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Polycyclic aromatic hydrocarbons (PAHs) in sediment environments in Vietnam: Analytical methods and contamination status

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ABSTRACT

Introduction: Polycyclic aromatic hydrocarbons (PAHs) are prevalent organic pollutants that garner attention due to their toxicity and carcinogenic properties. These hydrophobic compounds tend to accumulate in the organic components of sediments, posing significant ecological risks.

Methods: This study synthesizes analytical methodologies and assesses the contamination status of PAHs in Vietnamese sediments through a review of previous studies complemented by recent findings from our research group. Various extraction techniques, including Soxhlet extraction, pressurized liquid extraction, continuous solid-liquid extraction, and ultrasonic extraction, were employed using organic solvents of low to moderate polarity (e.g., hexane, acetone, toluene, dichloromethane). Purification was achieved via column chromatography using sorbents like silica gel, alumina, and Florisil, followed by gel permeation chromatography. Separation and analysis were primarily conducted through gas chromatography/mass spectrometry (GC/MS).

Results: PAHs were detected in all samples, with concentrations ranging from 33 to 6400 ng/g, indicating widespread contamination. The sources of PAHs in Vietnamese sediments appear to be predominantly urban and informal waste processing areas. The levels of sedimentary PAHs in Vietnam are on par with those reported in other Asian countries, with the majority of PAHs being traced back to pyrogenic sources, as opposed to petroleum products.

Conclusions: Our findings offer a detailed understanding of the distribution, patterns, and potential sources of PAHs in Vietnamese sediments. The notably higher concentrations of PAHs in urban and waste processing sites underscore the urgency of implementing effective management and remediation strategies. The study advocates for further monitoring of PAHs and related pollutants to fully elucidate their contamination scope, origins, and ecological consequences.

Key words: PAHs, sediment, GC/MS, Vietnam

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous organic pollutants that have garnered global concern due to their adverse effects on both environmental and human health [1]. Recognized for their carcinogenic and genotoxic potential, PAHs have been the subject of extensive research worldwide [2]. Interest in these compounds surged following the US Environmental Protection Agency's (US EPA) identification of 16 unsubstituted PAHs as priority pollutants in the 1970s [3]. Subsequently, several PAHs and their derivatives have been classified as probable or possible human carcinogens [3]. While a fraction of low-molecular-weight (LMW) PAHs can be traced back to the chemical industry or natural processes, the majority are inadvertently produced through thermal processes involving petroleum products [4]. Owing to their hydrophobic nature, PAHs tend to adsorb onto suspended particles and accumulate in sediments, making these environments crucial for assessing pollution levels and ecological risks associated with PAHs [1,5].

In Vietnam, the inaugural study on sedimentary PAHs conducted in Ho Chi Minh City in 1999 revealed elevated PAH concentrations in urban sediments [6]. Further investigations across the country have since highlighted traffic and waste processing as primary sources of these contaminants [6–11]. Despite these efforts, comprehensive and contemporary data on PAHs in Vietnamese sediments remain scarce.

Traditionally, PAH extraction from sediments has employed organic solvents of low to moderate polarity, such as hexane, acetone, toluene, and dichloromethane, alongside methods like Soxhlet extraction [6,12]. While effective, Soxhlet extraction is criticized for its lengthy duration and significant solvent usage [13]. Consequently, alternative techniques like accelerated solvent extraction (ASE) and

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ultrasound-assisted extraction (UAE) have gained favor for their efficiency and reduced solvent demands [14]. Post-extraction, sample purification typically involves column chromatography and gel permeation chromatography, avoiding harsh treatments that could degrade the PAHs [7–12,15].

Quantification of PAHs has evolved from high-performance liquid chromatography with a fluorescence detector (HPLC/FLD) to predominantly gas chromatography/mass spectrometry (GC/MS) due to the latter's superior separation, selectivity, and sensitivity [6–10,12]. This study aims to examine the concentrations of 16 US EPA-prioritized PAHs in surface sediment samples from the south central coast and Hanoi rivers. Utilizing ultrasonic extraction and chromatographic purification, we seek to delineate the contamination levels, accumulation patterns, and potential sources of PAHs in these regions. This investigation is further bolstered by a literature review, providing a holistic overview of PAH occurrences in Vietnamese sediments.

MATERIALS AND METHODS

Chemicals

A native standard mixture of PAHs (H-QME-01 Quebec PAH Mix; AccuStandard, USA) comprised naphthalene (Nap; 98.4%), acenaphthylene (Acy; 99.1%), acenaphthene (Ace; 100%), fluorene (Flu; 100%), phenanthrene (Phe; 100%), anthracene (Ant; 99.6%), fluoranthene (Flt; 98.6%), pyrene (Pyr; 98.1%), benz[a]anthracene (BaA; 98.9%), chrysene (Chr; 100%), benzo[b]fluoranthene (BbF; 99.7%), benzo[j]fluoranthene (BjF; 98.8%), benzo[k]fluoranthene (BkF; 99.9%), benzo[a]pyrene (BaP; 99.9%), dibenz[a,h]anthracene (DA; 99.9%), indeno[1,2,3-cd]pyrene (IP; 100%), and benzo[ghi]perylene (BP; 97.5%). Deuterated surrogate standards (ES-2044 PAH Surrogate Cocktail) including naphthalene-d8 (Nap-d8; 100%), acenaphthylene-d8 (Acy-d8; 100%), phenanthrene-d10 (Phe-d10; 99.1%), fluoranthene-d10 (Flt-d10; 98.6%), pyrene-d10 (Pyr-d10; 100%), benzo[a]pyrene-d10 (BaP-d10; 98.4%), and benzo[ghi]perylene-d12 (BP-d12; 99.4%), along with internal standards (Chrysene-d12, >98%), were acquired from Cambridge Isotope Laboratories, Inc., USA.

Solvents such as acetone (99.5%; Macron Fine Chemicals^{TM}, Germany), hexane (96%; Daejung, Korea), dichloromethane (99.5%; Daejung, Korea), and toluene (99.7%; J.T.Baker, USA) were used. Silica gel (60 N, 63–210 μm, Merck, USA) and Florisil (0.150–0.250 mm, Merck, USA) were activated at 130°C for 3 h and 12 h, respectively, prior to use. Anhydrous sodium sulfate (>99.5%, Merck, Denmark) was baked at 400°C for 2 h.

Sample Collection and Pre-treatment

Surface sediment samples were collected from the south central coast (n = 5, CC-1 to CC-5) in Binh Dinh Province and from urban rivers (n = 5, UR-1 to UR-5) in Hanoi. The coastal samples were affected by various activities, including shipping and tourism, while the Hanoi samples were influenced by residential, commercial, and traffic emissions. Sediments were collected using a Van Veen sampler, air-dried, ground, sieved through a 1-mm sieve, and stored at –20°C until analysis.

Chemical Analysis

Approximately 0.2 g of dry sediment was spiked with surrogate standards and extracted using a VCX 130 ultrasonic processor (Sonic & Materials, Inc.) with a mixture of acetone/hexane (1:1, v/v) and toluene. After centrifugation at 3500 rpm for 10 min, supernatants were collected, evaporated, and the residue was reconstituted in hexane for clean-up. A clean-up column was prepared with layers of sodium sulfate, silica gel, and Florisil, and eluted with an ethyl acetate/hexane (5:95, v/v) mixture. The final extract was spiked with an internal standard and reconstituted in ethyl acetate for GC/MS analysis.

PAHs were quantified using a GCMS-QP2010 Ultra system (Shimadzu, Japan) equipped with a DB-5ms column. The temperature program began at 110°C, with gradual increases to a final temperature of 310°C. Helium was used as the carrier gas. The MS detector operated in electron impact (EI) mode with selective ion monitoring (SIM) for quantification.

Quality Assurance and Quality Control (QA/QC)

Procedural blanks and spiked sediment matrices were analyzed to ensure the absence of PAH contamination and to verify recovery rates, which ranged from 70% to 110%. The method detection limits (MDLs) were established based on blank levels, ranging from 0.02 to 0.80 ng/g. The accuracy of the analytical procedure was confirmed against Standard Reference Material® 1944 from the National Institute of Standards and Technology (USA), with results showing good agreement with certified values (RSD < 25%).

RESULTS

Concentrations and Profiles of PAHs in Urban River Sediments

PAHs were ubiquitously detected across all urban sediment samples analyzed, underscoring the pervasive presence of these contaminants. In the Hanoi river sediments, concentrations of the 16 evaluated PAHs ranged from 53 to 1800 ng/g, with a median of 820 ng/g. Notably, PAH levels in the Red River sediment (UR-1) were substantially lower, at 53 ng/g, compared to 310–2000 ng/g found in other urban rivers (UR-2 to UR-5). The distribution of PAHs by their ring numbers showed a descending order of prevalence: 4-rings (31.7% ± 8.0%) were the most common, followed by 6-rings (26.2% ± 11%), 3-rings (18.3% ± 7.2%), 5-rings (17.6% ± 2.3%), and 2-rings (6.2% ± 1.8%). This pattern indicates a dominance of high-molecular-weight (HMW) PAHs (4–6 rings; 76% ± 9.0%) over low-molecular-weight (LMW) compounds (2–3 rings; 24% ± 8.8%), pointing to pyrogenic sources as the major contributors. Specifically, in the Red River sediments, phenanthrene (Phe), benzo[b/j]fluoranthene (Bb/jF), chrysene (Chr), pyrene (Pyr), fluoranthene (Flt), and benzo[ghi]perylene (BP) were identified as predominant compounds. In contrast, inner-city river sediments exhibited a significant dominance of BP (24% ± 8.9%), along with notable amounts of Phe, Flt, Pyr, Chr, and Bb/jF, suggesting nearby sources of gasoline vehicle emissions, such as road surface runoff and atmospheric deposition.

Concentrations and Profiles of PAHs in Coastal Sediments

Similar to urban rivers, PAHs were detected in all coastal sediment samples, with total 16 PAH concentrations ranging from 38 to 200 ng/g (median 71 ng/g). The highest concentrations (120–200 ng/g) were observed in samples from regions impacted by aquaculture, fishery, and transportation activities, particularly around economic zones and bridges (CC-3 and CC-5). These elevated levels imply industrial, fishing, and transportation activities as significant PAH sources. Conversely, sediments from CC-1, CC-2, and CC-4 showed lower PAH concentrations (38–71 ng/g), comparable to those in the Red River sample. HMW PAHs (63% to 88%, average 79%) predominated over LMW PAHs (12% to 37%, average 21%), suggesting high-temperature processes as the primary PAH source rather than direct petroleum inputs. The estuary sample (CC-2) had a higher proportion of LMW PAHs (37%), indicating a diverse contamination profile. The sample under a bridge (CC-5) displayed a significant presence of HMW PAHs (88%), pointing to traffic emissions as a key pollution source. Overall, the 4-ring PAHs were most prevalent (41% ± 6%), followed by 5-rings (24% ± 3%), 3-rings (18% ± 6%), 6-rings (13% ± 6%), and 2-rings (4% ± 2%). Predominant compounds in the south central coast sediments included pyrene (Pyr), fluoranthene (Flt), phenanthrene (Phe), and benzo[a]pyrene (BaP).

DISCUSSION

Comparison of PAH Concentrations in Vietnamese Sediments

The detected concentrations of 16 PAHs in river sediments from Hanoi ranged between 53 to 1800 ng/g, showcasing higher levels compared to those in the south central coast, which varied from 38 to 200 ng/g. The urban river sediments displayed PAH concentrations akin to those found in sediments from an e-waste recycling site in Hung Yen Province, northern Vietnam, which spanned from 340 to 2100 ng/g with a median of 650[8]. Metropolitan areas within Vietnam, including the Kim Nguu River[7], lakes in Hanoi[16], and the Saigon River[16], exhibited significantly elevated PAH levels in contrast to lower concentrations observed in rural or suburban locations like the Ky Cung River in Lang Son Province[7] and the water bodies in Hue City[16]. This variation suggests that PAH pollution in sediments is closely linked to the level of urbanization[6,7,16]. Sediments from Bangkok's canals reported PAH levels that were comparable or even exceeded those found in Hanoi's urban rivers, with a mean concentration of 2290 ng/g and a range from 512 to 8400 ng/g[17]. In comparison, PAH levels in the Red River were notably lower than those in the Menam Chao Phraya River in Thailand[17], the Sungai Mahakam River in Indonesia[18], and rivers in Malaysia[19], indicating that PAH contamination in Vietnamese surface sediments varies from low to moderate relative to other Southeast Asian countries[20].

Despite the abundance of studies on riverine and urban sediments, research on marine sediments in Vietnam remains limited. Thuy et al. (2021) documented PAH concentrations in the coastal sediments of Can Gio wetland ranging from 0.73 to 518 ng/g, with an average of 62 ng/g[11]. Interestingly, sediment samples collected during the 2017 wet season exhibited higher PAH levels compared to those from the 2018 dry season[11]. The PAH concentrations in our coastal sediment study (38–200 ng/g) align with those reported for the Can Gio coast[11], yet remain lower than figures from other international locations, such as Haizhou Bay, China[21], and South Korea[22]. This underscores the necessity for further investigations across various Vietnamese coastal sites.

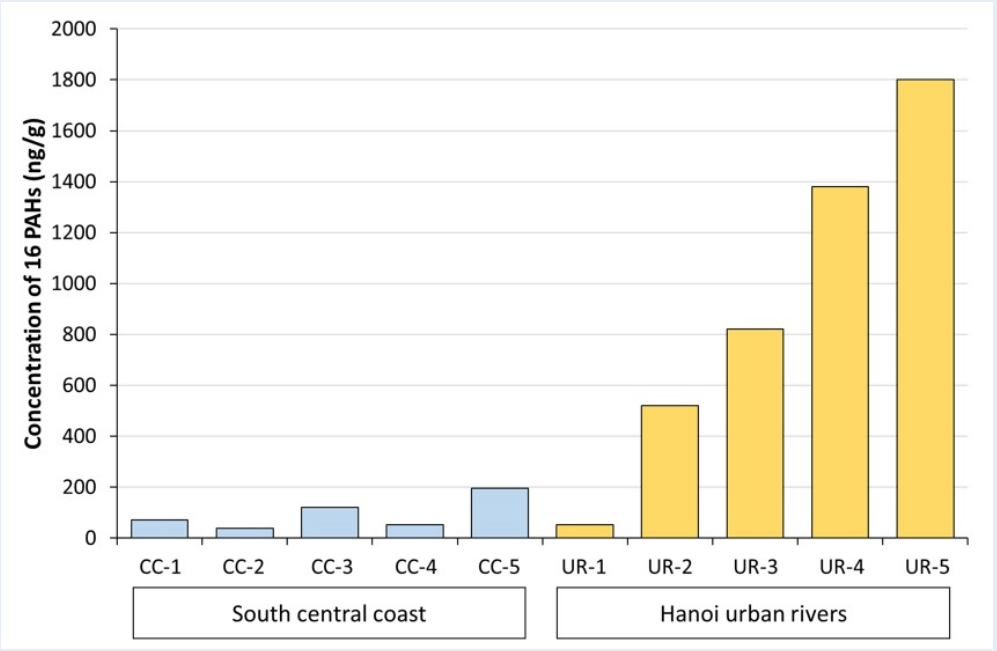

Figure 1: Concentrations of total 16 PAHs in sediment samples of this study

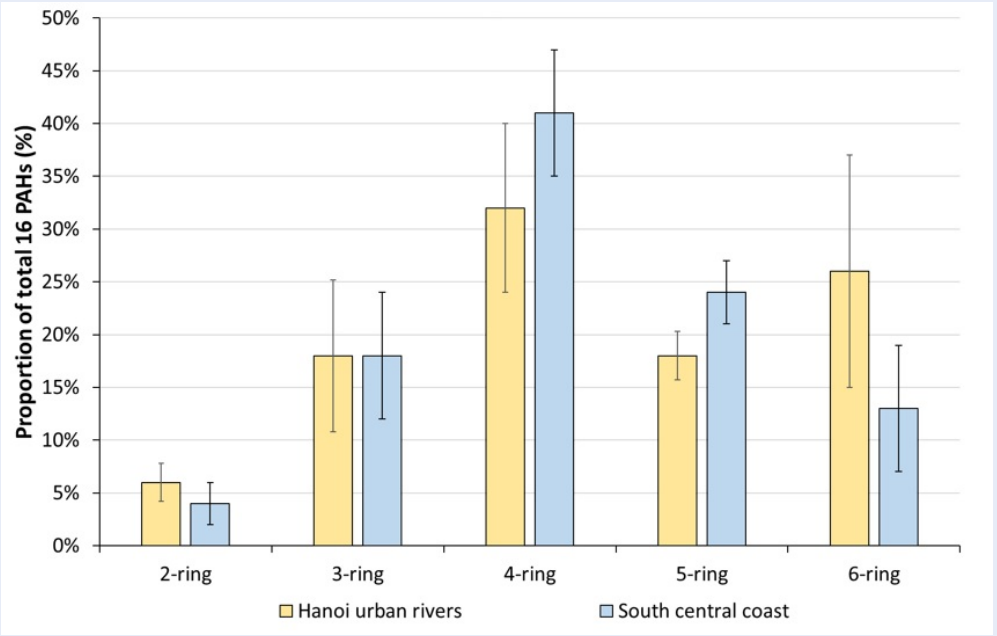

Figure 2: Compositions of PAHs in sediment samples by ring numbers

Profiles and Source Estimation of PAHs in Sediments

Analysis of both urban and coastal sediment samples indicated a dominance of high-molecular-weight (HMW) PAHs (4-rings or more), pointing towards pyrogenic sources—principally vehicle emissions—alongside surface water runoff and atmospheric deposition. The significant representation of 6-ring PAHs, particularly benzo[ghi]perylene (BP), in Hanoi river sediments, as opposed to those from the central coast and the Red River, underscores the influence of local urban sources and the constrained mobility of these compounds. Diagnostic ratios such as BaA/(BaA + Chr) [23], BaP/BP [24], and Flt/(Flt + Pyr) [25] validate the inference that PAHs predominantly stem from gasoline combustion and traffic pollution. The Flt/(Flt+Pyr) ratios, falling between 0.41 and 0.50, alongside BaP/BP ratios exceeding 0.9 in sediment samples, elucidate the pyrogenic nature of PAH emissions [26]. Notably, direct indicators of petroleum-related contamination were not distinctly evident, suggesting the necessity for future studies to enhance source identification methods for PAHs in sediment matrices.

CONCLUSIONS

This investigation delineated the concentrations, profiles, and potential sources of polycyclic aromatic hydrocarbons (PAHs) in surface sediments from the urban environment of Hanoi and the south central coast of Vietnam. Utilizing ultrasonic extraction followed by column chromatography cleanup for the sample preparation, this study highlighted the efficiency, speed, simplicity, cost-effectiveness, and minimal cross-contamination risk of the ultrasonic extraction method. Future research should aim at expanding the range of targeted PAH compounds, optimizing low-cost and high-throughput sample preparation techniques, and implementing robust quality assurance and control (QA/QC) measures for the accurate determination of PAHs in Vietnamese sediments.

Our findings contribute valuable insights into the contamination levels of PAHs across Vietnam, revealing notably higher concentrations in urban and informal waste processing areas compared to those in suburban, rural, or coastal regions. The detected PAH levels in urban Vietnamese sediments are within the range of or below those observed in other Southeast Asian countries. Additionally, our study suggests that PAH contamination in certain coastal areas of Vietnam is relatively lower than in other regions, such as China and South Korea. The PAH distribution patterns predominantly point to pyrogenic sources, including fossil fuel combustion and vehicular emissions, rather than direct inputs from petroleum products.

The elevated PAH concentrations identified in urban settings and waste processing sites underscore the urgent need for effective management and mitigation strategies. To adequately address the environmental impact of PAHs and their derivatives, further comprehensive research is essential. Such studies should aim to build a complete database regarding the pollution status, sources, and environmental risks posed by PAHs in Vietnam, facilitating informed decision-making and policy development for pollution control.

COMPETING INTERESTS

The authors declare no competing interests.

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ABBREVIATIONS

AOAC: Association of Official Analytical Chemists
GC/MS: Gas chromatography/mass spectrometry
HMW: High-molecular-weight
LMW: Low-molecular-weight
MDL: Method detection limit
PAHs: Polycyclic aromatic hydrocarbons
SIM: Selected ion monitoring

AUTHOR'S CONTRIBUTIONS

Tu Binh Minh and Shin Takahashi conceptualized the research methodology. Hoang Quoc Anh supervised the study. Nguyen Duc Hieu wrote the original draft. Tran Manh Tri, Dang Minh Huong Giang, Hoang Bich Ngoc, Vu Van Tu, Nguyen Thi Hong Yen conducted experiments and data processing. All authors discussed and edited the manuscript.

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