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Levels, Distribution and Source Characterization of Polycyclic Aromatic Hydrocarbons (PAHs) in Topsoils and Roadside Soils in Esbjerg, Denmark

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Abstract A soil survey was performed to determine the levels, distributions and sources of 6 polycyclic aromatic hydrocarbons (PAHs) in 9 selected soil environments in Esbjerg, Denmark. In all, 24 soil samples were collected and the PAHs present were extracted with dichloromethane and analysed using GC/MS/MS with ion trap detector (TCD). There were elevated levels of the individual as well as the total PAHs in the soil samples and also, all 6 PAHs were present in all the soil samples. The most abundant components were fluoranthene, benzo(b)fluoranthene and benzo(a)pyrene. The average sum of the 6 PAHs in all soil samples was 2.5 mg.kg^{-1}, with range from 0.24 to 7.6 mg kg^{-1}. The total mean PAH concentration obtained was 1.67 times higher than the total limit set by the Danish Environmental Protection Agency (DEPA) whiles the mean benzo(a)pyrene (BaP) concentration (0.6 mg kg^{-1}) also 6 times the Soil Quality Criteria (Human Health) (0.1 mg kg^{-1}) by DEPA and 2 times the Maximum Permissible Concentrations (0.26 mg kg^{-1}) by the Netherlands for BaP. This shows that there are elevated levels of PAH deposition on the Esbjerg soil environment which needs an urgent attention. The diagnostic ratios and the correlation analysis identified mixed petrogenic and pyrogenic sources as the main contributors of PAHs on the Esbjerg environment.

Keywords Source characterization · Polycyclic aromatic hydrocarbons · Top soils · Road dust

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Polycyclic aromatic hydrocarbons (PAHs) are a group of dangerous xenobiotic compounds that man introduces into the environment in large quantities daily with little or no awareness. These are a suit of organic compounds released into the environment as gas particles during incomplete combustion of organic material. PAHs have a number of sources including: mobile sources such as cars, buses, trucks, ships, and aircrafts; industrial sources such as power generation, steelworks, coke ovens, aluminium production, and cement kilns and oil refining (Biswas et al. 2003). Domestic sources include combustion for heating and cooking especially solid fuel using coal and wood. Fires and smokes resulting from burning of vegetation in agricultural process, bushfires, grilling of food, or tobacco smoke (Arias-Estévez et al. 2007) results in the releases of large amount of PAHs into the environment.

Polycyclic aromatic hydrocarbons (PAHs) occur ubiquitously in the environment (Liu et al. 2005) and can be found in sediments, soils and water either in solution or adsorbed on particulate material. Most PAHs in the environment are from incomplete burning of carbon—containing materials like oil, wood, garbage or coal. Even though PAHs are associated with combustion processes, it has also been suggested that biological sources may be more important than previously assumed. There is some evidence that at least some PAHs are produced biologically.

Thus, the soil is contaminated with PAHs mainly from atmospheric depositions, directly or via vegetation, or in the case of arable soil with biowaste used as fertilizers (Maliszewska-Kordybach et al. 2008). Soil contamination by PAHs is considered to be a good indicator of the level of environmental pollution by human activities (Chung et al. 2007). It can provide information on regional pollution sources, the long-range transport of PAHs, the rates of pollutant retention and their ultimate destination (Nam et al. 2008; Yin et al. 2008).

Health effects resulting from PAH exposure have been discussed extensively in recent times (Shen et al. 2008). This include growth retardation, low birth weight, small head circumference, low IQ, damaged DNA in unborn children and the disruption of endocrine systems, such as estrogens, thyroid and steroids. Skin changes (thickening, darkening, and pimples) and reproductive related effects such as early menopause due to destruction of ovum have also been identified with PAHs. PAHs are dangerous, thus, increases risk of cancer (breast cancer) and creates advance glycogen end product which leads to an increased risk of coronary heart disease and diabetes (Anyakora et al. 2004; Gray 2008). PAHs from soil and water are very dangerous to human health because plant root uptake, bioconcentrate and translocate them (Liu and Korenaga 2001).

In 2002, air quality was measured in four Danish cities and at two background sites. Nitrogen dioxide (NO_2) and PM_{10} were at several stations found in concentrations above the new EU limit values, which member States would have to comply within 2005 and 2010. Esbjerg a Danish harbour city in southwest Denmark has not had its air quality measured over the period under review. Esbjerg with a population of 71,025 (2009) is the fifth largest city in Denmark. Its municipality has a population of 114.806 (Esbjerg Kommune: Population in Esbjerg 2nd quarter 2009). Esbjerg derives most of its energy from coal-run power plant and incineration facility which are all possible sources of PAHs in the environment (Biswas et al. 2003).

Although private car ownership in Esbjerg is low by OECD standards (35 vehicles per 100 persons), the number of diesel-powered cars increased in the last decade, resulting in high PM pollution in closure to traffic roads. From 1985 to 2004, usage of diesel fuel in the transport sector increased by 32%. Estimates show that the concentration of PM 2.5 can be reduced by 20% by installing filters on all diesel-power/heavy vehicles in Denmark even very old ones and by introducing more low emissions zones in the cities (Ministry of Traffic 2003). It would be very helpful to also improve education about eco- driving.

A study by Stepien et al. (2010) concluded that the average measured concentrations of PM 10 in 8 sites out of 13 of Esbjerg were above 20 $\mu g/m^3$. The particulate matter levels from the study were quite high compared to the annual national averages in Denmark. The reason for such a high PM pollution in the city of Esbjerg has been investigated and three possible main PM sources were identified; vehicular traffic, industrial activities and the activities at the harbour (Stepien et al. 2010).

The present study looked at the levels, distribution and characterized the possible sources of the 6 carcinogenic/mutagenic polycyclic aromatic hydrocarbon depositions on the soils of the Esbjerg environment.

Materials and Methods

Top soils (at the depth of 0–5 cm) used in this study were collected from 9 main different functional sections in the Esbjerg area. These sampling areas could be categorized into industrial, non—industrial and roadside. Road dust soil samples were also collected for comparison. In total 24 soil samples were collected, and each sample consisted of 3–4 sub-samples collected randomly from the surrounding of each site and bulked together to form one composite sample. Among the 24 soil samples, 6 were from roadsides (Station, Beach Area), 9 were from an incineration facility and a power plant (Incineration, 100 m off Incineration Plant, Power Plant), 3 were from industrial areas (Industrial Area), 3 were from waste water treatment facility (WWTA) and 3 road dust soils. All the samples were immediately wrapped in an aluminium foil and transported to the laboratory. The composite soil samples were air dried for 4 days and sieved through 2-mm mesh after removing stones, residual roots and other unwanted materials, then sealed in an aluminium foil and kept in the refrigerator prior to analysis of PAHs.

Dichloromethane, acetone, acetonitrile, isooctane and cyclohexane solvents were of chromatographic grade and purchased from Sigma–Aldrich, Germany. The stock reference standard of 6 PAHs (not a mixture) from Sigma–Aldrich, Germany includes fluorene, fluoranthene, benzo (k) fluoranthene, benzo (b) fluoranthene, benzo (a) pyrene, and benzo (ghi) perylene at 98% purity.

Soil samples (about 10 g dry weight) were weighed precisely and were then covered with 50 mL dichloromethane (DCM) and sonicated for 24 h at room temperature (Jorgensen 1999; Guerin 1999). The particle/liquid suspension and additional DCM used to rinse the jar were then filtered using a 0.2-μm pore size PTFE filter. The solvents were removed by rotary evaporation after adding 5 mL of isooctane and 3 mL internal standard Methylheptadecanoic acid (IS MHD). This was further concentrated (at 55°C) to approximately 2 mL prior to clean-up. The samples were cleaned up using solid phase extraction (SPE) columns (Isolute SPE column USA: Isolute PAH 1.5 g/6 mL Part # 927-0150-C). Dichloromethane was used to elute the PAH fractions from the SPE (Cui et al. 2002) and finally evaporated to dryness under a gentle stream of pure nitrogen, then dissolved to 3 mL with cyclohexane for analysis of the PAHs. The final concentrated extracts from the soil samples were analyzed using Varian 8200 Cx Auto Sampler and Varian 3800 GC coupled with Varian Saturn 2000 GC/MS/MS with ion trap detector (TCD).

The analytical precision and recovery was checked by the analysis of variance for each triplicate of soil samples and PAH results were not significantly different at the 95%

confidence level. There was a 98% recovery of each triplicate analysis of the internal standard Methylheptadecanoic acid (IS MHD). The internal standard was also run along with the samples to ensure accurate readings. The Limits of Detection (LOD) is as follows: [fluorene, benzo (k) fluoranthene, benzo (b) fluoranthene, benzo(a)pyrene and benzo(ghi)perylene] = 0.001 mg kg^{-1} and [fluoranthene] = 0.005 mg kg^{-1}.

Results and Discussion

The total PAHs in topsoil of Esbjerg city were from 0.24 mg kg^{-1} to 7.6 mg kg^{-1} (dry weight) (Table 1), and the mean concentration of total PAHs was 2.5 mg kg^{-1} for the 6 PAHs. The total mean concentration obtained is 1.67 times higher than the total limit set by the Danish Environmental Protection Agency (DEPA) (1.5 mg kg^{-1}) (CCME (Canadian Council of Ministers of the Environment) 2008). High molecular weight PAHs with 4–6-rings had elevated levels of the total soil PAHs in topsoil of Esbjerg city.

The mean benzo (a) pyrene (BaP) concentration in Esbjerg soil is 0.6 mg kg^{-1} and this value is 6 times the Soil Quality Criteria (2002) (Human Health) (0.1 mg kg^{-1}) set by DEPA (2002) and 2 times that of the Maximum Permissible Concentrations (0.26 mg kg^{-1}) set by the Netherlands for BaP (CCME (Canadian Council of Ministers of the Environment) 2008). This shows that there are elevated levels of PAH deposition on the Esbjerg soil environment which needs an urgent attention.

The highest concentrations of the 6 selected PAHs were identified in soils which were very close to heavily traffic roads and near an incineration facility. Influences from most PAH sources (industrial and transport) are evident. The PAH atmospheric dispersion is greater in areas with heavy vehicular activity, and a high level of PAH deposition occurred at roadside soils.

All the 6 PAHs analysed (fluorene, fluoranthene, benzo (k) fluoranthene, benzo (b) fluoranthene, benzo(a)pyrene and benzo(ghi)perylene) were present in all the top soil samples (Table 1). However, fluoranthene was generally higher than the other PAHs at almost all the nine (9) sites. A similar study conducted in Great Britain (Barnabas et al. 1995) stated that fluoranthene usually has the highest concentrations in topsoil. In fact, total PAH concentration in main roadsides soils recorded 7.6 mg kg^{-1} with benzo (a) pyrene concentration of 2.2 mg kg^{-1} which is about 30%. The benzo(a)pyrene levels are high and there is the potential for exposure to high levels of carcinogenic PAHs for road users and those living in those areas (Essumang et al. 2006). In Esbjerg, most of the residential areas are very close to major roads thereby putting many people at the risk of PAH exposure. The high levels of benzo(a)-pyrene in the soils is an indication of both pyrogenic and petrogenic sources of PAH pollution on the environment. This is quite worrying as BaP is a known carcinogen (Denissenko et al. 1996).

Polycyclic Aromatic Hydrocarbons (PAH) ratios of selected compounds are generally considered to be a good indicator of the pollution sources and of the mechanism of PAH transport into the soil. The ratios Ind/(Ind + BghiP) and Flt/(Flt + Pyr) are often used to distinguish between pyrogenic and petrogenic sources. Combustion-derived PAHs (pyrogenic) usually contains the high molecular weight PAHs (HMW-PAHs) which are abundantly generated at high temperature, while the petroleum-derived residues (petrogenic) contain relatively high abundances of Low molecular weight PAHs (LMW-PAHs) (Mai et al. 2003). Then, the ratio of LMW/HMW could be an index used to apportion the anthropogenic sources of PAHs.

Table 1 Mean concentration of PAHs in the soils of the Esbjerg environment (n = 3) in mg/kg

PAHs	Fluorene	Fluoranthene	Benzo (k) fluoranthene	Benzo (b) fluoranthene	Benzo(a) pyrene	Benzo(ghi) perylene	∑6 PAHs (Total)
Station	0.02	1.3	0.85	0.32	0.54	0.29	3.31
Industrial area	0.01	0.43	0.04	0.04	0.03	0.02	0.57
Incineration	0.01	0.36	0.03	0.04	0.06	0.03	0.53
Power plant	0.03	0.11	0.02	0.02	0.04	0.02	0.24
100 m off incinerat Plant	0.04	0.91	1.0	1.6	1.7	1.4	6.70
Beach area	0.02	0.80	0.75	0.27	0.37	0.28	2.50
WWTA	0.02	0.21	0.08	0.05	0.05	0.05	0.47
Harbour	0.05	0.48	0.08	0.09	0.02	0.06	0.78
Road dust	0.05	2.0	1.65	0.62	2.2	1.1	7.6
Averages	0.03	0.73	0.50	0.34	0.55	0.36	2.5
Std. Dev.	0.015	0.605	0.588	0.526	0.812	0.511	2.820
Variance	0.00002	0.366	0.345	0.277	0.660	0.261	7.954

A value of Ind/(Ind + BghiP) >0.5 indicates grass/coal/wood combustion sources. Values of Flt/(Flt + Pyr) >0.4 indicates pyrogenic sources, and we can distinguish between the values 0.4 and 0.5 for fuel combustion and >0.5 for grass/coal/wood combustion sources.

The ratio of (LMW/HMW) BaP(252)/BghiP(276) as stated above can also be used as an indicator for the determination of traffic and non-traffic sources, when the value >0.6 is characteristic of traffic sources (Maliszewska-Kordybach et al. 2008; Yin et al. 2008). The values of Ant/(Ant + Phe) <0.1 and Baa/(Baa + Chry) <0.2 correspond to petrogenic sources; values >0.1 and >0.35, respectively, indicate pyrogenic sources. The values of the ratio Baa/(Baa + Chry) between 0.20 and 0.35 indicate mixed petrogenic and pyrogenic sources (Pies et al. 2008; Zhang et al. 2006).

The ratios in this study demonstrated mixed petroleum and combustion source with values ranging from 0.014 to 2.623. Most of the BaP/BghiP ratios were between 0.96 and 2.071 (>0.6) indicating traffic sources of PAHs (Maliszewska-Kordybach et al. 2008; Yin et al. 2008; Plachá et al. 2009). The station sites shows petrogenic source with mixed unburned petroleum source (<0.1 and <0.4) and with values >0.35 demonstrating predominance of combustion source. Almost all the results showed similar trends with those close to highway (100 m of Incineration Plant, Beach Area and Harbour) demonstrating distinct mixed unburned petroleum and combustion situations (Yunker et al. 2002; Zhu and Wang 2004). There is also wood burning and coal burning situations observed with values ranging from 1.2 to 5.0 for B(k)F/B(ghi)P and BaP/BghiP ratios for all the sites except for WWAT (Maher and Aislabie 1992). The diagnostic ratios are presented in Table 2.

There were similar variation patterns for the PAHs especially for fluoranthene and benzo(ghi)perylene at all the sites which may indicate a common emission sources. These two are among other five PAHs which are considered vehicular markers (García-Alonso et al. 2003) meaning that there is enormous contribution from vehicular emissions.

To assess PAH associations and their possible origins, correlation analyses were conducted among the concentration of the individual PAHs in the soil samples. It is known that where two compounds have a common source, there is more likely to be a correlation between their concentrations (Gilbert et al. 2006). Strong positive significant correlation was observed between individual PAH benzo(ghi)perylene and benzo(b)fluoranthene showing the highest PAH interrelationship with correlation coefficient of 0.983 followed by benzo(b)fluoranthene/benzo(k)fluoranthene (0.967),benzo(b) fluoranthene/benzo(ghi)perylene (0.950) with benzo(b) fluoranthene/fluoranthene, benzo(ghi)perylene/fluoranthene, benzo(k)fluoranthene/fluoranthene correlated at 0.900 and 0.883 respectively significant at 0.01 levels. The least significant correlation at 0.05 significant level was between benzo(k)fluoranthene and benzo(a)pyrene (0.800) (Table 3). The following pairs also interrelated strongly at the significant level of 0.05: benzo(ghi)perylene/benzo(a)pyrene (0.783), benzo(a)pyrene/benzo(b)fluoranthene (0.733) and benzo(ghi)perylene/fluoranthene (0.717).

The results reveal that these compounds, to a lesser extent fluoranthene, were possibly derived from a common anthropogenic origin. No significant correlation was identified between fluorene and any of the other PAHs compounds measured which indicates heterogeneous source of these PAHs.

Interrelationships among sampling sites were also analysed by Spearman's correlation method and the result is reported in Table 4. Strong positive correlation was identified between WWTA and station which was the highest followed by WWTA and industrial area with correlation coefficient of 0.943 and 0.928 respectively at 0.01 significant level. Other strong positive significant correlation

Table 2 Sample sites/PAH Ratios

Sample site/PAHs ratios	F/F + FTH	FTH/FTH + BkF	B(k)F/B(ghi)P	BaP/BghiP	BkF/BkF + BbF	BbF/BbF + BaP
Station	0.014	0.606	1.285	1.891	0.729	0.369
Industrial area	0.023	0.919	1.727	1.409	0.5	0.551
Incineration	0.037	0.917	1.179	2.071	1	0.376
Power Plant	0.213	0.828	1.211	2.052	0.511	0.361
100 m of incineration plant	0.046	0.476	0.735	1.259	0.378	0.489
Beach area	0.027	0.516	2.623	1.296	0.733	0.425
WWTA	0.101	0.717	1.68	0.96	0.609	0.529
Harbour	0.091	0.855	1.266	0.375	0.485	0.782
Road dust	0.023	0.548	1.498	1.961	0.725	0.224

Fluorene (F), Fluoranthene (FTH), Benzo(b, k) Fluoranthene [B(b)F], Benzo (k) Fluoranthene [B(k)F],Benzo(a)pyrene (BaP), benzo(ghi)perylene [B(ghi)P]

Table 3 Correlation of the PAHs

	Fluorene	Fluoranthene	Benzo (k) fluoranthene	Benzo(b) fluoranthene	Benzo(a)pyrene	Benzo(ghi) perylene
Fluorene	1					
Fluoranthene	0.25	1				
Benzo (k) Fluoranthene	0.367	0.883**	1			
Benzo (b) Fluoranthene	0.417	0.900**	0.967**	1		
Benzo(a)pyrene	0.117	0.717*	0.800**	0.733*	1	
Benzo(ghi)perylene	0.433	0.883**	0.950**	0.983**	0.783*	1

* Correlation is significant at the 0.05 level (2-tailed)

** Correlation is significant at the 0.01 level (2-tailed)

Table 4 Correlation of PAHs with the sampling sites

	Station	Industrial area	Incineration	Power plant	100 m off incineration Plant	Beach area	WVWA	Harbour	Road dust
Station									
Industrial area	0.899*	1							
Incineration	0.829*	0.812*	1						
Power plant	0.543	0.348	0.6	1					
100 m off Incineration plant	0.086	0.087	0.371	−0.257	1				
Beach area	0.943**	0.754	0.714	0.486	0.029	1			
WWTA	0.829*	0.928**	0.6	0.143	−0.086	0.771	1		
Harbour	0.543	0.812*	0.429	0.029	−0.257	0.429	0.886*	1	
Road dust	0.771	0.493	0.771	0.543	0.429	0.829*	0.371	−0.029	1

* Correlation is significant at the 0.05 level (2-tailed)

** Correlation is significant at the 0.01 level (2-tailed)

coefficients at 0.05 levels were as follows: industrial area/station (0.899), harbour/WWTA (0.886), Road dust/Beach area and Incineration/Station, WWTA/Station (0.829) as well as Harbour/Industrial area and Incineration/industrial area recording 0.821. This demonstrates that, the sources of PAHs in these areas are likely coming from a common source. Power plant and 100 m off-incineration plant did not show any interrelationship with any of the other sites which probably means different source of pollution to these two areas. As it has been shown by the PAH diagnostic ratios of source assessment, it could be inferred that those areas correlated may be linked to petrogenic sources of pollution.

The result obtained from this study clearly demonstrates that the influences from most PAH sources (industrial, energy and transport) are evident. The average concentration of PAHs in the soil ranged from 0.029 mg kg^{-1}, for fluorene to 0.734 mg kg^{-1} fluoranthene. Most of these PAHs have been classified as carcinogenic and therefore their presence in the environment ought to be minimized. All the 6 PAHs analysed were present in the soils at significant levels exposing people to the harmful effects of these chemicals. The diagnostic ratios gave distinct mixed unburned petroleum and combustion situations (Zhu and Wang 2004; Yunker et al. 2002). There was also an observed wood and coal burning situations with values ranging from 1.2 to 5.0 or B(k)F/B(ghi)P and BaP/BghiP ratios for most of the sites (Maher and Aislabie 1992). The correlation analysis identified mixed petrogenic and pyrogenic sources as the main contributors of PAHs on the Esbjerg environment.

Carcinogenic PAHs pollution in Esbjerg owing to vehicular, thermal, industry in both outdoor and indoor air is hitherto a scarcely explored problem. More studies are ongoing to strengthen this database and investigate the range and profile of carcinogenic PAHs content in the Esbjerg environment.

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