) were transported to Delhi by two major wind patterns; western and north-western upwind disturbance (Gupta et al. 2017). Another prominent factor, responsible for increased PAE concentration levels in summer, is their weak intermolecular bonding to polymer matrices. Elevated temperature conditions in ambient atmosphere enhance volatilization of PAEs from plastics. Teil et al. (2006) suggested that the seasonal variations of PAEs in the atmosphere of Paris were primarily linked to temperature evolution and were next linked to rainfall pattern. The rainfall during monsoon may dilute or wash out the pollutants in ambient air, which could be the prime reason for low concentrations in monsoon and post-monsoon seasons.

The average concentrations of individual PAE in PM₁₀ are presented in Fig. 3. DEHP and DBP were the dominant species among all the PAEs. This is due to their high utility as plasticizers in manufacturing of articles made of PVC, even after the emergency ban put by the European Commission; DEHP is used globally as plasticizers in PVC due to its low

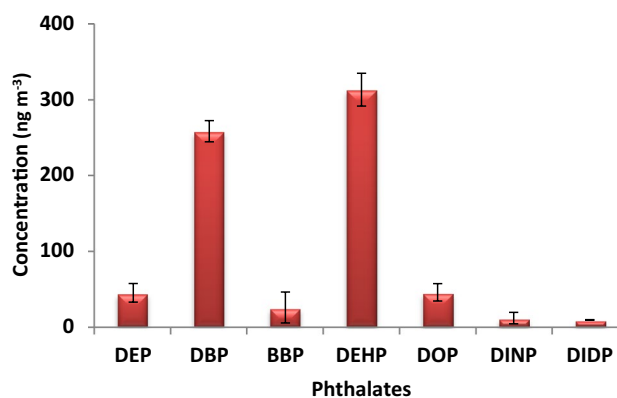


Fig. 3 Average concentrations of individual PAEs in PM₁₀ aerosols

cost and wide applications while DBP, being a low-molecular weight phthalate, is generally used in glues, glow sticks and personal care products. DMP was not detected in any sample, which may be related to its limited uses and existence in vapor phase. DEP, BBP and DIDP were not detected in most of the samples in winters.

PAEs concentrations in the present study are comparable and sometimes higher in other Indian and some Chinese cities (Table 1) (Teil et al. 2006; Wormuth et al. 2006; Fu et al. 2010; Giri et al. 2013; Kong et al. 2013a; Zhu et al. 2016). In a similar study, Phthalates in aerosols at Raipur, India varied between 2 and 926 ng m⁻³, with higher concentrations in winter (Giri et al. 2013). On the contrary, Fu et al. (2010) observed higher concentrations of phthalates in aerosols during summer than during winter at Chennai, India. Kong et al. (2013a) also observed higher concentrations of PAEs in winter at different sites of Tianjin, China, whereas in the same region, Zhu et al. (2016) observed higher level of PAEs in summer. This seasonal heterogeneity may be due to the varied conditions, sampling locations, time of year and particle size distribution, but all of them provided an indication of the association of PAEs in the ambient aerosols in different regions (Wang et al. 2008; Kong et al. 2013a).

Human exposure via inhalation is very significant for semi-volatile organic species like PAEs. PAEs are introduced in the atmosphere by direct out-gassing and inhaled directly by human beings. The gas and particle distribution of PAEs demonstrate a significant impact on atmospheric composition, carcinogenic and endocrine disruption effects on human beings. PAE concentrations in the gas phase, $C_{g,10}$ (ng m⁻³), can be calculated from the PAE concentrations in the particle phase by using following equation from Zhang et al. (2014):

$$C_{g,10} = \frac{F_{10}/PM_{10}}{K_p}$$

Table 1 Comparison of concentration levels of PAEs (ng m⁻³) in PM₁₀ with previous studies

Sampling site	n ^a	DMP	DEP	DBP	BBP	DEHP	DOP	DINP	DIDP	References
IIT, Chennai, India	49	0.13	9.03	231	–	550	–	–	–	Fu et al. (2010)
Raipur, India	17	–	–	145.35	–	121.05	–	–	–	Giri et al. (2013)
Tianjin, China	35	0.88	0.73	12.9	0.15	98.29	0.83	–	–	Kong et al. (2013a)
Tianjin, China	16	0.18	0.08	2.24	0.02	10.79	0.1	–	–	Zhu et al. (2016)
Paris, France ^b	–	0.5	10.7	22.2	4.6	18.9	0.5	–	–	Teil et al. (2006)
Delhi, India	69	–	44.1	258.1	24.6	313.1	44.7	10.6	8.1	Present study

^an is number of samples

^bMeasurements in total suspended particulates

where PM₁₀ is the average concentration of particulate matter (PM₁₀) (μg m⁻³) during the sampling period; F₁₀ is the concentration of PAEs in atmospheric PM₁₀ (ng m⁻³); K_p is the particle–gas partition coefficient (m³ μg⁻¹). The values of K_p have been calculated on the basis of octanol/air partition coefficient (K_{OA}) method used by Weschler et al. (2008). The K_{OA} values have been taken from the literature (Cousins and Mackay 2000). During open burning of MSW, plastics and related products, PAEs enter in the environment and get distributed between gas and particle phases. The values of C_{g,10} for DEP, DBP, BBP, DEHP, DINP, DOP and DIDP were 31.42, 11.45, 0.14, 0.26, 0.01, 0.01, 0.21 ng m⁻³, respectively. The concentrations of phthalates in the gaseous phase are significantly lower in the present study, indicating lower risk of exposure. C_{g,10} have the lower values for those phthalates which have relatively large abundance and low vapor pressure (Weschler et al. 2008). DBP and DEHP have the highest concentrations in particulate phase but have the lower values in gaseous phase than DEP. Similar results have also been reported in Berlin by Weschler et al. (2008). They showed that the DEHP was abundant in particle phase followed by DBP. On the contrary, Teil et al. (2006) observed that 64.9%–93.8% of PAEs in Paris were in the vapor phase; indicating that phthalates with alkyl chain with less than six carbon numbers (from DMP to BBP) are primarily in the vapor state whereas phthalates with longer alkyl chains are associated with particulates.

PCA was performed to categorize the possible sources of PAEs in atmospheric PM₁₀ of Delhi. PCA is a multivariate statistical technique that can be used to simplify

the interpretation of complex system and large datasets to extract a few new factors and convert the data into graphical form. Each of these factors can be identified as emission sources to assess the source profiles of organic tracers (Kong et al. 2013a; Zhu et al. 2016). Total 69 PM₁₀ samples have been analyzed through PCA with a varimax normalized rotation to maximize (or minimize) the values of loading factors of all compounds. To obtain more reliable results, n > 30 + (V + 3)/2 (where, n = number of samples and V = number of variables) (Callén et al. 2009) was considered for PCA. The statistical significant correlation among DBP–DEHP (0.726) and BBP–DOP (0.914) indicate that they have common origin and have similar environmental behavior. DIDP presented negative correlation, which may be due to photochemical degradation of the phthalate in ambient atmosphere (Table 2).

From PCA, comparison between different values of data analysis through a diagram laid the main trends and their hierarchical distribution (Fig. 4). Systematic investigations of the links between variables lead to the construction of a few synthetic variables called principal components. The same significance is attributed to each variable and the same weight to each subject. The cosine values, being the geometrical basis, follow same trends as the correlation coefficient (Teil et al. 2006). Two factors; Factor 1 (F₁) and Factor 2 (F₂) were extracted, which explained 67.76% of the data variance (Fig. 4). The first factor, F₁, loaded by DBP and DEHP, accounted for 39.39% of the data variance, being tracers of emissions from plasticizers, cosmetics and personal care products, as well as emissions from MSW, thermal power

Table 2 Statistical Pearson correlation between different phthalates

Variables	DEP	DBP	DEHP	BBP	DiNP	DOP	DIDP
DEP	1	0.613	0.252	0.143	0.440	0.197	–0.118
DBP	0.613	1	0.726	0.113	0.594	0.069	–0.358
DEHP	0.252	0.726	1	0.269	0.460	0.318	–0.403
BBP	0.143	0.113	0.269	1	0.156	0.914	–0.232
DINP	0.440	0.594	0.460	0.156	1	0.099	–0.316
DOP	0.197	0.069	0.318	0.914	0.099	1	–0.169
DIDP	–0.118	–0.358	–0.403	–0.232	–0.316	–0.169	1

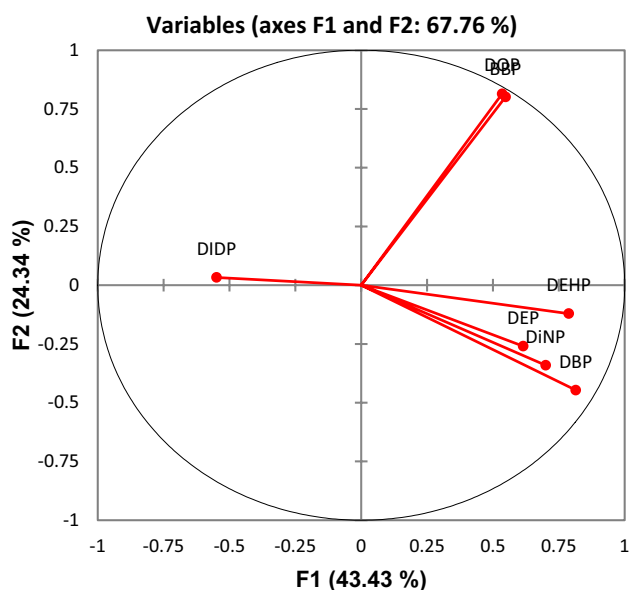


Fig. 4 Factor distribution of PAEs in PM_{10} aerosols

stations, industrial wastewater, cement plants and coke ovens (Kong et al. 2013a; Zhu et al. 2016). In a study by Cai et al. (2007), DEHP accounted for 24%–95% of the 6 measured species of PAEs in sewage sludge from 11 wastewater treatment plants of mainland and Hong Kong, China. Liu et al. (2010) observed that their samples were heavily loaded with DEHP and DBP (78% and 10%) of the detected 16 PAEs in surrounding environment of a MSW landfill. F_2 showed a load of DIDP and DOP, indicating the influence of vinyl flooring, synthetic leather, inks, adhesives and food products (Kong et al. 2013a; Zhu et al. 2016). After varimax rotation, the factor scores of each observation with the factors F_1 and F_2 shows that maximum number of samples well correlated to the first factor representing source profiles from plasticizers and personal care products. Other studies reported diversity in relevant sources; with diet as the major source of human exposure to PAEs in the United States, European Union and Japan (Wormuth et al. 2006; Itoh et al. 2007; Guo and Kannan 2011).

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