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Effects of Land use Pattern on the Distribution of Phthalate Esters in Sediments of U-Tapao River Basin, southern Thailand

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ABSTRACT

Phthalate esters (PAEs), a group of anthropogenic organic pollutants have generated serious concern globally, due to their ubiquitous nature and hazardous effects in aquatic ecosystem. This study investigated the influence of land uses on the distribution of 6 PAEs species in sediment of U-Tapao River basin, southern Thailand. 6 PAEs species including DEHP, DBP, BBP, DiNP, DnOP and DIDP, sediments samples were collected from urban (UR), agricultural (AG), and natural field (NF) zones of the watershed and were analyzed by GC-MS. Of the 6 PAE congeners only DEHP, DBP and DINP were detected. The mean values of Σ3PAEs in the sediment samples collected from UR, AG and NF were 1993.33, 706.67 and 298.24 ng/g, respectively. The average concentration of PAEs in urban zones, was almost three times higher than those in Agricultural fields and 7 times higher than natural fields. Land use pattern significantly influenced the PAEs concentrations in the river basin sediments. The concentrations of PAEs indicated strong and significant positive correlations with sediment organic matter (OM), and a strong and significant negative relationship with sediment pH. The main sources of PAEs in the sediments were associated with industrial, residential, commercial and agricultural activities. The estimated total PAEs inventory indicated 308.44 tons in period of 5 years (UR, 205.06; AG, 72.69; NR, 30.68 tons), suggesting that the contaminated sediments in the study area may be a potential source of PAEs in Songkla lake, the largest lake in Thailand. Thus, the necessity of including PAEs in the legislations and regulatory documents addressing the discharging of wastes containing hazardous pollutants into aquatic environments. Strategies for controlling PAEs pollution in the U-Tapao River basin was Proposed.

Keyword: *Phthalate esters, land use pattern, sediment and inventory*

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INTRODUCTION

Over the last few decades the ubiquitous occurrence and potential risks of phthalate acid esters (PAEs) in aquatic environment, especially river systems have become a global issue of increasing environmental and public health concern (Hu et al., 2013; Net et al., 2015). PAEs are mainly used as plasticizers in the production of plastic, rubber, polyvinyl chloride (PVC) and other polyethylene products to improve their flexibility, workability and durability (Wormuth 2007; Liu et al., 2014; Zhang et al., 2018). In addition, PAEs are also used to improve the quality of a large diversity of industrial and consumers products (Huang et al., 2008; Net et al., 2015). PAEs account for 70% of 8.4 million tons of plasticizers that were produced and consumed worldwide in 2014 and have been forecasted to increase to 10.3 million ton in future with PAEs contributing 65% (Zhang et al., 2018). PAEs are easily released into the environment, because they are not chemically or covalently bounded to the plastics matrices, thus, they can easily leach, migrate or evaporate into aquatic environments via industrial and municipal wastewater effluent, agricultural discharge, sewage sludge, storm water runoffs, and atmospheric deposition (Okamoto et al., 2011; He et al., 2013; Lee et al., 2019). Thus, PAEs have become so widespread in the aquatic

ecosystem and have been found in river water, particulate matter, sediments and aquatic biotas (Sun et al., 2013 Niu et al., 2014; Lee et al., 2019; Kingsley and Witthayawirasak, 2020).

PAEs are characteristic endocrine disrupting compounds (EDCs) which can cause serious adverse reproductive effects, thyroid hormone disrupting effects, insulin-like growth factor disruptions and children developmental-disrupting effects (Boas et al., 2010). The European Union and USEPA backlisted some PAEs congeners as priority pollutants of water because they get easily attached to suspended particles and sediments as a result of their hydrophobic characteristics and can therefore easily enter the food web (Sun et al., 2013;Chen et al., 2017).

River sediments are significant sources of distribution and bioaccumulation of PAEs in aquatic ecosystems and have been widely utilized as an environmental indicator for the evaluation of PAEs pollution in the aquatic environment (Gobas et al., 2003). In the aquatic environment, sediments act as sinks and deposition sources for PAEs because they have low values of aqueous solubility, volatility, and melting point (Mitsunobu and Takahashi 2006). Besides, PAEs tend to associate with particulate materials during the process of deposition in rivers and coastal ecosystems because of their hydrophobic characteristic and high octanol/water partition coefficients (Staplcs et al. 1997; Net et al., 2015). However, sediments of smaller particle size and a larger surface volume with high organic carbon content have greater potential to accumulate significant concentrations of PAEs (Rice et al. 1993; Mi et al., 2005). Moreover, river sediments, therefore, are significant sources for the evaluation of anthropogenic pollution in aquatic environment as they have a long residence time for the organic contaminants like PAEs (Heyden and New 2004). Furthermore, sediments are significant vectors in transporting organic chemicals including PAEs from river ecosystem to the ocean (Guan et al., 2009; Mi et al., 2019; Hu et al., 2020). Clear determination of the amount of PAEs transported by riverine sediments can shed light into the cross-boundary transfer and help in the estimation of PAEs inventory in aquatic ecosystem (Mi et al., 2019; Hu et al., 2020).

Previous studies have reported correlation between PAEs distribution in sediments and land use pattern, and anthropogenic activities (Sun et al., 2013; Wang et al., 2014; Zheng et al., 2014; Arfeania et al., 2019). The U-Tapao River, a major watershed in Southern Thailand is a river system surrounded by different land use patterns including industrial, residential, commercials, agricultural fields and natural fields (Gywali et al., 2012). The river flows through the center of many agricultural fields, industries, commercials and residentials areas. Thus, it receives both treated and untreated wastewater effluents from municipal and industrial activities as well as agricultural discharges. Besides, sediments can act as a sink and vector for many pollutants to accumulate and discharge into Songkla Lake, the largest lake in Thailand that discharges into gulf of Thailand. Previous studies assessed the impact of land use type on the water quality parameters of the basin. These studies reported that different land use type pose varying levels of water quality pollutions, ranging from low to high level of water quality degradation in the basin (Gyawali et al., 2012; Maprasit et al., 2016). Nevertheless, no study has evaluated the influence of land use pattern on the distribution of chemical pollutants in the river basin.

Therefore, in this present study, gas chromatography/mass spectrometer (GC-MS) analytical technique was applied i} to evaluate the levels of PAEs in sediment samples of U-Tapao River, ii) to assess the influences of land use type including, urban, agricultural and natural zones on the distribution of PAEs in sediments samples, iii) to evaluate the potential sedimentary inventory of PAEs in the river basin, that may be transferred to the lake. Six PAEs, viz. DBP, BBP, DEHP, DnOP, DINP and DIDP have been targeted for this study because they are classified as endocrine disruptors and major additives in preparation of consumer items and plastic products. Besides, there are scanty information on the major alternatives of DEHP such as DINP and DIDP in sediments. Vital baseline data brought out through this study, on the pollution status and influence of land use type on the distribution of PAEs in sediments of U-Tapao River basin are essential for the protection, sustainable and strategic management of the aquatic environment, and preserving the health of aquatic ecosystem and humans.

MATERIALS AND METHODS

Materials and chemicals

Solvents used for this work included HPLC grades of Hexane (99.9%), Methanol (99.9%), Acetone (99.9%) and Dichloromethane purchased from Sigma-Aldrich. Ultrapure water 18.2MΩ-cm, Milipore from Waters, USA. Phthalate Standards included, Di-n-butyl phthalate (DBP), Benzyl Butyl phthalate (BBP), Di-2-ethylhexy phthalate (DEHP), Di-n-octyl Phthalate (DnOP), Di-isononyl phthalate (DINP), Diisodecyl phthalate (DIDP) were purchased from AccuStandard, USA. Solid phase extraction cartridge (Florisil 1g 6cc, Chron and Sep). Other materials included GF/F glass filters Sampling bottles, 250ml, 500ml, 1000ml Amber, Fishers Scientific, Winchester bottles, GC vials, Amber, 2ml, Superlco, Sigma-Aldric, Micropipettes. Instruments used were, Aligent GC-MS (6890N/5973), Ultrasonicator, Analytical balance, Solid phase extraction Vacuum Manifold, pH meter, EC meter. Qualification and quantification analysis were carried out with internal standards and surrogate standards

Study area and sample collection

U-Tapao watershed lies within Latitude 6°27′–7°10′ N and Longitude 100°11′–100°39′ E, with total area of 2400 km^2 from Thai-Malaysian border to Songkla Lake, the largest lake in Thailand (Figure 1). This is one the most important watershed of Southern Thailand. The U-Tapao watershed,

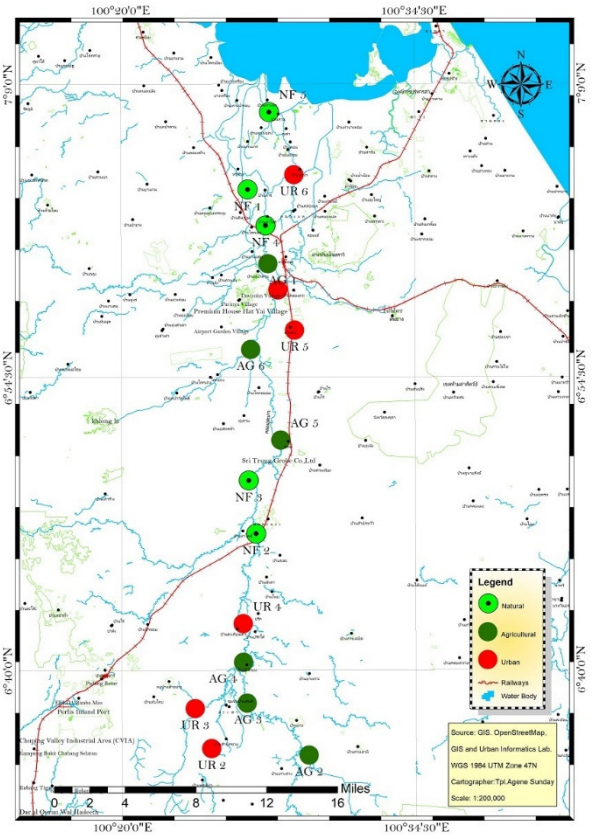

Figure 1 Sampling sites in U-Tapao River

flows through the center of many agricultural fields, industries and urbanized areas and discharges into Songkla Lake, which is connected to Gulf of Thailand. Thus, the occurrence and accumulation of pollutants in the aquatic environment and movement of river sediments into Songkla Lake and Gulf of Thailand have caused the biodiversity cycle of the region to be constantly under threat (Gyawali et al., 2012; Chuvanich et al., 2017; Kingsley and Witthayawirask, 2020). The increasing pollution and degradation of this major watershed, due to rapid urbanization and industrialization has generated serious concerns to the both the government and the general public in Thailand. The sampling sites are given in Fig. 1. A total of 17 (UR, n=6; AG.n=6; NF, n=5) sediment samples were collected from the river basin areas with three major different land uses pattern (urban, agricultural and natural zones). A stainless-steel grab sediment sampler was used to collect sediment samples from the basin. The collected samples were promptly placed inside glass amber containers which had been pre-washed with n-hexane, and transferred to laboratory alongside dry ice at –4 °C. In the laboratory, the samples were freeze-dried for 72 h, then passed through a 0.5mm sieve, and kept at –20 °C for further analyses.

Physiochemical properties

The pH of the sediment samples was determined using a pH-meter (PHS-3C pH meter model, China). The electrical conductivity (EC) of the samples was measured by multiparameter U-50 (HORIBA, Germany). To measure the total phosphorus content in the sediment samples, UV colorimetry method was used. A CHN analyzer (Perkin-Elmer, model 2400 CHN) was used to measure the organic carbon content (Dargahi et al., 2015). The particle size analyzer was employed to analyze the distribution of the sediment sample particles with a capability of analyzing sizes between 0.02 and 2000 µm (Sochan et al., 2012). Further, the percentage of three fractions of particles including clay, silt, and sand was also determined.

Sample preparation

Freeze-dried sediment samples were grounded and homogenized using a mortar and pestle and then sieved through a stainless-steel sieve (60-mesh) and stored in brown glass bottles at –20 °C until extraction. Weighted sediment samples 5.0 g were placed into glass centrifuge bottles, mixed with 10 mL acetone/hexane (1:1 v/v) and extracted ultrasonically for 30 min. The procedure was replicated twice, and each extract was subsequently filtered into a round bottom flask. Then, the filtrates were concentrated to 1–2 mL with a rotary evaporator, solvent exchanged with n-hexane and then cleaned by solid phase extraction cartridge of florisil. The SPE cartridge, florisil (1g 6cc, chrom and Sep), was successively activated by 15 mL hexane and then with 15 mL acetone/n-hexane (1:1 v/v), and the eluents were discarded. Then, the concentrated extract was transferred to the florisil solid phase extraction cartridge and eluted with 10 mL acetone/n-hexane (1:4 v/v), and the resulting eluent was collected. The fraction containing the PAEs was concentrated to 2 mL; next, the isotope surrogate standard was added, and the fraction was adjusted to a constant volume of 1 mL and passed through a 0.25µ membrane filter into a brown glass sample bottle for sample injection prior to instrumental analysis.

GC- MS analysis

All samples were analyzed using gas chromatography-mass spectrometry (Agilent 6890 GC-5973 MSD) operating in electron impact and selective ion monitoring (SIM) mode using a capillary column for chromatographic separation. Helium with a very high purity degree (99.9999%) was used with a constant flow rate of 1.2 ml/min as carrier gas. The thermal program used was as follows: the column oven on 50 °C for 1 min and then with 15 °C/min, it reached 200 °C and was kept at this temperature for 1 min. Next, it was brought to 280 °C at 80 °C/ min, and was kept at this temperature for 3 min. The samples were injected to the system with injector temperature at 250 °C in non-pulse and splitless states. The transfer line temperature was set at 280 °C, and the post-run temperature was adjusted as 285 °C for 2 min. PAEs concentration in the sediments was calculated based on dry weight.

Quality control and quality assurance

Firstly, to prevent any kind of potential contamination, no plastic equipment was used during the sampling and sample processing. Instead, glass materials were immersed in K2CrO4/H2SO4 solution, and washed with NaOH, deionized water, and acetone. Parallel to the extraction of environmental samples, the control samples were also prepared with a similar process without sediment sample. A stock standard solution of 100 mg PAEs L^{-1} was prepared by diluting the original standard solution with acetone/n-hexane. The standard samples (with concentration levels of 0.5, 1, 5, 10, 20, 30, 40, 50 mg L^{-1}) for quality control were prepared by adding the standard solution to acetone/n-hexane with 1:1 (v/v) ratio. After that, the calibration curve was drawn for each PAE and calibration solutions were replaced every two weeks. In addition, spiked samples were also prepared by adding certain amounts of the standard solution to the samples. The devices were calibrated on a daily basis by calibration standards. A method blank, a spiked blank, a matrix spike, and replication of samples for each batch were performed on the samples. The mean recovery, limit of detection (LOD) and limit of quantification (LOQ) values for each of the PAE compounds were also determined by spiked sample method prior to the analysis of samples. The data obtained are presented in (Table 1). Finally, trace amounts were observed for some compounds in the method blank samples, with these values suitably subtracted from the values read in the samples.

Table 1: The mean recovery, LOD and LOQ values for individual PAE congener

S/NO	PAEs Congeners	Mean of Recovery (%)	LOD (ng/g)	LOQ (ng/g)
1	DBP	89.7	0.39	1.98
2	DEHP	93.2	0.48	2.99
3	DINP	98.6	0.87	2.78
4	BBP	91.0	0.16	1.86
5	DnOP	92.4	0.44	2,43
6	DIDP	96.3	1.08	2.86

Data analysis

The inventory of sedimentary transfer of PAEs from U-Tapao River to Songkla Lake was calculated using equations 1 (Zhao et al., 2011):

$$Inventory = C \times p \times D \times a \quad (1)$$

where C (ng/g) is the mean concentration of PAEs in the sediment
p (g/cm3) is the dry density of the sediment
A (cm2) represents the area of the watershed
D (cm/y) is the sedimentation rate
a (y) is the number of years considered in the inventory

A statistical analysis method was employed for the data analysis (e.g., mean, median, and maximum and minimum values), including the spearman correlation matrix (SCM), principal component analysis (PCA) and hierarchical cluster analysis (HCA) were performed using SPSS 20.0 for window (IBM SPSS Inc. Chicago, IL). The level of significance was set at $p \leq 0.05$. Figures, maps were plotted as well as calculations using SPSS and Microsoft Excel, version 2010.

RESULT AND DISCUSSION

PAE concentrations and congener profiles in sediments of the rivers

The individual concentration of the six most common PAEs in sediments samples collected from U-Tapao River basin are indicated in Table 2. These PAEs include Di-n-butyl phthalate (DBP), Benzyl Butyl phthalate (BBP), Di-2-ethylhexy phthalate (DEHP), Di-n-octyl Phthalate (DnOP), Di-iso=nonyl phthalate (DINP), Diisodecyl phthalate (DIDP). As seen in (Table 2), only three PAEs congeners (DBP, DEHP and DINP) exhibited values of concentration above detection limit (LOD). The Σ3PAEs concentration in sediment samples ranged from 170 to 3300 ng/g with a mean value of 1993 ng/g. The spatial distribution of measured PAEs in sediments samples collected from the investigated watershed is shown in Figure 2. The highest concentration of ∑3PAEs occurred mainly in the urban zones, following the order of UR 6>UR1>UR2>UR5>UR3>UR4. Almost all of these sampling sites were located in urban areas which include industrial, residential and commercial zones. Large amounts of PAEs are released from industrial and municipal wastewater effluent into the investigated watershed. In addition, PAEs may be washed out by rainwater, carried in drainage water, leached from dumpsites and indiscriminately dumped plastics to the watershed and finally deposited in the sediments. Relatively high Σ3PAEs concentrations were found at sampling sites located in the agricultural areas in the increasing order of AG 3>AG2>AG6>AG1>AG4>AG5. These sampling sites were situated adjacent to rubber plantation, paddy fields, vegetable farms, shrimp farms and livestock breeding farms. The major sources of PAEs here are likely fertilizers, plastic mulching films, agrochemicals, animal feeds and feces. The low concentrations were found in the natural field. The natural field were located near forest and reserved areas. The major source of PAEs in this region is atmospheric deposition.

The mean concentrations of total ∑3PAEs in UR, AG, and NF areas were 1993.33, 706.67, 298.33 ng/g, respectively. The mean concentration of individuals PAEs across the land use type for DEHP were UR (958.33), AG (376.67) and NF (128.33). The concentrations of DINP in UR, AG, and NF zones were 726.67 ± 0.67, 186.67 ± 0.65, and 128.33 ± 1.36 ng/g, respectively. For DBP, the values were 308.33 ± 0.52, 143.33± 0.84 and 41.67 ± ng/g, respectively. The detection frequencies of these compounds in the sampling sites were 95.12%, 87.8%, and 95.12%, respectively. The highest concentration of PAEs in sediment was observed at UR6 (∑3PAEs), which

Table 2: Individual concentration of PAEs in sediments of U-Tapao river basin (ng/g).

PAEs	UR			AG			NF		
	MIN	MAX	Mean	MIN	MAX	Mean	MIN	MAX	Mean
DBP	210	540	308.33	80	170	143.33	30	60	41.67
DEHP	690	1740	958.33	280	490	376.67	80	180	128.33
DINP	590	1020	726.67	100	380	186.67	60	130	128.33
BBP	ND	ND	ND	ND	ND	ND	ND	ND	ND
DnOP	ND	ND	ND	ND	ND	ND	ND	ND	ND
DIDP	ND	ND	ND	ND	ND	ND	ND	ND	ND
∑3PAEs	1490	3300	1993.33	460	1040	706.67	170	370	298.24

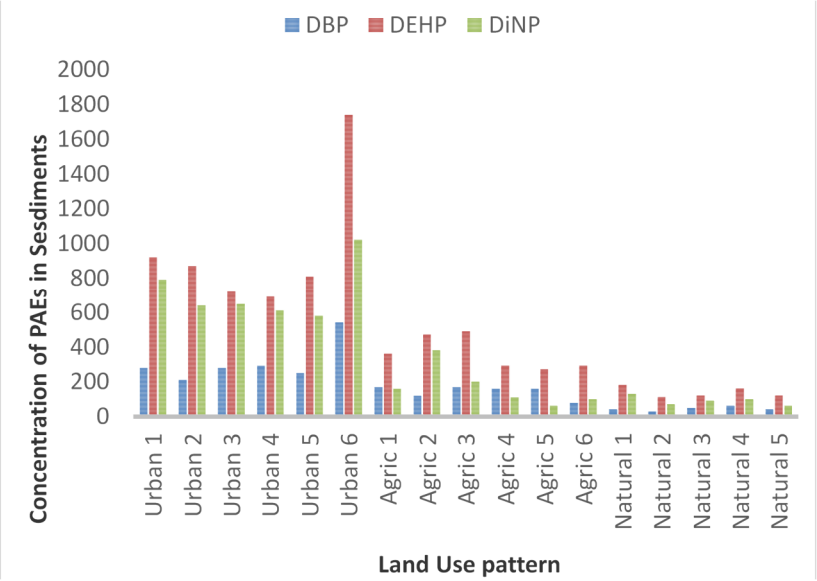

Figure 2: Spatial distribution of 3 PAEs congeners in sediments from U-Tapao River basin.

is located beside residential and industrial areas in Hat Yai city, situated downstream of the U-Tapao river. The second highest PAEs concentration was found at UR2 (∑3PAEs) in Prik districts, located beside the midstream of the river. The third highest PAEs level was detected at site UR 5 (∑3PAEs) situated besides the municipal provincial water supply reservoir, located at Hat Yai, the largest drinking water reservoir in Songkla Province. The lowest PAEs concentration was detected at NF (∑3PAEs 50ng/g) located near natural field in Sadao district which is located upstream of the river basin.

Researchers have suggested that the analysis of individual PAEs and congener profile (composition) can provide information on pollution source and environmental biogeochemical characteristics of PAEs in multimedia environments (Sun et al., 2013; Wang et al., 2014). The frequency of detection (FOD) of individual PAEs congeners indicated the order of DEHP > DINP > DBP. However, the relative percentage contributions (RPC) of the total level of ∑3PAE congeners, revealed that, DEHP had the highest relative contribution in the UR, AG and NF. The relative percentage contribution of ∑3PAE congener detected in the sediment samples analyzed in this present work were in the order of DEHP>DINP>DBP. This is consistent with previous studies that reported the dominant of DEHP congeners in sediment samples of river systems (Srivastava et al., 2010; Adeniyi et al., 2011; He et al., 2013; Sirivithayapakorn et al., 2014; Selvaraj et al., 2014; Kingsley and Witthayawirasak, 2020).

Considering individual PAEs congener, DEHP is the most prevalent, accounting for more than 50% of the total industrial PAEs output in many countries such as Canada, Sweden, China, Malaysia, Thailand, South Africa and Nigeria (Tan 1995; Mackintosh et al., 2006; Cousin et al., 2007; Fatoki et al., 2008; Adeniyi et al., 2011; Wang et al., 2013; Kingsley and Witthayawirask, 2020). Thus, the level of DEHP may be used as an indicator to predict the concentration of PAEs pollutants in aquatic sediments (Wang et al., 2014; Arfeania et al., 2019). This study indicated that DEHP was preponderant PAEs in investigated river sediments which may be attributed to its high-volume usage, strong sorption and low degradation rate (Sun et al., 2013; Zheng et al., 2014; Wang et al., 2014).

Effects of land use pattern on physiochemical properties of sediments

Table 2 and Figure 2 show the physiochemical properties of sediment samples collected from U-Tapao river basin, in Urban (UR), agricultural (AG) and Natural fields' zones. As indicated, the clay content ranged from 21.19 to64.74% in UR, 13.86 to 61.13% in the AG and 7.42 to 28.44% in the NF. The silt content lied within the range of 19.42 to

Table 3 Physiochemical parameters of sediments from U-Tapao river basin.

Parameter	UR			AG			NF		
	MIN	**MAX**	**Mean**	**MIN**	**MAX**	**Mean**	**MIN**	**MAX**	**Mean**
pH	5.25	6.75	5.78	6.78	7.45	5.79	7.34	8.04	7.73
EC (µS/cm)	166.64	524.12	173.30	245.32	412.62	169.10	14.82	28.48	22.21
OC (%)	0.58	3.86	1.48	0.65	3.72	1.63	0.23	1.32	0.73
WC (%)	0.76	3.74	2.23	0.39	2.76	1.51	0.16	1.07	0.76
OM (%)	1.78	4.68	2.01	1.11	3.91	2.46	0.42	1.68	1.03
TP (mg/kg)	329.66	2500.67	988.53	130.48	1200.12	607.35	66.77	94.12	79.16
Clay (%)	21.19	64.74	41.58	13.86	61.13	37.08	7.42	28.44	9.69
Silt (%)	19.42	33.43	25.69	19.97	42.97	31.30	21.63	46.42	35.54
Sand (%)	46.35	69.39	32.74	11.91	66.17	31.62	26.27	39.09	31.59

33.43 % in UR, 19.97 to 42.97% in AG and 21.63 to 46.42% in NF. The sand content lied within the range of 46.35 to 69.39% in UR, 11.91 to 66.17% in AG and 26.27 to 39.09% in NF. Our result is consistent with (Sirinawin and Somponchaiyakul, 2005). This finding suggests that the dominant texture of the sediments in U-tapao river basin includes clay and sand across all the three zones. The high clay texture in the watershed was in the order of UR>AR>NF. Clays, because of their finer-grained characteristic can trap and store more pollutants. This may have influenced the high concentration of PAEs in the UR region as compared to the levels reported for AR and NF zones in the basin.

The study results revealed that pH values of sediments in the three areas including UR, AG and NF, lied within the ranges of 5.25 to 8.04, means of 5.78, 5.79 and 7.73 respectively (Table 3). The pH of the samples in the UR and AG areas lies within the category of mild acidic to neutral, while the NF zone, lied within mild alkaline to neutral. The results of the present study is in agreement with previous studies that reported the pH of sediments in urbanized and Agricultural areas as slightly acidic (Adeniyi et al., 2011; Arfaenia et al., 2019). The lower pH in sediments samples collected from the UR and AG areas may be ascribed to the discharge of wastewaters containing large amounts of organic compounds. The degradation of these large quantity of organic compounds causes the formation of humic acid, which decreases the pH value in the riverine sediment (Nobi et al., 2010; Abdoallahzadeh et al., 2016). In addition, human activities including discharge of acid-containing industrial and municipal wastewater, use of fertilizer and pesticides in the agricultural lands, breeding of fish and shrimp in the studied riverine can acidify the sediments (Jafari et al., 2018). Furthermore, the acidic pH may lead to reduction of anoxic degradation rate and promote OM and TP accumulation, indicating high pollution of the river basin.

The level of electrical conductivity (EC) in the sediment samples lied within the ranges of 166.64 to 524.12 (mean 173.30), 245.32 to 412.62 (mean 169.10), 4.82-28.48 (mean 22.21) µS/cm in UR, AG and NF respectively. EC is the capacity of aquatic media including sediment to conduct an electric current, it approximates the overall concentration of total dissolved inorganic ions in sediments (Talling, 2009). The value of EC is commonly used to assess the potential pollutants pathways. Regions of lower EC values generally indicate coarser-grained sediment texture, which are more permeable. These more permeable regions allow pollutants to migrate in the subsurface. Whereas, the finer-grained sediments including clay can trap and store more pollutants. In this study, the highest minimum and maximum EC values were observed at the UR zones. Similarly, the percentage of clay were detected in UR zones. Thus, suggesting that the increasing EC values in freshwater ecosystem typically indicate increasing clay content. Notwithstanding, it is worthy of note that this is not always true, as clay mineralogy and the presence of brines can influence the EC value. Furthermore, EC may be employed as surrogate parameter for total phosphorus as well as total nitrogen concentration (Miguntanna et al., 2010; Yamazaki et al., 2014). Eventually, the Total phosphorus (TP) concentration lied within the ranges 329.66-2500.67 (mean, 988.53), 130.48-1200.12, (mean 607.35) and 66.77-94.12 (mean, 79.16) mg/kg in UR, AG and NF respectively. Both EC and TP levels are in the order of UR>AG>NF. TP value in this study is higher than those reported in previous studies (Sun et al., 2013; Arfaenia et al., 2019), and comparable with those observed by (Wang et al., 2014). The highest TP accumulation was detected in UR6, which is located within the industrialized area of U-Tapao river basins, indicating high pollution of the affected areas of the river, due to municipal and industrial wastewater discharges. This is consistent with the observation in sediments from Yangtze River basin, China (Wang et al., 2014). The concentration of water content in the sediment samples lied within the ranges of 0.76-3.74 (mean 2.23) in UR, 0.39-2.76 (mean, 1.51) in AG and 0.16-1.07 (mean, 0.76) in NF.

Organic carbon (OC) concentrations lied within the ranges of 0.58-3.86 (mean=1.48), 0.65-3.72 (mean, 1.63), 0.23-1.32 (mean, 0.73) % in UR, AG and NF respectively. As can be seen, the concentration of OC is in the order of UR >AG>NF. Given the high value of organic carbon portioning (Koc), the high concentration of the high molecular weight such as DEHP and DINP in sediments in the urban areas, may be attributed to the lipophilic properties, which make them absorb well on carbon-rich surfaces. In addition, their hydrophobic characteristics makes it easy to dissolve in macromolecular organic compound, such as humic compounds and finally deposit to sediments (Adeogun et al., 2015; Zhang et al., 2018). The concentration of organic matter (OM) in the sediments samples ranges from 1.78 to 4.68% (mean of 2.01) in UR, 1.11% to 3.91% (mean of 2.46) in AG and 0.42-1.68, (1.03) %. The values of OM content in sediments in this study are

lower than those reported for Yangtze River basin situated in southern Jiangsu (Wang et al., 2014) and comparable to those reported for the Pearl River delta in South China (Liu et al., 2014). The OM is the major factors that regulates the sorption of organic chemical compounds in aquatic environment (Mai et al., 2005). Among the urban stations, the maximum OC and OM level were observed in UR 6. This station is located in Hat Yai city which is the most populated and industrial city in Songkla province, which receives large amounts of both industrial and domestic wastewater rich in organic compounds. These findings show that human activities are the major source of carbon in the sediments of the basin and can bring nearly a significant effect on carbon cycle processes in the river basin sediments (Li et al., 2014).

Effects of land use pattern on PAEs level in sediments

The individual concentration of the most commonly used ∑3PAEs compounds detected in sediments collected from the UR, AG and NF zones of U-Tapao river basin are shown in (Table 2), whereas, the spatial distribution of 3PAEs is presented in Figure 2. As indicated, 3PAEs congeners including DEHP, DBP and DiNP were detected in all the collected sediment samples, suggesting that these species of PAEs are among the ubiquitous contaminants in the studied river sediments. The Σ3PAEs levels lied within the ranges of 1490-3300ng/g, with mean of 1993.33ng/g in urban (UR) sites, 460-1040, with mean of 706.67 in agricultural (AG) areas, and 170-370 with mean of 298.33 in natural field (NF). It is obvious, that the mean concentration in the urban sediment samples, was almost 3 times higher than that of the agricultural sites and 7 times higher than the natural field areas. This is consistent with previous studies that reported that PAEs concentration in the order of UR>AG>NF (Wang et al., 2014; Zhen et al., 2014; Arfaenia et al., 2019). The result in this present study strongly reveals that pollution level of PAEs in aquatic environment are markedly influenced by anthropogenic sources and land use pattern. The elevated levels of PAEs in investigated river basin sediments from the urbanized areas were mainly contributed by the intensity of industrial and commercial activities and population density (Liu et al., 2010; Gywali et al., 2012). In addition, the high PAEs values in UR sites may be attributed to frequent discharging of industrial and municipal wastewater from industries such as glove manufacturing industries, rubber factories, plastic industries, electronic/electric equipment factories, chemical factories, industries that produce plastic pipes, packaging industries, para wood factories, plastic recycling industries, furniture making industries, food factories and agrochemical industries (Worawit et al., 2008; Nobi et al., 2010; Gywali et a., 2012; Zheng et al., 2014). Moreover, PAEs may be released from municipal solid waste sites that are located near the river via runoff, especially during the incidence of floods (Pongpiachan et al., 2013). This is consistent with previous studies that indicated that PAEs in runoffs water from waste dumpsites may be released into receiving rivers and deposited to sediments (Arukwe et al., 2012; Adeogun et al., 2015). Furthermore, PAEs may be released from the indiscriminate plastic waste dumping on the river and then deposited in the sediments (Adeogun et al., 2015; Zhang et al., 2018; Kingsley and Witthayawirasak, 2020). PAEs may be washed out by rain, carried in the drainage water to the river and finally deposited in the sediments, due to high rate of absorption and low transport potential in sediment (Sirivithayapakorn and Thuviang, 2010; Arukwe et al., 2012; Net et al., 2015).

However, relatively high ∑3PAEs concentration was detected in sediments collected from agricultural zones. All these sites are located adjacent to rubber plantation, shrimp farms, rice paddy fields, vegetable farmsteads and livestock breading areas. The main sources of PAEs here are likely pesticides, fertilizers, aquatic feeds, animal fecal matter, agrochemicals, seafood and vegetable packaging, atmospheric depositions and long-range transport (Zheng et al., 2014; Wang et al., 2014; Niu et al., 2014; Net et al., 2015). Moreover, the water used for irrigation (irrigation canals) are supplied by some tributaries around the river basin, which are contaminated by industrial and municipal discharge (Céspedes et al., 2006; Wang et al., 2014). Irrigation by contaminated wastewater even after the treatment may significantly increase the levels of toxic contaminants such as metals, PAEs, and polychlorinated biphenyls in agricultural lands (Hongjun et al., 2013). According to Niu et al. (2014), the use of plastic films, irrigation by wastewater, and chemical fertilizers are major sources of PAEs in agricultural lands and may be released into the river and deposited in the sediments because of their hydrophobic and lipophilic characteristics. Moreover, relatively low ∑3PAEs were measured in samples collected from natural field sites. The level of PAEs detected in this zone may be attributed to atmospheric deposition of the identified and quantified 3PAEs compounds in the river basin. The spatial distribution of PAEs in sediment samples was influenced by land use pattern. PAEs levels in urban samples collected from urbanized areas which include residential, commercial and industrial regions were significantly higher than those from agricultural and natural fields (Figure 2).

Relationships between different congeners of PAEs and physicochemical parameters of sediments

It is generally stated that the environmental fate and transport of organic pollutants in sediments are controlled by the physiochemical characteristics of sediments particularly OM, OC, sediments texture (clay, silt and sand), WC and pH (Wang et al., 2014; Arfaenia et al., 2019). Pearson correlation (PC) was carried out to obtain a full picture of the relationships between the measured parameters (physiochemical properties and PAEs). Hierarchal cluster analysis (HCA) was used to discriminate the different sampling sites. The PC results are presented in (Table 4), while HCA discrimination of sites is presented in (Figure 3). The PC results indicated that physiochemical parameters, particularly, OM, WC, TP and grain sizes (particularly clay and sand) had relationship with the

Table 4: Pearson correlation matrix between the measured physiochemical parameters of sediments and PAEs in U-Tapao River.

	DBP	DEHP	DINP	PH	EC	OC	OM	TP	CLAY	SILT	SAND	WC
DBP	1											
DEHP	.958**	1										
DINP	.900**	.948**	1									
PH	-.868**	-.890**	-.849**	1								
EC	0.372	0.356	0.249	-0.39	1							
OC	.508*	0.453	0.283	-.507*	.706**	1						
OM	.718**	.636**	.549*	-.638**	.717**	.878**	1					
TP	.793**	.746**	.656**	-.771**	.592**	.852**	.914**	1				
CLAY	.682**	.641**	.521*	-.682**	.810**	.908**	.929**	.919**	1			
SILT	-0.138	-0.207	-0.347	0.341	0.247	.547*	0.345	0.22	0.283	1		
SAND	.525*	.508*	0.45	-.531*	.694**	.845**	.833**	.848**	.841**	.480*	1	
WC	.666**	.667**	.608**	-.608**	.605**	.727**	.850**	.810**	.788**	0.282	.802**	1

** Correlation is significant at the 0.01 level (2-tailed).
* Correlation is significant at the 0.05 level (2-tailed).

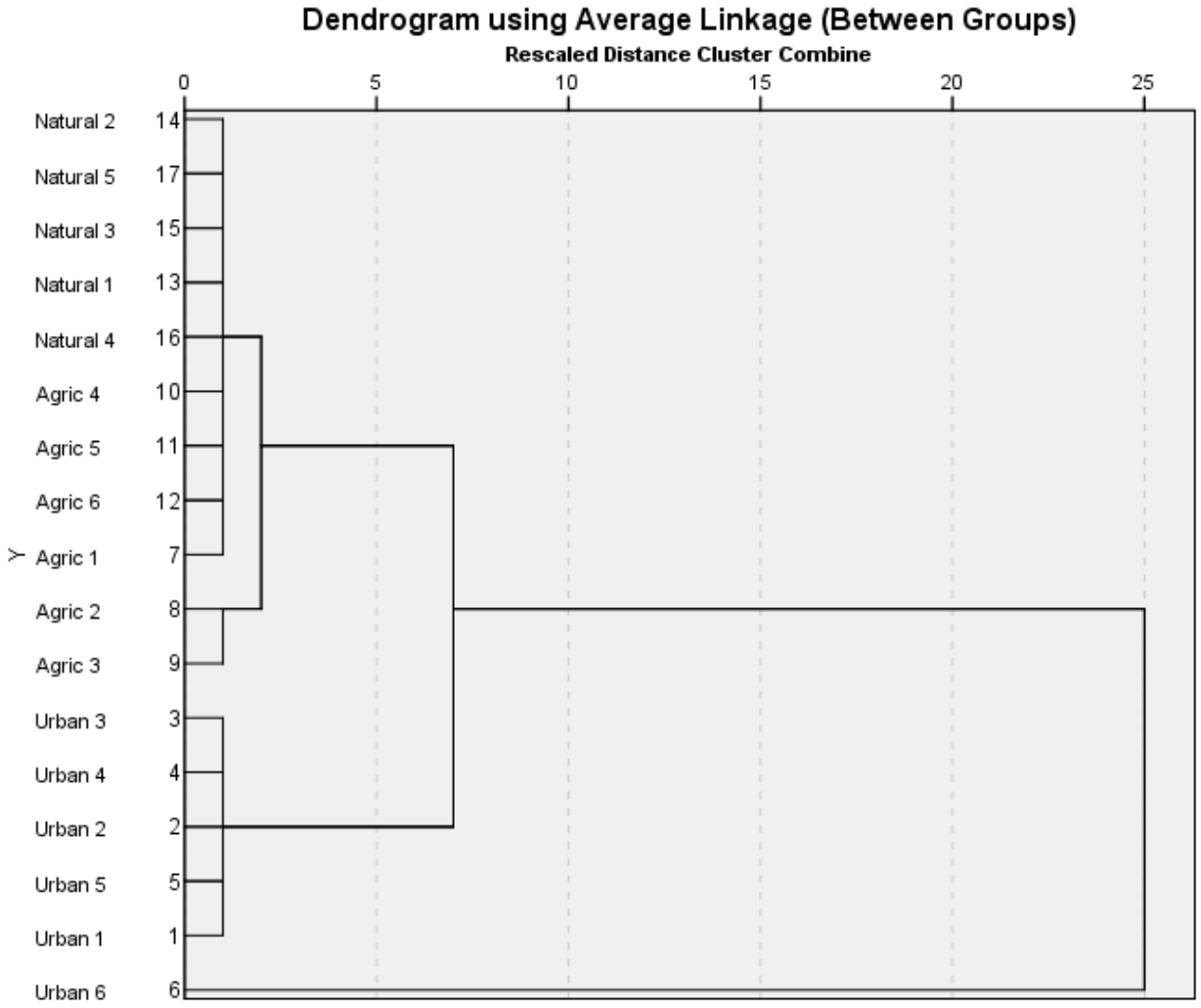

Figure 3 Cluster analysis of sampling sites

concentration of PAEs congeners. The significant relationship between OM and the three species of PAEs is due to well documented evidence that organic matters facilitate or enhances the adsorption of hydrophobic organic pollutants such as PAEs, into sediments (Raeisi et al., 2016). However, the association between the three species of PAEs concentration and OM values was more significant than the relationship between 3PAEs concentration and grain sizes of sediments. This implies that distribution of PAEs compounds is mostly influenced by the organic matter of sediments rather than the size of particles. This is majorly attributed to the fact that PAEs are a group of hydrophobic compounds and are quickly absorbed by the organic part of sediments and

accumulated (Magdouli et al., 2013). Besides, Strong and significant ($p<0.01$) negative correlation coefficients were observed between pH and all the measured PAEs (DBP, DEHP and DINP). This result may be attributed to the fact that the sorption of PAEs to sediments has been found to be pH dependent and inversely related (Zhao et al. 2004). A negative but significant relationship ($p < 0.01$) was also observed between pH value and OM and TP. Suggesting that low pH values, can reduce the rates of anoxic degradation, which will enhance the accumulation of TP and organic portion in the sediments (Jia et al., 2011). While a negative and a weak relationship was observed between pH and OC. Strong relationship ($P<0.01$) was observed between the three congeners of PAEs, suggesting that the measured PAEs probably had common sources of pollution and transport pathways, such as the urban runoff, surface runoff from waste disposal sites, discharges from industrial and municipal wastes as well as discharges from agricultural fields (Sun et al., 2013; Wang et al., 2014; Arfeania et al., 2019). Moreover, no significant associations were observed between EC, and ΣPAEs, suggesting that EC are not determinant parameters in the distribution of PAEs in sediments. There were also strong and positive correlation ($p<0.01$) between EC vs OC and OM, OC vs WC and OM, OM and TP, Clay vs WC and TP and clay vs WC. Strong and negative correlation were observed with EC and sand, clay and sand, silt and sand as well as sand and WC. The results of cluster analysis (Fig. 3) classify the study sites into two groups, group1 and group 2 according to their contamination degree and physiochemical parameters. All group 1, were located in low pollution areas, these include all the sites in natural field (NF1 to NF 5) and three sites in the agricultural zone including AG1, AG 2 and AG3. Group 2 include sampling sites UR 2, UR3, UR1, AG4, AG5, AG6, UR4, UR 5 and UR6 in urban and agricultural regions and were located in moderate-to-high pollution regions.

Comparison with other studies

Investigation on most commonly used PAEs congener in sediment around the world, including Thailand are lacking. A comparison of available data in (Table 3) shows that U-Tapao river basin has higher PAEs concentration than those of Cianjhen river in Taiwan (Yang et al., 2017) and Jiulong river estuary in China (Li et al., 2017), though it is lower than values reported elsewhere, Kaoshiung Harbor, Taiwan (Chiu et al., 2017), Shannon international river basin, Ireland (Kelly et al., 2010) and Garda Sedimentation facility in Sweden (Bjorklund et al., 2010), but comparable with PAEs levels reported for Jiulong river, China (Li et al., 2017) and False Creek Harbor , Canada (Mackintosh et al., 2002). The concentration of DBP is comparable among several studies (Mackintosh et al., 2002; Chen et al., 2017; Li et al., 2017). The highest DiNP and DEHP occurred in the Garda Goteborg Facility in Sweden (Bjorklund et al., 2010), followed by False Creek Vancouver, Canada (Mackintosh et al., 2006) and Kaohsiung Harbor in Taiwan (Chiu et al., 2017). The lowest DEHP concentration occurred in the Cianjhen River, Taiwan. For DINP and DBP the lowest concentration occurred in Jiulong River Estuary (Li et al., 2017), and China and Cianjhen river, Taiwan (Yang et al., 2017), respectively. Furthermore, comparison by PAEs pollution index (PPI) shows that the pollution status of U-Tapao river sediments is well below those reported for Bhoai and Yellow seas, China and Epe and Lagos Lagoon in Nigeria (Adeogun et al., 2015; Zhang et al., 2018), comparable with values reported for Ogun River, Nigeria; but higher than levels recorded for Asjire and Eleyele lakes, Nigeria (Adeniyi et al., 2011; Adeogun et al., 2015). The PPI in several studies indicated that sediments are significant sinks of PAEs in aquatic environment (Adeogun et al., 2015; Zhang et al., 2018).

Inventory of PAEs in the suburban river sediments

Riverine runoffs are an important vector in transporting organic pollutants from terrestrial sources to oceans. Clearly determining the amounts of organic pollutants transferred by riverine runoff can shed light into the cross-boundary transfer and help estimate the regional contributions of chemical contaminants to global inventory (Guan et al., 2009). Previous studies revealed that sediments do not only act as sinks to organic pollutants, but serves as vectors to transfer organic chemical pollutants from riverine ecosystems to coastal aquatic environment (Zhao et al., 2011; Mi et al., 2019). In this work, the inventory of sedimentary transfer of PAEs from U-Tapao River basin to Songkla Lake was calculated using equations 1 (Zhao et al., 2011):

$$Inventory = C \times p \times D \times a \quad (1)$$

where C (ng/g) is the mean concentration of PAEs in the sediment
p (g/cm3) is the dry density of the sediment
A (cm2) represents the area of the watershed
D (cm/y) is the sedimentation rate
a (y) is the number of years considered in the inventory.

In this present study, the values of sedimentation rate and the dry bulk density of the U-Tapao River basin were 0.78 cm/y and 1.0 g/cm3, respectively (Santi Rakswong, 2017). Based on the total area of U-Tapao watershed of 2393 km^2, 5 years accumulation and total mean concentration of PAEs, it was estimated that the inventories of the total PAEs was 308.44 tons in 5years.Based on land use pattern the urban area (UR) contributed the highest inventory of 205.06, followed by AG with 72.69 and NF contributing the least inventory value of 30.68 ton. Suggesting that the contaminated sediments in the study area may be a potential source of PAEs in Songkla lake. The estimated total inventory of PAEs in U-Tapao river basin was higher than the values reported for the Mobile Bay (13.82 tons) and the eastern Mississippi Sound (1.61 tons) sediments, in Alabama, United State of America (USA) (Lu et al., 2023), below values reported for Taihu lake (389.44 tons) in China (Zhang et al., 2021). It is worthy of note that the total area of U-Tapao river basin is smaller

Table 5: Concentration of 3PAEs in this present study compared with other global studies (ng/g).

Location	DBP	DEHP	DINP	References
Kaoshiung Harbor Taiwan	0.0-34.6	152.6-14,646.6	0.00-67,495.9	Chiu et al., 2017
Shannon international river basin, Ireland	9460-19440	7090-25270	ND-6160	Kelly et al., 2010
False Creek Vancouver, Canada	9320-63,900	7,350-136,000	14,700-50,400	Mackintosh et al., 2006
Jiulong River Estuary, China	1.6 -92.8	4.3-394.7	Nd-110	Li et al., 2017
Dianbao River, Taiwan	400 -1865	494-1947	361-1277	Yang et al., 2015
Jiulong River, China	3.0-230	7.00-1160	ND -470	Li et al., 2017
ChangJiang river Estuary, China	340-7080	260-8550	NA	Zhang et al., 2018
Cianjhen river, Taiwan	<1-1.00	217-445	189-398	Yang et al., 2016
Fengshan River, Taiwan	ND-112.7	29-11,519	68.3-17,668	Chen et al., 2017
Houjing river,Taiwan	1987	2144	934	Yang et al., 2015
U-Tapao canal, Thailand	ND-280	190-890	ND-840	Kingsleyand Witthayawirasak, 2020
GardaGoteborg, Sweden	100-860	1600-95000	65000-351000	Bjorklund et al., 2009
Masan Bay, Korea	1.20–70.8	23.5–3570	27.1–22,700	Kim et al., 2020
Korean coastal waters, Korea	0.48–56.2	1.00–2740	21.3–553	Lee et al., 2020
Fengshan River, Taiwan	ND- 112.7	29- 11,519	68.3- 17,668	Lin et al., 2022
Salt River, southern Taiwan	1.1-132.8	33- 9246	92-5373	Chen et al., 2022

than those previously studied aquatic ecosystem, including Bohai Sea (78,000 km^2), Yellow Sea (380,000 km^2) and East China Sea (1,200,000 km^2). Among the individual PAEs species, DEHP had the highest inventory in the basin, followed by DINP and then DBP. For instance, the inventories of DEHP, DINP and DBP in watershed were 12.03; 8.34; 3.93 tons, respectively. The inventories reflected in this present work would represent deposits in the last five years. Presumably, sediments of U-Tapao river basin may have served as a significant reservoir and a potential source of PAEs contamination in Songkla Lake, the largest natural Lake in Thailand, which flows into the Gulf of Thailand and then global ocean. We recommend a comprehensive assessment of the spatial and seasonal distribution of 16 species of PAEs in all the environmental media such as surface water, suspended particulate matter and sediments as well as aquatic biota in U-Tapao river basin and Sonkla Lake.

Conclusion

This study has provided the primary data on the level of PAEs in the sediment in U-Tapao watershed in areas with different land uses including urban, agricultural, and natural zones. Our result revealed that PAEs are ubiquitous environmental pollutants in this area, and the main compounds were DEHP, DINP and DBP. In addition, we observed that the average concentrations of Σ3PAEs were significantly higher in the UR zones, compared with AG and NF regions of the aquatic ecosystem. The concentration of the PAEs compounds had a positive and significant relationship with OM, indicating that the distribution of PAEs in the sediments of U-Tapao river basin is profoundly influenced by the organic content of the riverine sediments. Also, the level of PAEs species had a strong and significant negative correlation with pH, suggesting that the sorption of PAEs in sediments is pH dependent and inversely related. Moreover, the concentration of PAEs compounds had a positive and significant relationship with each other, suggesting that the sources of pollution and pathway transport of PAEs in the sediments of the watershed were similar. Large amounts of PAEs are discharged from industrial and municipal wastewater effluent into the watershed. In addition, PAEs may be washed out by rainwater, carried in drainage water, leachates from dumpsites and indiscriminately dumped plastics to the watershed and finally deposited in the sediments. Furthermore, other sources of contamination of PAEs in the river basin include application of fertilizers and pesticides, plastic mulching films, agrochemicals, animal feeds and dumps and polymer irrigation pipes. The inventory of the total ∑3PAEs, suggested that the contaminated sediments in the study area may be a potential source of PAEs discharge into the largest lake in Thailand, named Songkla Lake. Consequently, we recommend the inclusion of PAEs in the legislations and regulatory documents addressing the discharging of wastes containing hazardous pollutants into aquatic environments in Thailand.

Conflict of Interest

The authors declare no conflict of interest with respect to the authorship or publication of this work

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