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[Research]

Transport of Polycyclic Aromatic Hydrocarbons in a Calcareous Wetland Soil

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ABSTRACT
Knowledge of transport and degradation of polycyclic aromatic hydrocarbons (PAHs) is important in assessing PAH contamination of soils and water resources. The transport of naphthalene, anthracene, pyrene and phenanthrene was determined in a contaminated calcareous soil obtained from the Shadegan wetland (Khozestan, Iran) considering a column study in laboratory conditions. The PAHs were added to the top 5 cm of soils at 75 and 150 mg kg^{-1} and leachates were collected for 10 pore volumes. PAH concentrations in the leachate fractions and soils (0-5, 5-10, 10-15 and 15-20 cm depths) were determined using high pressure liquid chromatography. Results showed that naphthalene had the most mobility among the PAHs, and differences among the mobility of selected PAHs were related to their water solubility and structure. Mass balance demonstrated that among the selected PAHs, anthracene and naphthalene were the most and the least recalcitrant compounds, respectively. Hence, naphthalene could have the greatest potential to contaminate surface and ground waters which should be considered apparently.

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are recalcitrant organic pollutants which typically formed during the incomplete burning of organic materials including wood, coal tar, oil, gasoline and garbage (Blumer, 1976; Freeman & Cattel, 1990; Lim *et al.*, 1999). Historically, PAHs have been associated with human activities such as cooking, heating homes and industries and fuel for operating automobiles, and also with natural resources, such as forest fires (Harvey, 1991; Jenkis *et al.*, 1996). The presence of PAHs has been reported in soil, sediment and natural water samples at various locations around the world (Chen *et al.*, 2004; Tang *et al.*, 2005).

Some PAHs and their derivatives found to show strong carcinogenicity and mutagenicity (IARC, 1983), the fate of PAHs released in the environment has attracted much attention. Transport and biochemical transformations control the fate of PAHs in contaminated soils. Therefore, they are not expected to be uniformly distributed in the soil profile, but vary in a pronounced way down the profile (Luthy *et al.*, 1994; Weigand *et al.*, 1998). Solute transport at any site depends on various factors including abundance and activity of soil biota, soil properties as well as site factors (e.g. slope position, drying intensity, vegetation) and management (e.g. cropping, tillage, traffic) (Jarvis, 2007).

The seepage of PAHs in contaminated soils depends on the rates of PAH release from the source materials, the affinity of dissolved PAHs toward soil constituents, and the composition of the solution phase (Powers *et al.,* 1994; McGroddy *et al.,* 1996). Dissolved organic matter may modify contaminant mobility by forming an association with the PAHs and increasing the solubility of these compounds (Gauthier *et al.,* 1986; Kogel-Knabar & Totsche, 1998). Depending on the affinity of the dissolved organic matter toward the solid phase, mobility of the PAHs may be enhanced (Magee *et al.,* 1991) or diminished (Totsche *et al.,* 1997).

Weigand et al. (2002) reported that the rates of sorption and release of anthracene are limited and its mobility is controlled by non-equilibrium processes. In column experiments, PAHs are often introduced with percolating solution (Brusseau et al., 1991a; Liu & Amy, 1993) or with a separate contaminated solid phase (Grathwohl et al., 1993). Estimation of contaminants transport is performed by observation of breakthrough curves (BTCs). The shape of BTCs is used for identification of specific interactions between the solution and solid phase.

Since marine, estuaries and wetlands are important ecosystems, understanding transport and fate of pollutants (e.g. PAHs and heavy metals) in these regions is also important (Nasrollahzadeh, 2010). In spite of anthropogenic pressures such as land use change (Charkhabi *et al.,* 2008), estuaries remain dynamic and productive ecosystems, and are areas of immense ecological, environmental and economic value. Varied habitats and high rates of primary production allow estuaries to be home to a diversity of plant and animal life that support recreational and commercial fisheries (National Science and Technology Council, 1995). Consequently, understanding the fate and transport of PAHs in estuarine environments is imperative to maintaining the health of these invaluable environments. Shadegan wetland is an important ecosystem in southwestern Iran, polluted with PAHs (Shafiee *et al.,* 2006) and heavy metals (Soleimani *et al.,* 2009). The existence of PAHs and heavy metals in the soil and water resources in this wetland with almost 400000 ha area reflects the necessity of research regarding transport of these compounds in the soil.

Most of the research on PAHs (e.g. Wayland et al., 2008; Khoshbavar-Rostami *et al.,* 2012; Wang *et al.,* 2012) have focused on monitoring these compounds and a little information is available on specific aspects of PAHs mobility or degradation and the interactions between these processes in wetland soils and sediments. To obtain reliable estimates for the transport of PAHs from contaminated soils to surface and ground water, we must distinguish losses of PAHs due to transport. Little information is available about the transport of PAHs in Shadegan wetland. Hence, our objective was to determine the mobility of phenanthrene, anthracene, naphthalene and pyrene in the soil samples taken from Shadegan wetland under saturated conditions.

MATERIALS AND METHODS
Soil Sampling

Two composite soil samples (0-30 cm depth) including 4 sub-samples were collected from Shadegan wetland (30°00`- 31°00` N and 48°20`- 49°20` E) in Khuzestan, Iran. Mean annual rainfall, evaporation, and mean temperature in this region are 224.7 mm, 2685 mm, and 24.9 °C, respectively. High clay content, massive structure soils, with low permeability, poor internal drainage and high electrical conductivity (EC) are typical features of soils in this wetland (Soleimani *et al.,* 2009). Selected physical and chemical properties of soil including pH and EC (in saturated paste extract), cation exchange capacity, texture and the amounts of organic materials and calcium carbonate (Table 1) were determined using standard laboratory methods (ISRIC, 1986).

Table 1. Selected physical and chemical properties of soil samples

Properties	pH	ECe (dS m-1)	OM† (g kg-1)	Clay (g kg-1)	Silt (g kg-1)	Sand (g kg-1)	CEC‡ (cmol (+) kg-1)	CaCO3 (g kg-1)
Value	7.8	89	8.7	490	360	150	9	125

†Organic Matter   ‡Cation Exchange Capacity

Column Study

Air dried soil was crushed and passed through a 4 mm sieve, and used in the column experiments. Polyvinyl chloride (PVC) pipes (20-cm height and 6-cm inside diameter) were packed to a bulk density of 1.30 g cm^{-3}. Pore volume for each column was calculated from the difference in the mass of soil at saturation and that at oven-dry. One day before chemical application, columns were pretreated with 500 mL of distilled water supplemented with CaCl$_2$ and HgCl$_2$ (0.1 M) to prevent the dispersion of soil aggregate and biodegradation (Bayard *et al.*, 2000).

Prior to the leaching process, the top 0.5 cm of each column was covered with a fine quartz sand to (i) minimize disturbance of soil, (ii) provide an even distribution of leaching water, and (iii) minimize volatilization of PAHs. To determine the extent of PAHs leaching, the top 5 cm soil of the columns was contaminated by anthracene, pyrene, phenanthrene, and naphthalene at concentrations of 75 and 150 mg kg^{-1}. Briefly, the PAHs stock solutions (dissolved in acetone) were thoroughly mixed with the top 5 cm soil in dark conditions to avoid photodegradation of added PAHs from soil surface. The soil was regularly turned over to allow acetone to escape prior to beginning the leaching process every 12 h and replicated 4 times. A control treatment was also used without adding any PAHs. All treatments were replicated six times. One day after PAH application, columns were leached with 1000 mL of distilled water, at a rate of 100 mL h^{-1} and water was let to drain. The application rate allowed a water head of approximately 1 cm on soil surface throughout the leaching experiment. The leachate fractions were collected at approximately one pore volume from the saturated soil columns. A total of 10 pore volume water was leached through each soil column. The leachate from each pore volume was collected for PAH determination. After the leaching experiments, soil cores were removed from the PVC pipes and divided into four increment depths (0-5, 5-10, 10-15, and 15-20 cm). Soil and leachate samples taken from soil columns were kept in a refrigerator at 4 °C until analysis.

Chemicals and Analyses

Anthracene, pyrene, phenanthrene, and naphthalene were obtained from Merk (Darmatadt, Germany). All PAH analyses were carried out using a high performance liquid chromatograph (HPLC, Younglin model AS3000) equipped with a fluorescence detector on a C$_{18}$ Chromosorb, 250 × 4mm ID column..HPLC has been used to identify PAHs in soil samples and river sediments, and good sample recoveries (Black, 1982; Anderson, 1983). HPLC, with either fluorescence or UV detection, is an US-EPA recommended method for the analysis of both solid and liquid hazardous waste.

The PAHs were extracted from soil using ultrasonic method (US-EPA procedure No. 3035B). A twenty gram soil sample was poured in a 250 ml beaker and 150 ml of dichloromethane was added to the beaker. The content of beaker was sonicated for 10 minutes and then vacuum filtered. The extracted solvent was collected in a 500 ml flask and the remaining soil on the filtration paper (Whatman Grade No. 42) was sonicated and vacuum filtered with another 150 ml of n-hexane. This 300 ml of extracted solvents were mixed and concentrated with Kuderna-Danish apparatus.

To calculate the mass balance, concentrations of PAHs in soil columns after and before leaching were considered

as residual and initial concentration, respectively. The amount of PAHs which were volatilized was calculated from the difference between initial concentration and residual and leachate concentrations.

RESULTS AND DISCUSSION

The amount of PAHs (i.e. naphthalene, phenanthrene, pyrene and anthracene) in soil columns after leaching processes are shown in Figure 1. The background amount of PAHs in the soil was very low (Table 2). However, adding PAHs to the soil significantly increased the amount of these chemicals in the soil. The amount of PAHs was uniformly increased from the top to the lower layers of soil column, except for the 0-5 cm top layer, mostly due to evaporation from the soil surface. In the case of anthracene due to low solubility and low volatilization (Table 3), the changes of concentration in the whole of soil column was uniform.
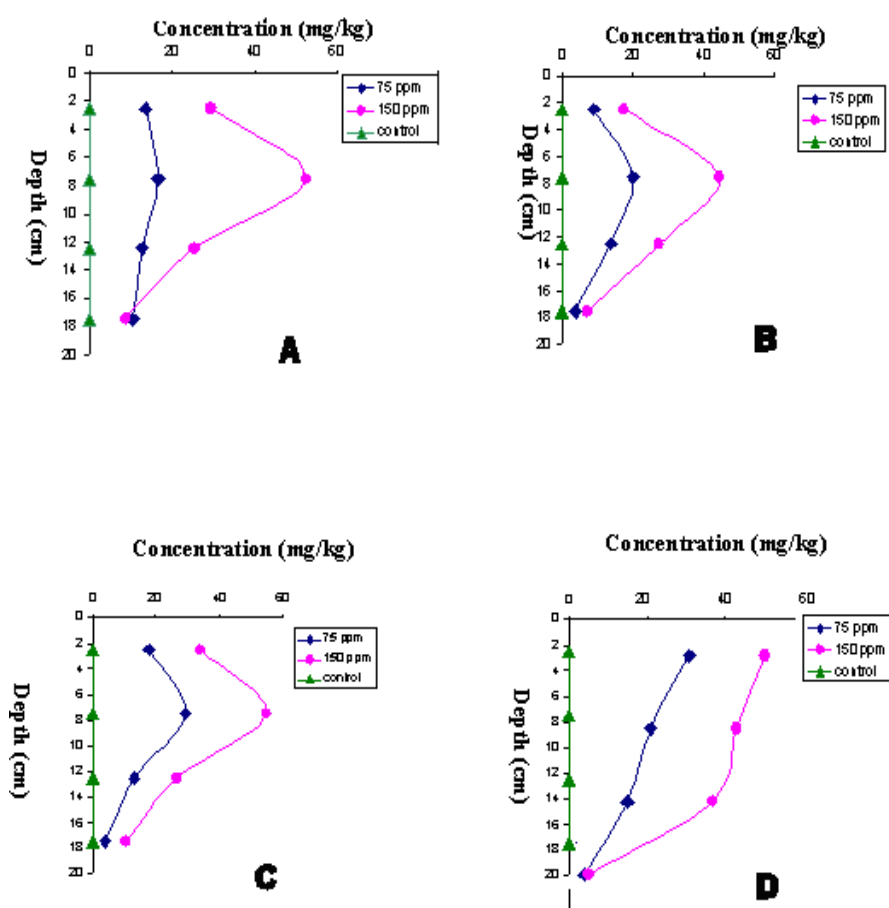

Fig. 1. Changes in concentration of phenanthrene(A), naphthalene(B), pyrene(C) and anthracene(D) in soil columns after the leaching experiment

At the lower layer of all columns, the concentration of all selected PAHs was less than 10 mg kg^{-1}. Park *et al.*, (1990) reported that volatilization of PAHs was negligible, except for naphthalene and substituted naphthalene. Two parameters affect PAH movement in the soil profile, inherent properties of PAH compounds and soil physical characteristics. The solubility of PAH compounds decreased with increasing the number of rings, while their hydrophobicity increased (Table 3).

Besides, soil properties such as organic matter can affect the fate and transport of PAHs (Raber *et al.*, 1998). Soil structure may be changed due to organic amendments and affects macropore flow promoted by colloidal particles and dissolved organic matter (Jarvis, 2007). In the long term experiments organic matter can affect biodegradation rates and sorption constants of mobile organic pollutants (Jarvis, 2007).

Table 2. Background concentration (µg kg^{-1}) of some PAHs in a soil sample from Shadegan wetland.

Naphthalene	Anthracene	Florene	Pyrene	Benzoanthracene	Chrysene	Floranthene	Phenenthrene
1027	56	108	92	52	56	53	157

Naphthalene showed the highest mobility in soil. Water solubility of naphthalene and the low molecular weight of this compound (Table 3) could increase its movement under saturated conditions in soil profile considering that water solubility is an important factor influencing PAH mobility in soil.

Breakthrough curves were obtained by plotting the normalized effluent concentrations C/C_0 where C is the outlet concentration and C_0 is the influent concentration as a function of pore volume. Trends of BTCs shape were similar at both PAH tested concentrations (Figs. 2 and 3) with the following trend: naphthalene>phenanthrene>anthracene> pyrene. PAHs mobility in the soil column showed a definite correlation with the number of their rings and molecular weight which affected their water solubility.

Table 3. Some characteristics of the PAHs (Binet et al., 2000; Wunch and Feibelman, 1997)

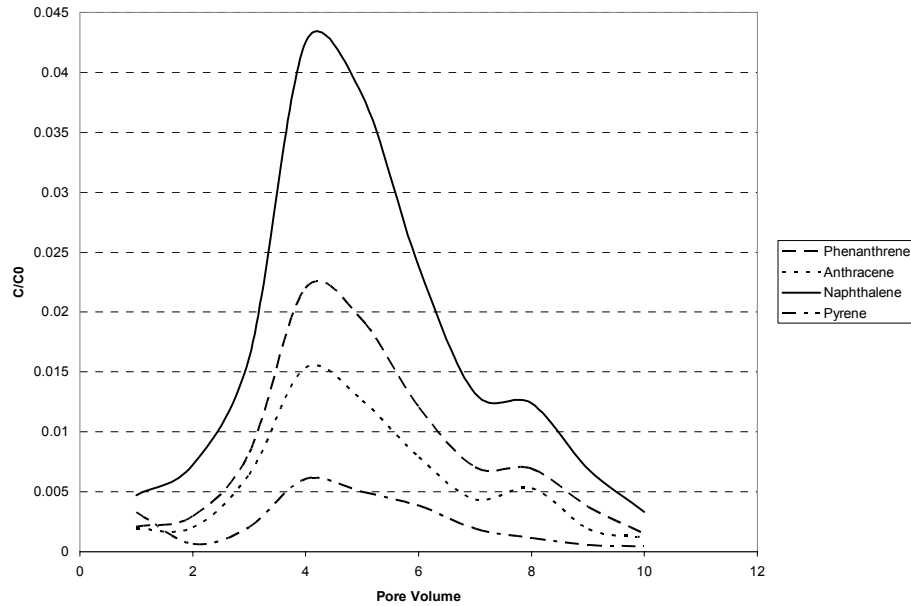| Compounds	Number of rings	Molecular weight (g)	Aqueous solubility (mg L^{-1})	Hydrophobicity (log K$_{ow}$)
Naphthalene	2	128	31.7	3.37
Phenanthrene	3	178	1.6	4.46
Anthracene	3	178	0.075	4.45
Pyrene	4	202	0.135	5.18
Chrysene	4	228	0.006	5.61
Dibenzo(a,h)anthracene	5	278	0.0005	5.97
Benzo(g,h,i)perylene	6	276	0.00026	7.23

Fig. 2. Breakthrough Curve (BTC) for treatments having 75 mg of each PAHs per kg soil at zero time
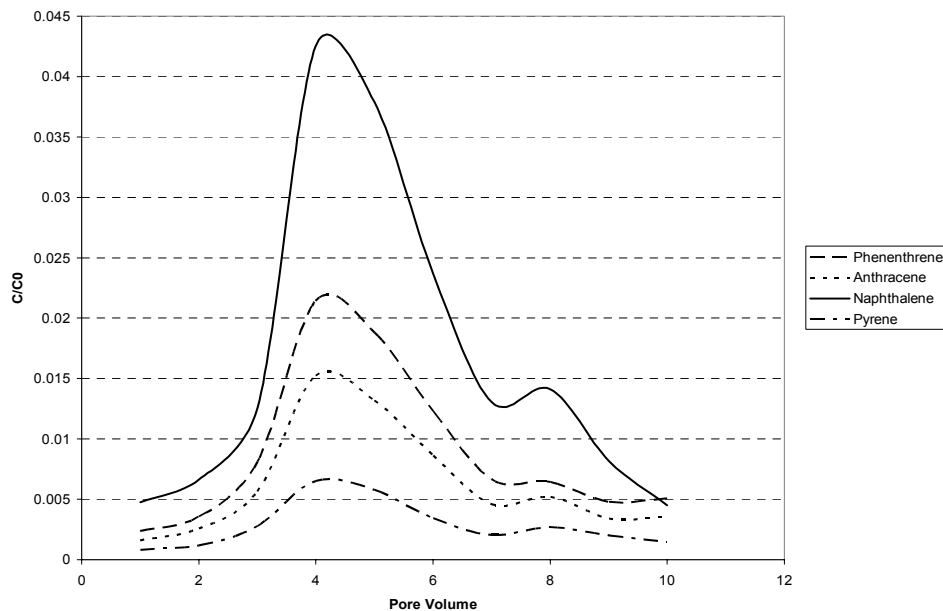

Fig 3. Breakthrough Curve (BTC) for treatments having 150 mg of each PAHs per kg soil at zero time

Mass Balance Analysis

Mass balance analyses of the five PAHs were investigated for better understanding of their fate in soil. More than 50 percent of all PAHs remained in soil. The data related to PAHs mass balance for both treatments are shown in Tables 4 and 5. Leaching of PAHs increased with enhancing their solubility in water. Greater water solubility of naphthalene compared to other PAHs might enhance its movement in soil profile probably contaminating water resources. The high percentage of naphthalene was volatilized from soil as compared to other PAHs in both treatments. More than 15% of the total added naphthrene, phenanthrene and pyrene were volatilized

from the soil, while this was only 5.8 to 8.6% for anthracene (Tables 4 and 5). Thus it may be concluded that anthracene is more recalcitrant than the other PAHs in soil media. This is due to low mobility of this compound in the water (Table 3).

Table 4. Mass balance of PAHs in soil. The amount of each PAH has been 75 mg kg^{-1} at zero time

PAHs	Leached (%)	Volatilized (%)	Residual in soil (%)
Naphthalene	24±2.4†	19. 1+2.2	56.9±5.6
Phenanthrene	12.4±1. 6	21.9±3.0	65.7±4.6
Anthracene	8.57±0.	5.8±.07	85.7±6.2
Pyrene	3.7±0. 5	17.3±1.5	78.9±4.4

† mean±standard deviation

Table 5. Mass balance of PAHs in soil. The amount of each PAH has been 150 mg kg-1 at zero time.

PAHs	Leached (%)	Volatilized (%)	Residual in soil (%)
Naphthalene	23.8±3.2†	17.2±2.4	59.1±4.9
Phenanthrene	12.8±1.9	16.3±2.1	71.0±6.3
Anthracene	9.1±2.1	8.6±1.1	82.3±5.6
Pyrene	4.2±1.0	18.3±3.5	77.5±3.2

† mean±standard deviation

CONCLUSION

This study showed that PAH compounds (naphthalene, phenanthrene, pyrene and anthracene) had the transport potential in the soil profile under saturated conditions and this is a risk for ecosystems specially wetlands. Naphthalene had a higher potential for leaching and it is expected that it can migrate through soil to pollute water resources and potentially enter the food chain. Anthracene could be more recalcitrant in soil than naphthalene, phenanthrene and pyrene. Meanwhile, the environmental risk of all selected PAHs together with non-selected ones should be considered. It is recommended that ongoing researches focus on new methods to stabilize and/or remove PAHs in the soils of Shadegan wetland considering biological approaches such as phytoremediation and bioremediation.

ACKNOWLEDGMENT

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حرکت و انتقال هیدروکربن‌های آروماتیک چندحلقه‌ای در یک خاک آهکی تالابی

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چکیده

آگاهی از حرکت و تجزیه‌ی هیدروکربن‌های آروماتیک چندحلقه‌ای در ارزیابی آلودگی این ترکیبات در منابع آب و خاک مهم است. در این مطالعه حرکت ترکیبات نفتالن، آنتراسن، پیرن و فنانترن در یک خاک آهکی آلوده نمونه برداری شده از تالاب شادگان (خوزستان، ایران) در یک ستون خاک در شرایط آزمایشگاهی بررسی شد. هیدروکربن‌های آروماتیک چندحلقه‌ای در غلظتهای 75 و 150 میلی‌گرم بر کیلوگرم به 5 سانتی متر بالایی خاک اضافه شد و محلول خروجی از ستون خاک حاصل از 10 حجم منفذی (پور والیوم) جمع آوری گردید. غلظت هیدروکربن‌های آروماتیک چندحلقه‌ای زهابها و نیز 3 عمق خاک (0 تا 5، 5 تا 10 و 10 تا 15 سانتی‌متر) با استفاده از دستگاه کروماتوگراف مایع فشار بالا اندازه‌گیری شد. نتایج نشان داد که از بین هیدروکربن‌های آروماتیک چندحلقه‌ای مورد مطالعه، نفتالن متحرک‌ترین ترکیب بود و تفاوت حرکت هیدروکربن‌های آروماتیک چندحلقه‌ای مورد مطالعه در خاک به ساختار و حلالیت آنها مرتبط بود. بررسی توازن جرم نشان داد که از بین هیدروکربن‌های آروماتیک چندحلقه‌ای مورد مطالعه آنتراسن و نفتالن به ترتیب بیشترین و کمترین ترکیب مقاوم از نظر تجزیه بودند. با توجه به حلالیت بالای نفتالن در آب، پتانسیل آلودگی منابع آب سطحی و زیرزمینی با آن بالاست که این موضوع باید مورد توجه جدی قرار گیرد.

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