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Polycyclic Aromatic Hydrocarbons (PAHs) Potential Sources in Sediments of Plawangan Timur, Segara Anakan, Cilacap: Occurrence and Distribution

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Abstract

Polycyclic Aromatic Hydrocarbons (PAHs) have the potential to contaminate the coastal area of Plawangan Timur, Cilacap, Indonesia. Therefore, this research aims to determine the composition, distribution, source, and contamination level of PAHs in this region. Sediment samples were taken at seven stations. The research findings indicate that among the 16 types of PAHs, the proportion of each PAH varied between 3-12%. Notably, Benzo(a) Pyrene (BaP) had the highest proportion at 12%, while Naphthalene had the lowest proportion at 3%. According to the distribution pattern, PAHs with a high molecular weight (HMW) are more prevalent than those with a low molecular weight (LMW). The total amount of PAHs ranged from 185 to 1289.9 mg.kg^{-1} (dw), with an average of 1016.97 mg.kg^{-1} (dw). Using a diagnostic ratio, it was determined that the origin of the PAHs was either petrogenic, pyrogenic, mixed petrogenic and pyrogenic, or pyrolytic. As a result, Plawangan Timur's degree of pollution was classified as a very high polluted. The average concentration value of each PAHs is higher than the values for the Effect Range Low (ERL) and Effect Range Median (ERM) ranges, indicated there is a significant ecological risk that could have negative consequences on marine life and the ecosystem. It is urgently to handle PAHs pollution due to their relatively high content in sediments. This can be achieved by improving the management of wastewater treatment plants (WWTPs) for industry, optimizing the role of mangrove forests in reducing pollution in the waters, as well as building and increasing collaboration between related agencies in the prevention of marine oil spills.

Keywords: *Segara Anakan, Hydrocarbons, Crude oil, Bioremediation*

Introduction

Petroleum hydrocarbons are commonly employed as a source of energy in many industrial applications, particularly within the transportation domain (Sa'diyah and Juliastuti, 2015). However, it is important to note that their utilization might lead to environmental contamination in aquatic ecosystems (Adipah, 2018; Truskewycz *et al*., 2019; Achyani *et al*., 2021). The occurrence of hydrocarbons in water arises from both natural origins, such as forest fires, volcanic eruptions, and petroleum seepage, and anthropogenic causes, encompassing petroleum spills, combustion engines, and automobile fuel leaks (Jazza *et al*., 2016; Wahyuni *et al*., 2017; Kurniawan *et al*., 2018). Yasmin and Wulansarie (2018) have found that the occurrence of oil spills in marine environments is a significant risk and can result in substantial harm to coastal ecosystems.

Plawangan Timur, Sagara Anakan, Cilacap is among the coastal regions that is facing the potential risk of petroleum hydrocarbon pollution. Plawangan Timur is an area characterized by the presence of various oil and gas industrial activities, Cement industry, PLTU Cilacap, and a significant volume of port traffic (Syakti *et al*., 2013; Dwi *et al*., 2019). In addition, hydrocarbon pollution can arise from several sources such as fisheries, agricultural practices, home activities, Sleko Port, Tanjung Intan Cilacap Port, and urban garbage (Hidayati *et al*., 2014; Syakti, 2016; Piranti *et al*., 2020). The

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Polycyclic Aromatic Hydrocarbons (PAHs) have garnered significant scientific interest owing to their enduring nature and harmful properties, including toxicity, mutagenicity, and carcinogenicity (Liu *et al*., 2012; Zhang *et al*., 2016; Ji *et al*., 2021).

This research seeks to investigate the composition, distribution, and source of polycyclic aromatic hydrocarbons (PAHs) in Segara Anakan, as well as assess the extent of pollution caused by these substances. Given the detrimental environmental consequences associated with PAHs and the significance of understanding their presence in water bodies, this study aims to ascertain the composition, distribution, source, and extent of PAH pollution in Segara Anakan, Cilacap.

Materials and Methods

This research was conducted in September 2020, in Plawangan Timur Segara Anakan, Cilacap, consisting of seven stations as shown in Figure 1. Plawangan Timur is situated within the Segara Anakan Cilacap Lagoon ecosystem area, which is the estuary of the Sapuregel Kembang Kuning and Donan. The water in this area has a depth ranging from 5 to 10 m and is affected by both saltwater and freshwater. These characteristics provide an environment suitable for the growth of mangrove plants, resulting in the formation of a mangrove ecosystem. The seven stations are SA-1 (Kembang Kuning River Estuary), SA-2 (Banyusrep), SA-3 (Banyusrep, Lomanis), SA-4 (Cimeong, Kutawaru), SA-5 (Kalianget, Cidapur), SA-6 (Donan River Estuary), and SA-7 (Tambakreja, Sleko).

Sediment sampling

Sediment samples were obtained using an Ekman grab at three distinct locations for each station, resulting in composite samples. These samples were then placed in aluminium trays and thoroughly mixed to achieve homogeneity. The specimens are inserted into an aluminium standing bag and afterwards stored within a temperature-controlled container set at 4°C. Sediment samples were analized in the Soil/Land Resources Laboratory of the Faculty of Agriculture (TOC Analysis) and the Research Laboratory of Jenderal Soedirman University.

Polycyclic Aromatic Hydrocarbons (PAHs) analysis

A sediment sample weighing 100 g was introduced into a plastic container and subjected to freezing at a temperature of -80°C within a freezer. The sample was put into the freeze-drying apparatus, where the temperature was adjusted to a value below -50^{o}C, while the pressure was maintained below 1000 millitorr. Once the hoover has reached a state of stability, it initiates its operation, causing the water and ice particles to undergo evaporation and subsequently flow through the hose. The water underwent filtration and afterwards discharged into a receptacle designated for the residual water.

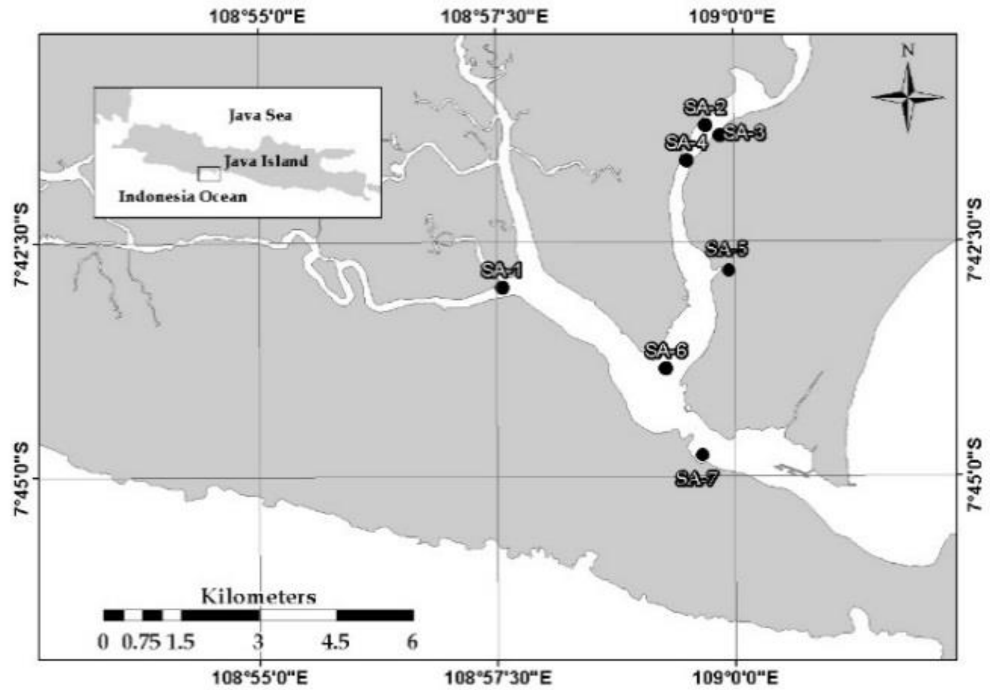

Figure 1. Sampling location in the Plawangan Timur

Extraction and separation

Sediment samples were extracted at 10 g with 150 ml of SupraSolv grade DCM/HEX (1:1.v:v) in soxtherm for 3.5 h. The extraction of 2 ml was concentrated and evaporated on a rotary evaporator and weighed before fractionation. The Aliphatic (F1) and Aromatic (F2) fractions were separated using a 1.0 x 30 cm borosilicate glass column with 8 g of silica gel and 8 g of alumina (the bottom and top of the column). Both adsorbents were deactivated with 5% (w:w) distilled H_2O before use and F1 was eluted with 30 ml of HEPT and 20 ml of HEPT/DCM (90:10,v:v), while F2 was eluted with 40 ml of HEPT/DCM (80:20,v:v). Subsequently, both fractions were concentrated, the result was accommodated and evaporated on a rotary evaporator, and placed into a vial bottle.

PAHs analysis: Gas Chromatography-Mass Spectrometry

Fractions F1 and F2 were analyzed using GC-MS (Gas Chromatography-Mass Spectrometry). Each sample were put into vials the spitless injection (30 s) and Elite 5MS column (30 m x 0.25 mm x 0.25 µm) methods were used. The column was programmed at 40°C for 2 min and raised from 5°C.min^{-1} to 120°C. After the final at 310°C, it was maintained for 20 min using helium as the carrier gas at a constant 1 ml.min^{-1}. The injector was programmed from 50°C to 250°C and the sample was injected using a micro syringe. Analyte molecules are carried by helium gas through columns. Subsequently, it was captured by a detector, which was sent to a recorder for processing to produce chromatograms of the different types of compounds.

Total Organic Carbon (TOC)

Sediment samples of 0.500 g were weighed and put in a measuring flask of 100 ml. A 5 ml of $K_2Cr_2O_7$ 1 N was added and shaken, followed by 7.5 ml of H_2SO_4, which was also shaken, set aside for 30 min, and diluted with aquadest. The mixture was allowed to stand for 24 h and the sample analysis was measured with a spectrophotometer (561 nm). As a comparison, standard series solutions of 0 ppm and 250 ppm were prepared by pipetting 0 and 5 ml of standard 5,000 ppm solution into a 100 ml measuring flask with the same treatment.

The measurement data were compared descriptively to determine the distribution and composition of PAHs. The source was analyzed by a diagnostic ratio as shown in Table 1 and the analysis of contamination with the total levels of PAHs (Low Molecular Weight (LMW) + High Molecular Weight (HMW)) obtained was classified by the degree of contamination (Table 2.). The PAHs levels were compared with standard values of ERL (Effect Range Low) and ERM (Effect Range Median) pollution, as well as sediment comparison with Sediment Quality Guidelines (SQGs) according to the Canadian Council of Ministers of the Environment (CCME, 2001). Furthermore, the relationship between PAHs and TOC was analyzed by a correlation test.

Table 1. Diagnostic Ratio

PAHs Ratio	Criteria	Source
LMW/HMW	< 1	Pyrogenic
	>1	Petrogenic
Flu/(Flu + Pyr)	<0.4	Petrogenic
	0.4-0.5	Mixture
	>0.5	Pyrogenic
Ant/(Ant+Phe)	<0.1	Petrogenic
	>0.1	Pyrogenic
Phe/Ant	>10	Petrogenic
	<10	Pyrogenic
Flu/Pyr	<1	Petrogenic
	>1	Pyrogenic
Ant/178	<0.1	Petrogenic
	>0.1	Pyrogenic
BaP/(BaP + Chy)	0.6-0.9	Petrogenic
	<0.2	Pyrogenic
	0.4-0.6	Mixture
IndP/276	<0.2	Petrogenic
	>0.35	Pyrogenic
	0.2-0.35	Mixture
Flu/202	<0.4	Petrogenic
	>0.5	Pyrogenic
	0.4-0.5	Mixture
BaAnth/228	<0.2	Petrogenic
	>0.35	Pyrogenic
	0.2-0.35	Mixture

Table 2. Level of Contamination of PAHs in Sediments ($ng.g^{-1}$)

Category	Contamination value ($ng.g^{-1}$)
Low	0-100
Moderate	100-1000
High	1000-5000
Very High	> 5000

Table 3. Composition of PAHs (%) in sediments

Types of PAHs	Composition (%)
Naphthalene (NaP)	3
Acenaphthylene (Acy)	4
Acenaphthene (Ace)	5
Fluorene (Fl)	8
Anthracene (Ant)	6
Phenanthrene (Phe)	9
Fluoranthene (Flu)	11
Pyrene (Pyr)	8
Benzo(a)anthracene (BaAnt)	7
Chrysene (Chr)	6
Benzo(b)fluoranthene (BbF)	4
Benzo(k)fluoranthene (BkF)	5
Benzo(a)pyrene (BaP)	12
Indeno(1,2,3-cd)pyrene (IcdP)	4
Benzo(g,h,i)perylene (BghiPer)	5
Dibenzo(a,h)Anthracene (DbahAnt)	4

Result and Discussion

Composition and distribution of PAHs

The composition of PAHs obtained in sediments in Plawangan Timur varied from 3 to 12% with 16 types as shown in the Table 3 The two most abundant compounds are Benzo(a) Pyrene (BaP) (12%) and fluoranthene (Flu) (11%). According to previous research, BaP and Flu are HMW compounds that originated from motor vehicle engines and petroleum spills (Hasan, 2020), with 5 and 4 benzene rings (Edward, 2012). The lowest composition is naphthalene (3%), which originated from industrial activities, oil spills, and urban waste (Edward, 2017) and has 2-3 benzene rings (Deng *et al*., 2013; Munawir and Yogaswara, 2018).

The low molecular weight PAHs (LMW-PAHs), which consist of 2-3 aromatic rings, are known to be very dangerous compounds. On the other hand, the high molecular weight PAHs (HMW-PAHs), containing 4-6 aromatic rings, are widely acknowledged as genotoxic substances (Lu *et al*., 2021). The distribution of PAHs in Plawangan Timur based on LMW and HMW as shown in Figure 2 was 185 $mg.kg^{-1}$ - 1847.1 $mg.kg^{-1}$ with an average of 1016.97 $mg.kg^{-1}$.

The highest level of PAHs was found in Banyusrep and Kali Anget, Cidapur, while the lowest is in the Kembang Kuning River Estuary. Banyusrep and Kali Anget, Cidapur, are industrial areas and the disposal of residual waste oil and gas from oil refinery industry, which enters the waters. Both locations have various activities such as industrial, domestic, fisheries, and ship traffic that can be a source of pollution by PAHs (Syakti *et al*., 2013; Hidayati *et al*., 2014; Dwi *et al*., 2019). The PAHs levels in Plawangan Timur is relatively high and the distribution is uniform. This is because sediments in the Plawangan Timur area accumulate more PAHs pollutants and sediment textures in the form of mud. This makes the sediments have a high content of organic matter, thereby absorbing more PAHs (Ahmad, 2012; Edward, 2012).

The PAHs distribution shows that those with 3 and 4 benzene rings are more dominant in all stations than those with 2, 5, and 6 (Figure 3.). The examples of PAHs with 3 to 4 benzene rings are Acenaphthylene, Acenaphthene, Fluorene, Anthracene, Phenanthrene, Fluoranthene, Pyrene, Benzo(a) Anthracene, and Chrysene. Meanwhile, the most general types were found from petrogenic sources and had more than 3 benzene rings, which were included in HMW (Yogaswara *et al*., 2019).

Potential sources of PAHs

The origin of PAHs compounds can be determined using the diagnostic ratio method

(Yunker *et al.,* 2002), which can differentiate between petrogenic and pyrogenic sources (Aichner *et al.*, 2007; Dominguez *et al.,* 2010). Based on the results of the diagnostic ratio analysis in Table 4, it was discovered that the sources of PAHs pollution in Plawangan Timur are petrogenic, pyrogenic, their mixtures, and pyrolytic. In this research, the origin of PAHs in Plawangan Timur is the same as Syakti *et al.* (2013) because the location is an industrial and residential area. The existence of the oil and gas industry, cement industry, ports, sea transportation routes, and other activities such as domestic, fisheries, agriculture, and waste inputs from rivers in Plawangan Barat, which come from domestic waste, are also become a source of pollution by PAHs.

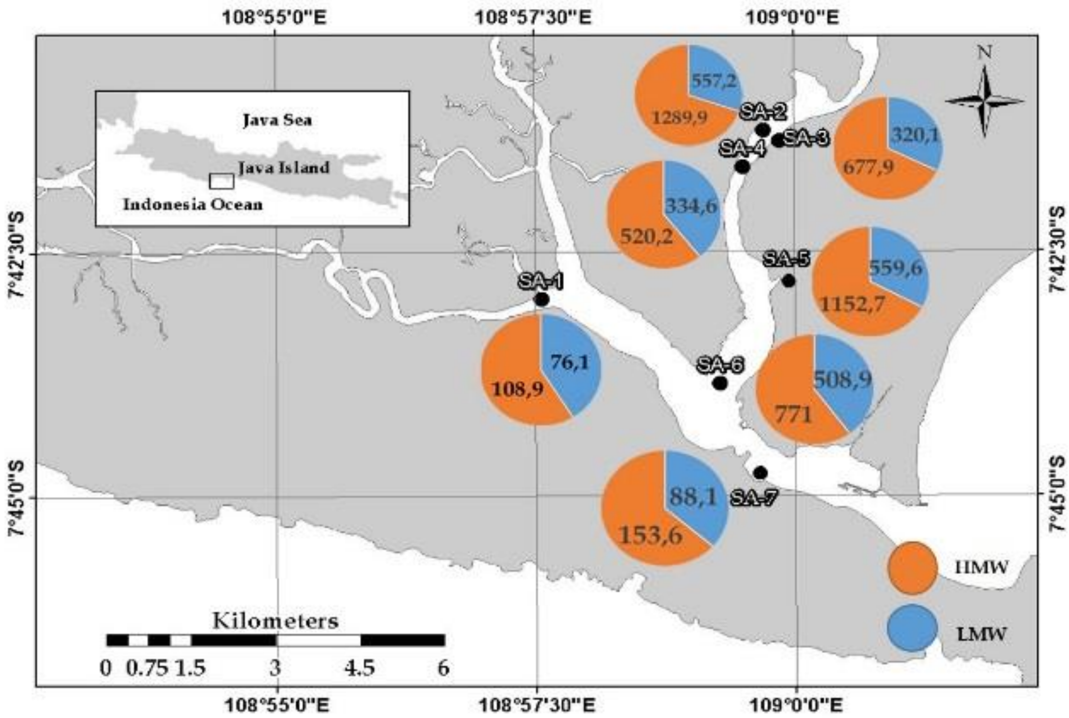

Figure 2. The distribution of PAHs (mg.kg^{-1}) based on LMW and HMW

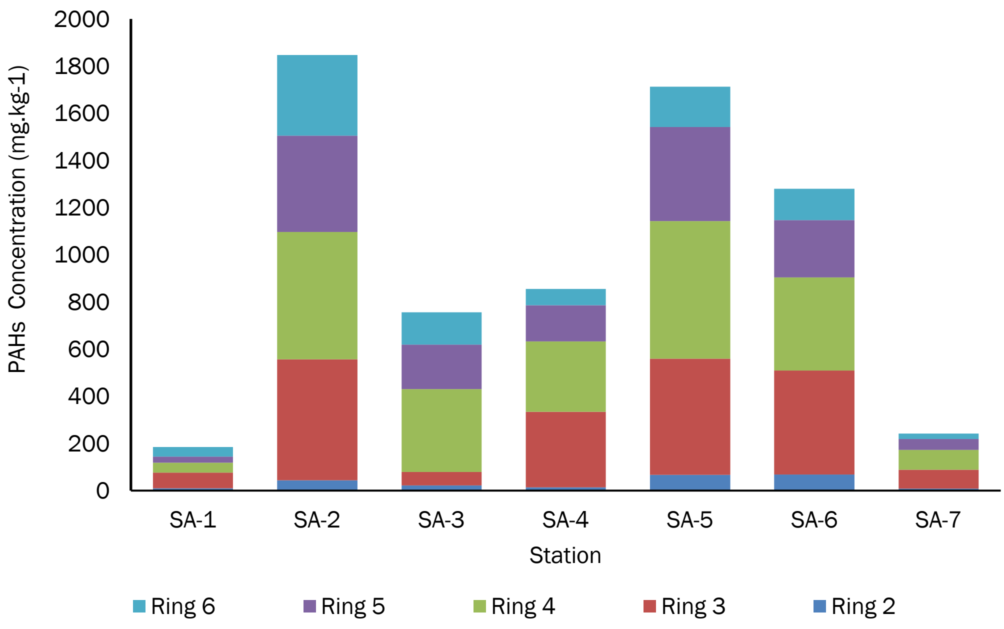

Figure 3. The Distribution of PAHs Based on the Benzene Ring

Table 4. A Diagnostic Ratio of Individual PAHs in Sediments

Diagnostic Ratio	Station						
	SA-1	SA-2	SA-3	SA-4	SA-5	SA-6	SA-7
LMW/HMW	0,70 b	0,43 b	0,47 b	0,64 b	0,49 b	0,66 b	0,57 b
BaA/(BaA + Chr)	0,39 e, b	0,59 e, b	0,56 e, b	0,84 e, b	0,51 e, b	0,43 e, b	0,48 e, b
InP/(InP + BghiP)	0,57 b	0,31 a	0,65 b	0,36 a	0,80 b	0,35 a	0,37 a
Flu/(Flu + Pyr)	0,40 c	0,53 B	0,51 b	0,52 b	0,72 b	0,60 b	0,56 b
Ant/(Ant+Phe)	0,57 b	0,40 b	0,41 b	0,46 b	0,33 b	0,36 b	0,57 b
BaP/(BaP + Chy)	0,45 c	0,75 a	0,60 c	0,86 a	0,70 a	0,43 c	0,38 b
Pyr/(IcdP + BghiPer)	0,65 b	0,63 b	1,18 b	2,00 b	0,99 b	0,85 b	1,26 b
Phe/Ant	0,77 b,d	1,49 b,d	1,44 b,d	1,17 b,d	2,04 b,d	1,80 b,d	0,76 b,d
Flu/Pyr	0,66 a	1,11 b	1,03 b	1,07 b	2,61 b	1,48 b	1,25 b
BaAnth/228	0,03 a	0,61 b	0,39 b	0,34 c	0,49 b	0,34 c	0,09 a
Ant/178	0,07 a	0,55 b	0,32 b	0,43 b	0,45 b	0,31 b	0,14 b
IndP/276	0,04 a	0,26 c	0,19 a	0,06 a	0,29 c	0,13 b	0,02 b
Flu/202	0,05 a	0,79 b	0,49 c	0,53 b	1,30 b	0,64 b	0,11 a
Fl/Pyr	1,50 d	0,85 a	0,83 a	0,90 a	1,43 d	1,25 d	0,65 a

Note = a : Petrogenic; c : Mixture of petrogenic and pyrogenic; e : Vehicle emission; b : Pyrogenic; d : Pyrolytics

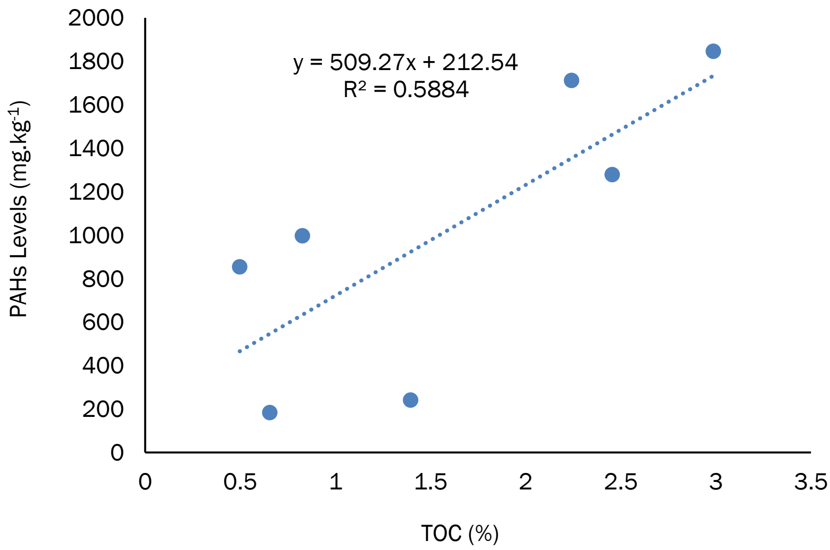

Figure 4. Correlation of PAHs and TOC in Sediments

Correlation of PAHs and TOC

The results showed that the content of TOC is positively correlated with PAHs. This indicated that the higher the content of TOC in sediments, the greater the PAHs. The regression analysis gave an equation of y= 509.27x+212.54 with a coefficient of determination R^2 of 0.5884 as shown in Figure 4. This demonstrates a moderate association between PAHs and TOC, with TOC exerting a 58.8% influence on the concentration of PAHs in this study, while the remaining influence was attributed to other variables. The correlation values (R^2) are categorized based on value, namely 0.00 -0.20 (very weak), 0.21-0.40 (weak), 0.41-0.70 (medium), 0.71-0.90 (strong), and 0.91-1.00 (very strong) (Mutaqin *et al.,* 2014). The texture of sediments in Plawangan Timur is in form of mud, which has a high organic matter, therefore, it easily absorbs PAHs content (Edward, 2018).

PAHs contamination rate

The level of total PAHs (LMW+HMW) ranged from 185 $mg.kg^{-1}$ to 1847.1 $mg.kg^{-1}$, with an average of 1016.97 $mg.kg^{-1}$ as shown in Table 5. It was discovered that the level of contamination of PAHs in Plawangan Timur is in the category of highly polluted. According to Nascimento *et al.* (2017) and

Table 5. Total PAHs Levels ($mg.kg^{-1}$) in Sediments

No.		Type of PAHs	Station Name						
			SA-1	SA-2	SA-3	SA-4	SA-5	SA-6	SA-7
1	BMR	NaP	10,2	43,8	21,5	14,5	67,3	67,5	9,1
2		Acy	9,7	48,7	38,7	21,4	46,5	91,4	14,2
3		Ace	11,4	97,2	41,2	43,9	61,2	87,6	8,5
4		Fl	21,3	122,5	79,4	89,2	143	109,5	11,9
5		Ant	13,3	98,3	57,2	76,3	79,5	54,6	25,2
6		Phe	10,2	146,7	82,1	89,3	162,1	98,3	19,2
	Σ BMR		76,1	557,2	320,1	334,6	559,6	508,9	88,1
7	BMT	Flu	9,4	159,7	98,2	106,1	262	129,7	22,7
8		Pyr	14,2	144,3	95,3	99,4	100,3	87,9	18,2
9		BaAnt	7,3	139,2	88,4	78,1	112,3	76,5	20,8
10		Chr	11,4	96,3	69,4	14,5	108	100,4	22,6
11		BbF	8,8	56,4	37,2	23,7	67,6	78,5	14,8
12		BkF	7,4	59,2	46,9	36,1	78,1	89,3	17,9
13		BaP	9,4	293,1	105,2	92,8	253,4	76,2	13,7
14		IcdP	12,4	71,2	52,6	17,9	81,1	35,9	5,3
15		BghiPer	9,4	158,4	28,3	31,7	20,4	67,2	9,1
16		DbahAnt	19,2	112,1	56,4	19,9	69,5	29,4	8,5
	Σ BMT		108,9	1289,9	677,9	520,2	1152,7	771	153,6
	BMR+BMT		185	1847,1	998	854,8	1712,3	1279,9	241,7
	∑BMR+BMT				1016,97				

Table 6. SQGS Concentration of PAHs in Sediment ($ng.g^{-1}$)

No.	Type of PAHs	ERL	ERM	CCME (2001)	Range of research results	Mean
1	NaP	160	2100	34,6	9.100-67.500	33.410
2	Acy	44	640	5,87	9.700-91.400	38.660
3	Ace	16	500	6,71	8.500-87.600	50.140
4	Fl	19	540	21,2	11.900-122.500	82.400
5	Ant	85.3	1100	46,9	13.300-98.300	57.770
6	Phe	240	1500	86,7	10.200-146.700	86.840
7	Flu	600	5100	113	9.400-159.700	112.540
8	Pyr	665	2600	153	14.200-144.300	79.940
9	BaAnt	261	1600	74,8	7.300-139.200	74.660
10	Chr	384	2800	108	11.400-100.400	60.370
11	BbF	-	-	-	8.800-78.500	41.000
12	BkF	-	-	-	7.400-89.300	47.840
13	BaP	430	1600	88,8	9.400-293.100	120.540
14	IcdP	-	-	-	5.300-81.100	39.490
15	BghiPer	-	-	-	9.100-158.400	46.360
16	DbahAnt	63.4	260	6,22	8.500-112.100	45.000
	Total	4022	44792	-	153.500-1.970.100	1.016.960

Nasher *et al.* (2013), when the waters are contaminated by PAHs with a level of >5 μg.g^{-1}, it is categorized as very high. In sediments of Plawangan Timur, the level of the contaminant has passed the threshold value based on PP RI No. 22 of 2021, which is 0.003 ppm or 3 ppb for marine life. This high value has caused the death of fish, other marine life, and damaged mangrove ecosystems. This is characterized by the presence of several fish floating, fishermen's catches, and mangrove forest areas that are reduced. According to Yogaswara *et al.* (2019) and Harisam *et al.* (2018), high levels of PAHs in sediments will be accumulated by PAHs, causing death in fish and mangroves.

Environmental risk analysis has also been carried out to determine the potential impact of the presence of PAHs in sediments (Han *et al.,* 2019; Edward *et al.,* 2020). The total concentrations ranged from 153.500 ng.g^{-1} – 1.970.100 ng.g^{-1} with an average of 1.016.960 ng.g^{-1} (Table 6.). This indicated that the PAHs value is greater than ERL and ERM, indicating a high level of environmental risk in Plawangan Timur which affected the organism. Based on sediment quality guidelines (SQGS), when PAHs are greater than ERM, the effects that occur will be harmful to biological communities (Han *et al.,* 2019; Hong *et al.,* 2016). The toxicological effects of the pollutants of organic compounds PAHs can refer to the Canadian Council of Ministers of the Environment (CCME, 2001) (Jiang *et al.,* 2009). The results showed that the PAHs levels were greater than the standard pollution criteria according to CCME. This means that the levels on sediments can have harmful effects on marine life and the surrounding environment.

Conclusions

The composition of PAHs in Plawangan Timur ranged from 3 to 12%, where BaP (12%), and Flu (11%) have the highest composition, and naphthalene (3%) has the lowest. The distribution of PAHs shows that the proportion of HMW is greater compared to LMW and dominated by those with 3 and 4 benzene rings. In the research area, the PAHs originated from pyrogenic, petrogenic, a mixture of both, and pyrolytic. Meanwhile, the most influential sources are pyrogenic and petrogenic. The PAHs levels ranged from 185 mg.kg^{-1} -1847.1 mg.kg^{-1} with an average of 1016.97 mg.kg^{-1}, which indicated that the level of contamination is included in the category of highly polluted. An environmental risk analysis based on ERL and ERM showed that PAHs values are greater than the pollution criteria standards of the Canadian Council of Ministers of the Environment. Therefore, Plawangan Timur has a high level of ecological risk, which can cause harmful effects on marine life and the surrounding environment.

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