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Distribution and sources of polycyclic aromatic hydrocarbon (PAH) on sediment around Makassar coast

Muhammad Syahrir

Chemistry Department, Faculty of Mathematic and Natural Science, Universitas Negeri Makassar, Jl. A.P. Pettarani Gedung Phinisi

*m.syahrir7406@unm.ac.id , syahrir_gassa@yahoo.com

Abstract. This study conducts an analysis related to distribution and source of PAH in sediment around Makassar coast. Sediment sample has been taken up using Van Veen grab and put in a plastic sac, brought to laboratory, added with salt, stored in dried freezer, and followed for PAH analysis. Sample preparation has been carried out using DCM and n-hexane (1:1) solvent to obtain extract of organic matter (EOM). The EOM is fractionated to yield alkane fraction (F₁) and aromatic fraction (F₂) containing PAH. The GC-FID has been used to determine PAH concentration through analysis of F₂. Validation of the analytical method applies linearity, LoD, and LoQ. The recovery test has fulfilled for all PAH compounds except for *naphthalene* and *fluoranthene*. The highest concentration of PAHs is found at station 7 with *benzo(a)anthracene* 933.23±3.88 ppm and *perylene* 205.75±19.45 ppm. The lowest total concentration of PAHs is found at station 12 located at the estuary of Tello River caused by dilution from both river and sea water. The source of PAHs in sediment is generally from petrogenic and pyrolytic process.

1. Introduction

Beach in Indonesia generally suffered damage from both community and the community in marine sediments, especially near urban centers where oil was produced from offshore platforms. It is estimated that approximately 35 % of Indonesia oil production comes from oil wells offshore. Sedimentation of crude oil (such as n-alkenes and PAH) and other natural sources occurs because many components of hydrocarbons in the ocean is not soluble in water and thus immersed in the sediment [1]. The particles that make up marine sediments consist of two main origins: (1) forms of soluble compounds (mainly organic and inorganic) has been input into the ocean as a solid phase and (2) comes from the mainland or elsewhere as a result of waste [10].

Data on petroleum hydrocarbon contamination levels in the waters of Makassar Strait rarely obtained although the area is an important traffic of tanker, oil tankers coming from oil producers (in the Middle East) going to its main customers, Japan and North America. This route is an alternative pathway beside the Straits of Malacca which has become a narrow sea line but crowded, causing many oil tanker accident [13]. It also represents the main route of transportation of oil tanker from the Middle East toward the Java Sea and then through the Makassar Strait to their main consumers [14].

Research on PAH compounds has been carried out in several regions in the form of distribution, characteristics and accumulation in sediments, water and marine biota, but around the coast of Makassar has never been conducted quantitatively. Therefore, it is necessary to conduct a research



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study on distribution and sources of polycyclic aromatic hydrocarbon (PAH) on sediment in Makassar coast.

2. Reagents and Chemicals

The research used chemicals such as standard PAH (*Naphthalene*, *Pyrene*, *Fluoranthene*, *Phenanthrene*, *Acenaphthene*, *Benzo(a)Pyrene*, and *Perylene*). Other chemicals were sodium sulphate anhydrate, silica gel 70-240 mesh, alumina at p.a, while helium, hydrogen and nitrogen. Solvent n-hexane, dichloromethane with p.a. Water used was aquabidest distilled water.

Chemicals used in this research were glass equipment (Pyrex), rotary evaporator Buchii R II, vacuum pump (Hitachi, Ltd), sieve 1.00m no. 1 (Fisher Scientific Company), analytic balance Al.204 (Mettler Toledo), oven model 501 (Fisher), thermometer (100 °C) Hanna HI 314, hand refractometer, van Veen Grab, GPS (Global Positioning System) type Garmin 76 CSX, and gas chromatography detector FID from Shimadzu QP 2010.

3. Method

Sample used is sediment of Lae-lae island coast at Makassar, South Sulawesi at S 05°08.131' and E119°23.416' using van Veen Grab. Sediment sample was wrapped with aluminum foil and taken to laboratory at room temperature.

Sample of 10 grams was extracted for 1 hours with solvent dichloromethane and n-hexane (50:50=v/v=1:1) at 45 °C [4], [7]. For resulted extract, its solvent is evaporated with rotary evaporator Buchii to obtain organic material extract at 45°C [6]. Separation of other organic material carried during extraction was done with clean-up. The clean-up used alumina with base characteristic. The alumina was processed as follows: ±200 gram alumina was extracted with dichloromethane in soxhlet for about 16 hours, then heated in oven at 200 °C for 8 hours and inactivated by adding aquabidest (9-10% [16]. Distillate from clean-up process was separated using silica-gel – alumina (1:1) to obtain aliphatic and aromatic fractions. Extract PAH sample was analyzed with GC-FID chromatography with external standard method from GC-FID Chemical Engineering State Polytechnic of Ujung Pandang. PAH level in sediment is calculated using following formula:

$$C_x = \frac{C \times V \times fp}{B \left(1 - \frac{Ka}{100}\right)} \quad (1)$$

Note :

C_x = PAH concentration ($\mu\text{g/g}$)

C = Sample of concentration in linear regression ($\mu\text{g/g}$)

V = solution of volume in sample sediment (ml)

fp = $\frac{\text{final volume (ml)}}{\text{Initial volume (ml)}}$

B = Weight of test sample (gr)

Ka = Sample water content (%)

4. Results and discussion

4.1. Sampling of sediment

The sampling was carried out in the sediment of the surface around Makassar Coast in the same time in this area as many as 17 stations.

The sample taken was around 50 gram per one sheet. The length and the depth of sampling was ranged from 2 to 5 m depended on the condition of the sample, whether it was muddy or sandy. The water surface temperature in the location was between 28 – 31°C with acidity content (pH) of each station was 7.62 – 31°C.

Application of the method used from GC LPPT UGM method and is used in analyzing PAH using GC Chemical Engineering State Polytechnic of Ujung Pandang.

4.2. Method validation

The method validation used was using several parameters: Linearity, Limit of Detection (LoD), Limit of Quantification (LoQ), Accuracy and Linear interval [3].The target PAH analyzed using GC-FID QP-2010 for all target of PAH could be identified based on retention time and the high peak resulted was higher (Figure 1).

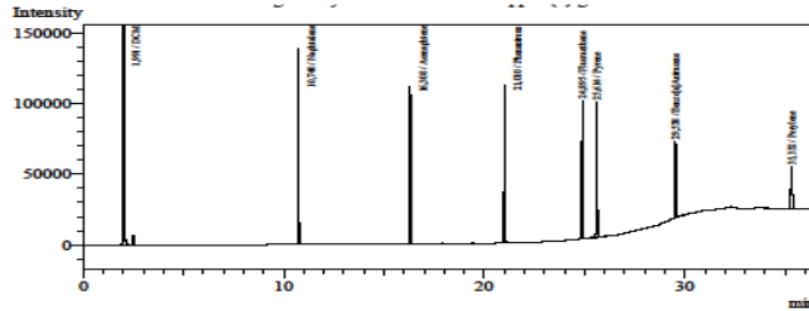


Figure 1. Chromatogram of Standard PAH of mixture of 500 ppm with GC-FID

Based on chromatogram of target PAH with mix of standard identified with retention time of each PAH (*Naphtalene: Acenaphthene: Phenanthrene: Fluorantene: Pyrene: Benzo(a)Antracene: and Perylene*) were 10.748; 16.308; 21.010; 24.895; 25.614; 29.538; 35.332 seconds respectively.

4.3. Separation evaluation

Retention factor (capacity = k');

Meeting the requirement of k' value more than 1 (one) except in Naphtalene.

Capacity factor is calculated using the equation:

$$k' = \frac{tr - tm}{tm} \tag{2}$$

In which tr is retention time of PAH, and tm is retention time of movement phases, and capacity factor (k').

Table 1. Determination of capacity factor

PAH	tr (minutes)	tm (minutes)	Capacity (k')
<i>Naphtalene</i>	10.74	1.991	4.399
<i>Acenaphthene</i>	16.308	1.991	7.193
<i>Phenanthrene</i>	21.01	1.991	9.556
<i>Fluorantene</i>	24.895	1.991	11.508
<i>Pyrene</i>	25.614	1.991	11.869
<i>Benzo(a)antracene</i>	29.538	1.991	13.835
<i>Perylene</i>	35.332	1.991	16.754

tr is retention time of PAH

tm is retention time from the phase of movement of 1.991 minutes

4.4. Selectivity factor (α)

Meeting the requirement from $1 < \alpha < 10$ [15]. The selectivity factor or separation factor (α) defined as the ratio of capacity factor of two peaks reflects the relative separation between the two nearest peaks

$$\alpha = \frac{tr_2 - tm}{tr_1 - tm} \quad (3)$$

If the value of α is above 1, it identifies that relative separation between the two peaks of nearest PAH was fairly good.

Table 2. Determination of selectivity factor

PAH	tr (minutes)	PAH Separation	Selectivity factor
<i>Napthalene</i>	10.74	<i>Napthalene and Acenaphthene</i>	0
<i>Acenaphthene</i>	16.308	<i>Acenaphthene and Fenanthrene</i>	1.634
<i>Fenanthrene</i>	21.01	<i>Fenanthrene and Fluorantene</i>	1.328
<i>Fluorantene</i>	24.895	<i>Fluorantene and Pyrene</i>	1.204
<i>Pyrene</i>	25.614	<i>Pyrene and Benzo(a)anthracene</i>	1.031
<i>Benzo(a)anthracene</i>	29.538	<i>Benzo(a)anthracene and Perylene</i>	1.661
<i>Perylene</i>	35.332	<i>Perylene</i>	1.21

From the result of determination of selectivity factor in Table 2, it showed the value of selectivity which is bigger than 1. This met the requirement as selectivity requirement of a separation between two nearest peaks which was good enough, except in naphthalene substance.

4.5. The number of Theoretical Plate

Meeting the requirement of N_{req} which is bigger than 7000 so that it is expected that the frequency of the happening of stability of analite in the stable phase enabled the creation of PAH separation perfectly [8] using the following equation:

$$N = 2 \pi \frac{(tr \cdot H)}{A} \quad (4)$$

Note:

tr = Retention Time
H = Peak Height
A = Peak Area

For example:

The number of theoretical plate (N_{req}) = $2 \pi \frac{(10748 \times 137081)^2}{326069}$ *Napthalene* substance = 2047713.32

The number of theoretical plate (N_{req}) = $2 \pi \frac{(16.308 \times 110906)^2}{286391}$ *Acenaphthene* substance = 1798535.48

From all types of PAH above, the value of N_{req} meet the requirement of higher than 7000, so that it is expected that the frequency of the happening of analit stability I the stable phase and moving phase enables the achievement of PAH separation perfectly [8].

Table 3. List of Factor of Capacity and the Number of Theoretical Plate of each PAH from the result of analysis of GC-FID column Rtx-5-ms

PAH	tr (minutes)	Number of Theoretical Plate (N_{req})	Capacity factor
<i>Naphtalene</i>	10.74	2047713.32	4.399
<i>Acenaphthene</i>	16.308	1798535.48	7.193
<i>Fenanthrene</i>	21.01	1973138.32	9.556
<i>Fluorantene</i>	24.895	1857240.92	11.508
<i>Pyrene</i>	25.614	1871640.96	11.869
<i>Benzo(a)anthracene</i>	29.538	1065960.92	13.835
<i>Perylene</i>	35.332	1001515.56	16.754

tr is retention time of PAH

tr is the retention time of moving phase of 1.991 minutes

From the above data for N_{req} toward GC-FID using column of Rtx-5-ms (Table 1), it has the theoretical plate of *Naphtalene* = 2047713.32, *Acenaphthene* = 1798535.48, *Phenanthrene* = 1973138.32, *Fluorantene* = 1857240.92, *Pyrene* = 1871640.96, *Benzo(a)anthracene* 1065960.92, and *Perylene* = 1001515.56, in which the number of theoretical plate from all target of PAH met the requirement of higher than 7000 so that it is expected the frequency of the happening of stability of analite in the stable phase and in moving phase can enable the achievement of separation of PAH perfectly [8].

Table 4. Retention time of average of each standard PAH in the analysis of GC-FID using column Rtx-5-ms

PAH	Retention time 1 (minutes)	Retention time 2 (minutes)	Average retention Time (minutes)
<i>Naphtalene</i>	10.748	10.755	10.751
<i>Acenaphthene</i>	16.308	16.318	16.313
<i>Phenanthrene</i>	21.010	21.023	21.017
<i>Fluorantene</i>	24.895	24.911	24.903
<i>Pyrene</i>	25.614	25.631	25.622
<i>Benzo(a)anthracene</i>	29.538	29.533	29.536
<i>Perylene</i>	35.332	35.364	35.348

Limit Detection (LoD) and Limit Quantification (LoQ) are shown in Table 5.

Table 5. The value of LoD, LoQ, Linear Interval, R, and Curve Equation Calibration of Standard PAH

PAH	LoD (ppm)	LoQ (ppm)	Linear Range (ppm)	R value	Curve Equation Calibration
<i>Naphtalene</i>	0,80	3.93	0 – 50 & 50 – 1000	0.994 0.996	$y = 551,4x + 807,9$ $y = 571,4x + 7099$
<i>Acenaphtene</i>	0,70	2.32	0 – 50 & 50 – 1000	0.986 0.997	$y = 492,9x + 2217$ $y = 507,2x + 7463$
<i>Phenantrene</i>	0,84	2.82	0 – 50 & 50 – 1000	0.992 0.998	$y = 544,8x + 1549$ $y = 571,6x + 4561$
<i>Fluorantene</i>	0,75	2.50	0 – 50 & 50 – 1000	0.989 0.998	$y = 500,3x + 2000$ $y = 545,4x + 4028$
<i>Pyrene</i>	1,13	3.78	0 – 50 & 50 – 1000	0.989 0.999	$y = 510,1x + 1862$ $y = 556,9x + 3281$
<i>Benzo(a)antracene</i>	0,96	3.20	0 – 50 & 50 – 1000	0.997 0.999	$y = 344,7x + 969,1$ $y = 342,8x + 2824$
<i>Perylene</i>	0,80	3.93	0 – 50 & 50 – 1000	0.991 0.998	$y = 374,9x + 603,0$ $y = 306,9x + 8049$

From the Table 5, it showed the value of LoD was lower than the concentration and it was not detected. Meanwhile, the value of LoQ was 3 times bigger than LoD. Besides the highest R value of 0.997 in the substance of *Benzo(a)antracene* and the lowest of 0.986 in the substance of *Acenaphtene*, which generally closed to 1 also supported the result.

4.6. Accuracy (Recovery test)

The accuracy of this method was determined by the method of adding standard and was stated with percentage of recovery (regain). The determination of recovery was carried out by adding 20 micro liter of standard PAH with concentration of 1 ppm, 2 ppm, and 4 ppm for the sample of sediment.

Table 6. List of value of recovery in the addition of standard PAH 1, 2, and 4 ppm toward the sample of sediment sheet of Lae-Lae Island

PAH	2 ppm (%)	SD
<i>Naphtalene</i>	33.8	5,4
<i>Acenaphtene</i>	80.2	11,2
<i>Phenantrene</i>	68.5	0
<i>Fluorantene</i>	128.4	34
<i>Pyrene</i>	110.8	6,7
<i>Benzo(a)antracene</i>	89.3	59,7
<i>Perylene</i>	95.6	22,9

The two recovery test method (Table 6) showed that the sample of sediment using addition standard method (addition into the sample) was found after it was analyzed using GC-FID with the highest value of 128.4 % in the substance of *Fluorantena* and the lowest of *Naphtalene* of 33.8 % and *Phenantrene* of 68,5 %. Meanwhile, other substances were in the interval of 80 -120% [2] as required, so this method is considerably accurate [11].

4.7. PAH concentration in the sediment around the Makassar Coast

Table 7. PAH concentration in the sample of sediment around the Makassar Coast in ppm (n=3)

PAH	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Naft	2,0± 0,2	6,1± 2,6	Nd	2,2± 0,0	nd 1,4±1	Nd	nd	nd 182,0	nd	nd	7,1±0, 8	Nd	nd	nd	nd	nd	nd
Ace	nd	nd	Nd	nd	,1	nd	nd	+31,0	nd	nd	nd	Nd	nd 11,	nd	nd	nd	nd
Fen	nd	5,3± 0,9	0,1±0 ,9	2,4± 0,2	17,6± 2,2	Nd	nd	nd	nd 205,1	nd 5,0±7	9,9± 8,4	6,5±0, 7	1,2±1, 7	1,±2 ,7	3,9±1,9 ,6	14,5±1	nd
Fluo	nd	nd	Nd	nd	nd	Nd	nd	+98,8	nd	nd	nd	Nd	nd	nd	nd	nd	nd
Pir	41,3± 4,6	nd	228,4 ±5,6	2,2± 0,0	nd	Nd	nd	nd	nd	nd	nd	Nd	nd	nd	524,0±	nd	nd
B(a)A	nd	nd	15,7± 4,9	0,2± 0,1	nd	Nd	933,2 ±3,9	nd	nd	nd	nd	Nd	nd	20,1	nd	nd	nd
Pery	nd	183,9 ±12,6	150,3 ±19,8	12,4 ±0,2	411,8 ±9,8	351,8 ±13,5	205,8 ±19,5	111,9 ±72,3	836,0 ±73,3	nd	292,2 ±29,9	98,9±4 3,3	36, 9	nd	313,3± 24,2	78,8±20,1	nd
PAH total	43,3±4, 8	195,3 ±16,1	394,4 ±31,2	19,4 ±0,5	430,7 ±13,1	251,7 ±13,5	1139 ±23,4	499± 202,1	840,9 ±80,3	9,94 ±8,4	305,8 ±30,6	100,1± 45,0	±39 ,6	527,9± 25,8	327,8± 25,8	78,8±20,1	nd

note:

nd = not detection

17 = station 17 (blank)

Based on the Table 7, it showed that the total PAH concentration of each station the highest was in station 5, station 7, station 8, station 9, and station 14, in sequence of (430,7± 13,2); (1139 ± 23,4); (499 ± 202,1); (840,9 ± 80,3) ; and (527,9±25,8) ppm respectively. Meanwhile, the lowest was in station 1, station 2, station 4, station 10, station 12, station 13 and station 16 were (43,3 ±4,8); (195,3±16,1) ; (19,4 ± 0,5) ; (9,94 ± 80,3) ; (100,1 ± 45,6) ; (222 ± 39,6) and (78,8 ± 20,1) ppm.

Station 5, station 7 and station 8 are complex of stores and Makassar Transtudio Complex and dense traffic condition. Station 9 and station 14 are close to the activities of citizen in dense condition and mainly activity around the water of Soekamo-Hatta Port. Besides, it is also near the orifice of Tallo River and industrial area of Makassar and TPA waste Antang, in which its waste is accumulated in river, so that it affects the increase of PAH content in station 9 and station 14 and the surrounding area. Total concentration of PAH in station 1, because there has been pollution in the volume of Jeneberang river and it is increasing when it meet with the sea with also bigger volume. Meanwhile, in station 11, it is an area with not so high pollution of PAH because it is around the coast and most of the citizen there is still in village and far enough from the activities of stores in the location. The concentration of PAH in station 10 in general was not detected the PAH because this area is a small island and far enough from Makassar Coast so that the result of the main activity in the land does not affect the increase of PAH around the station. Besides, the activities of the citizen around this area are generally fishermen and the sediment found there is general clean white. The low total concentration of PAH in station 12 was because it is in the area of orifice of Tello River, so that there is pollution in the concentration of PAH, either by the river of sea water.

Based on the regulation of MKLH year 2003 of the highest total PAH content from the station above. It showed that it has passed the limit of maximum border required for the concentration of PAH in the water for the living of sea biota, which is of 0.003 ppm, and the content of PAH in the sediment can give side effect toward sea biota which is of 45 ppm [13].

5. Conclusion

The validation method using parameter of linearity, linear range, LoD, and LoQ have complete the requirement of good validation. Recovery test for sediment sample has fulfilled the requirement for all of PAH compounds except for *Naphthalene* and *Phenantrene*. The highest concentration of PAHs is found at station 7 is *Benzo(a)anthracene* (933.2 ± 3.9) ppm and *Perylene* (205.7±19.4) ppm. The

¹ lowest total concentration of PAH compounds was found at station 12 because the station is located at the estuary of Tello River due to dilution from both river and sea water. Source of PAH in sediment sample generally came from petrogenic and pyrolytic process.

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