



# Polycyclic aromatic hydrocarbons in surface waters and riverine sediments of the Hooghly and Brahmaputra Rivers in the Eastern and Northeastern India

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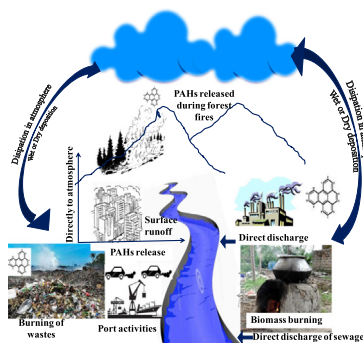
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## HIGHLIGHTS

- Mean concentration of total toxic PAHs were nearly ten folds higher in the surface riverine sediment of Hooghly compared with Brahmaputra.
- Industrial effluents and port activities were the suspected sources of heavier PAHs in Hooghly riverine sediment.
- Lighter PAHs in surface water are posing risk to fish species in both therivers.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Sixteen priority polycyclic aromatic hydrocarbons (PAHs) regulated by the United States Environmental Protection Agency (USEPA) were analyzed in surface waters and riverine sediments of Brahmaputra and Hooghly Rivers, along urban-suburban-rural transects.  $\sum_{16}$  PAHs concentrations were higher in Hooghly riverine sediment (HRS) (Avg, 445 ng g<sup>-1</sup>) than Brahmaputra riverine sediment (BRS) (Avg, 169 ng g<sup>-1</sup>) dominated by 4-ring PAHs. In contrast, PAHs concentrations in surface water of Brahmaputra River (BRW) (Avg, 4.04 µg L<sup>-1</sup>) were comparable with Hooghly River (HRW) (Avg, 4.8 µg L<sup>-1</sup>), with dominance by 3-ring PAHs. Toxic PAHs (BaA, Chr, BbF, BkF, BaP, InP and DBA) were dominant in sub-urban transect of HRS (Avg, 387 ng g<sup>-1</sup>) and BRS (Avg, 14 ng g<sup>-1</sup>). Diagnostic ratios, principal component analysis (PCA) and ring wise composition suggested combustion as the main PAHs source in these riverine belts. In BRS, higher PAHs in suburban and rural transects were attributed to incomplete combustion of fossil fuel and biomass burning. In HRS, >85% of high molecular weight PAHs were found in the industrial areas of the suburban transect possibly associated with the discharge of industrial effluents. Harbor and port activities were other major contributors of HMW-PAHs in Hooghly riverine system. Carcinogenic potency estimated in terms of toxic equivalent (TEQ) was several folds higher in HRS (Avg, 106 ng TEQ g<sup>-1</sup>) compared with BRS (Avg, 2.5 ng TEQ g<sup>-1</sup>). Mostly low molecular weight PAHs are likely posing a risk to fishes in both the rivers. Risk on edible fish species may be a matter of concern considering the regular consumption of fishes in this region.

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## 1. Introduction

Perennial rivers are major sources of freshwater for aquatic ecosystems worldwide. Pollutants from a variety of sources (i.e. run-off, wet and dry deposition) end up in the riverine environment and many eventually accumulate in sediments. The process of re-suspension and circulation in the aquatic environment forms a continuous pollution source worldwide (Kannan et al., 2005; Zeng and Venkatesan, 1999). Sediments have direct or indirect influence on the aquatic ecosystem providing nutrient sources to several aquatic plants, spawning and benthic organisms (Kemker, 2014; Kolok et al., 2014).

Because of toxicity, the United States Environmental Protection Agency (USEPA) has listed sixteen polycyclic aromatic hydrocarbons (PAHs) as priority pollutants. The toxicity of PAHs depends on different factors, including the species, exposure route and PAH's molecular structure (Douben, 2003).

The high molecular weight PAHs (HMW-PAHs) ( $\geq 4$  fused aromatic rings) are less water soluble, less volatile and more lipophilic than low molecular weight PAHs (LMW-PAHs) ( $\leq 3$  fused aromatic rings) and have probable carcinogenic effects on human beings (Hale et al., 2012; Khairy et al., 2009; Tavakoly Sany et al., 2014). PAHs contamination has gained much attention in developing countries like India, due to increasing emission sources associated with rapid urbanization and industrial growth (Lang et al., 2008; Zhang and Tao, 2009).

In 2004 maximum global atmospheric emission of 16 USEPA listed PAHs was from China ( $114 \text{ Gg y}^{-1}$ ) and India ( $90 \text{ Gg y}^{-1}$ ) followed by the United States ( $32 \text{ Gg y}^{-1}$ ) (Zhang and Tao, 2009). Major Indian cities like Kolkata and Guwahati are located along the River Hooghly in the lower stretch of Ganges and Brahmaputra, respectively. The bank of the River Hooghly houses  $>700$  industries, including iron and steel, heavy engineering, rail equipment, transport equipment, chemicals, oil refining, agro-processing, textiles, paper, fertilizers and diverse consumer goods. Industrial and automobile emissions contribute nearly 90% of the air pollution in Kolkata (Majumdar et al., 2008). The catchment of the combined Hooghly and Brahmaputra riverine sediments is about  $1.656 \times 10^6 \text{ km}^2$  (Wasson, 2003). HRS contributes up to 4% of the annual sediment discharge in the River Ganges (Mukhopadhyay et al., 2006). Past studies reported elevated PAHs contamination in sediment of Hooghly estuary (Guzzella et al., 2005) and Sunderban delta (Domínguez et al., 2010). Several legacy persistent organic pollutants (POPs) have been detected in various environmental matrices in and around the lower stretches of Hooghly River. Residues of organochlorine pesticides (OCPs) were observed in the ambient atmosphere (Chakraborty et al., 2010), surface soil (Chakraborty et al., 2015), surface riverine sediment (Guzzella et al., 2005) and surface water (Chakraborty et al., 2016a) in the Hooghly River. Similarly, industrial chemicals like polychlorinated biphenyls (PCBs) have been reported in air (Chakraborty et al., 2013), surface soil (Chakraborty et al., 2016b), sediment (Guzzella et al., 2005) and surface water (Chakraborty et al., 2016a) of the Hooghly River.

In northeastern states of India along the Brahmaputra River, PAHs and legacy POPs (OCPs and PCBs) were observed in the surface water of the Brahmaputra River (Hussain et al., 2011) (Chakraborty et al., 2014) (Chakraborty et al., 2016a), in sediment (Hussain et al., 2015) and surface soil (Hussain and Hoque, 2015; Devi et al., 2015). Moreover, a few studies evidenced that Assam, which is one of the eight sister states in northeastern India along the Brahmaputra valley, contributed nearly one-fourth of the total onshore crude oil and natural gas production in India (NG, 2014–15) and thus represents a potential PAHs contamination source.

In this study, we have therefore investigated the occurrence and level of PAHs in the surface waters and riverine sediment collected from the Hooghly and Brahmaputra Rivers, along urban-suburban-rural transects. Further, molecular diagnostic ratios and principal component analysis (PCA) were used to estimate distribution and source apportionment in order to better assess the potential ecological risks due to PAHs contamination.

## 2. Materials and methods

### 2.1. Study areas and sampling

The Hooghly River is approximately 260 km long and forms the lower stretch of the Ganges and drains to the Bay of Bengal. It passes through a heavily industrialized area with more than half of West Bengal's population. The Brahmaputra River originates from Angsi glacier, located on the northern side of the Himalayas in Tibet. It enters India through Arunachal Pradesh and flows through the valley of Assam to join Bangladesh as the Jamuna River. Details of sampling sites and details of the sampling locations are included in the Supporting information (SI Tables S1, S2, S3 and S4). Briefly, the drainage areas range from agricultural to heavily industrial and many areas of the rivers are used for fishing and bathing. Altogether 50 surface water samples and 53 surface riverine sediment samples were collected from the two rivers. For water samples, five sub samples (each 1 L) have been collected from each site. The sub samples were mixed in a steel pail, 1 L sample was taken as the representative sample for each site. For sediment samples, five sub samples were collected over an area of  $1 \text{ m}^2$  in a zig-zag manner to form a representative sample for each site. Sediment samples were stored in polyethylene zipped bags. After collection, all samples were stored in a cooler box, transported to the laboratory. Water samples were immediately extracted and sediment samples were stored frozen at  $-20 \text{ }^\circ\text{C}$  until further analysis.

### 2.2. Extraction and column clean-up

All sediment samples were dried and about 20 g of each sediment sample was Soxhlet extracted for 18 h with 150 mL of dichloromethane. Details of water samples extraction procedure have been given elsewhere (Chakraborty et al., 2016a). Briefly, 1 L of unfiltered water samples were subjected to liquid-liquid extraction using dichloromethane (DCM) thrice (50, 25, 25 mL). Each sample was mixed with 10 gram sodium chloride and shaken vigorously for 3–5 min for 3 times. The organic phase was collected, combined and then dried by passing through 3 cm of sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) placed on glass wool, and then concentrated using rotary evaporator. Deuterium labeled PAHs (Naphthalene-D8, Acenaphthene-D10, Phenanthrene-D10, Chrysene-D12, and Perylene-D12) were used as surrogate standards. A silica gel column clean-up (30 cm long and 3 cm diameter glass column packed with 5 g anhydrous sodium sulphate, on the top, and 10 g activated silica gel) was performed on sample extracts. PAHs were eluted using 100 mL of n-hexane and dichloromethane mixture (n-hexane/DCM, 1:1, V/V). Finally, the eluent solvent was concentrated to 20  $\mu\text{L}$  under a gentle stream of pure nitrogen. Hexamethylbenzene was added as an internal standard before analysis and used to quantify all analytes.

### 2.3. Analysis

Naphthalene (Nap), Acenaphthylene (Acy), Acenaphthene (Ace), Fluorene (Fl), Phenanthrene (Phe), Anthracene (Ant), Fluoranthene (Flu), Pyrene (Pyr), Benzo(a)anthracene (BaA), Chrysene (Chr), Benzo(b)fluoranthene (BbF), Benzo(k)fluoranthene (BkF), Benzo(a)pyrene (BaP), Dibenzo[a,h]anthracene (DBA), Benzo[g,h,i]perylene (BgP) and Indeno[1,2,3-cd]pyrene (InP) were analyzed in extracts of surface water and sediment samples from Brahmaputra River and Hooghly River in an Agilent 7890B Gas Chromatograph coupled with 5977A Mass Spectrometry using a HP-5MS capillary column ( $30 \text{ m} \times 0.25 \text{ mm i.d.} \times 0.25 \text{ }\mu\text{m}$ ).  $1 \text{ }\mu\text{L}$  of each sample was injected in split-less mode. High-purity helium was used as carrier gas, with a flow rate of  $1.83 \text{ mL min}^{-1}$ . Temperatures of injector and transfer line were kept at  $290 \text{ }^\circ\text{C}$  and  $300 \text{ }^\circ\text{C}$  respectively. The initial oven temperature was set at  $60 \text{ }^\circ\text{C}$  for 1 min, increased to  $290 \text{ }^\circ\text{C}$  at a rate of  $4 \text{ }^\circ\text{C min}^{-1}$  and then hold for 20 mins.

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