Aliphatics hydrocarbon content in surface sediment from Jakarta Bay, Indonesia

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Abstract. Sedimentary aliphatic hydrocarbons content have been studied quantitatively and qualitatively using GC/MS method in eight coastal stations located in the Jakarta Bay, North of Jakarta, Indonesia. The total concentrations \( n \)-alkanes have ranged from 480 \( \mu g.kg^{-1} \) to 1,935 \( \mu g.kg^{-1} \) sediment dry weight. Several ratios (e.g. CPI\textsubscript{24-32}, NAR, TAR, Pr/Phy, \( n-C_{17}/Pr \), \( n-C_{18}/Phyt \), \( n-C_{29}/n-C_{17} \), \( \sum n \)-alkanes/\( n-C_{16} \)LMW/HMW, Paq and TMD) were used to evaluate the possible sources of terrestrial-marine inputs of these hydrocarbons in the sediments. The various origins of aliphatic hydrocarbons were generally biogenic, including both terrigenous and marine, with an anthropogenic pyrolytic contribution (petrogenic and biogenic combustion). Two stations (G,H) were the highest concentration and had potential risk to environment.

1. Introduction

Jakarta Bay is located in northern part of Jakarta, Indonesia, which is considered as the most crowded in maritime traffic in Java sea. Jakarta Bay is defined by two flanking delta systems which supply large volumes of sediment from 13 rivers watersheds [1,2, 8]. Several human-related activities may become the pollutant sources i.e. domestic waste, industrial effluent, urban run off, shipping wastes and illegal fishing as well as the oil and gas exploitation activities. The later may occur via oil spills with has a potential hazard to the living biota and ecosystems. We used a group of compound namely aliphatic hydrocarbons i.e. \( n \)-alkane, pristine (Pr) and phytane (Phy) from terrestrial plants or their decomposition and metabolic activity of aquatic organisms [3,4]. Aliphatic hydrocarbons are one of the widespread contaminants in coastal, river, bay and lagoons. They are formed by biological (decarboxylation affecting linear and branched fatty acids), chemical or synthesized process. Taking into account that aliphatic hydrocarbons can enter the marine environment by natural (i.e. pyrolysis, diagenesis and biosynthesis) and anthropogenic processes (i.e. industrial activities, combustion of fossil compounds, oil plants and refineries, oil waste, urban waste, etc.) [3,4,9] source of aliphatic hydrocarbons are multiple and difficult to determine. Such saturated hydrocarbons generally have to range from \( n-C_{12} \) to \( n-C_{40} \) and might have particular predominance number of atoms \( n-C_{15} \) and \( n-C_{17} \) in...
the case of marine while the $n$-C$_{29}$ and $n$-C$_{31}$ in case of terrestrial apportionment. Hence, the objective of this study was to determine of aliphatic hydrocarbons level and potential source/origin of contamination of marine sediment in Jakarta Bay, Indonesia.

2. Materials and Methods

2.1 Study area and sampling

This study focused on the detection of aliphatic hydrocarbon condition in Jakarta Bay. A global positioning systems (GPS, Germin Etrex Summit HC, Kansas City USA) and Quantum GIS (Geographic information system) were used in determining different sampling sites. In December 2013, there are 8 stations of surface sediment were collected from Jakarta bay, 4 stations near from urban and industrial area (A, B, G and H). These stations were canalized from several rivers form an integral part of the stormwater and sewage transport systems within Jakarta’s urban centre [2,3], and 4 stations were characterized by coralline sand substrates with high traffic of shipping transport (C, D, E, and F) (Figure 1). Surface sediment samples were taken using a stainless steel grab sampler, and then placed immediately in an aluminium container and stored in a cool box to minimize microbial degradation during transport to the laboratory. Sediment sample were subsequently freeze-dried (Over on at -55 $^\circ$C and 0.0080 mtorr, Korea), crash it in mortar porcelain then sieved (stainless steel sieved) at 200 μm and homogenised.

2.2 Chemicals

All solvents were of Supra Solv grade (Merck, Darmstadt, Germany) such as n-Heptane (HEPT), Dichloromethane (DCM), HCl (37%) was of pure grade (Fisher, Scientific, Leicestershire, UK). Individual deuterated internal standard of aliphatic hydrocarbons [2H$_{40}$] n-nonadecane, m/z 66 (Sigma-Aldrich, St Louis, MO, USA); 1-eicosene, m/z 71 (Dr. Ehrenstrofer Laboratories, Augsburg, Germany) for $n$-alkanes standard control. Alumina, Silica gel 60 (200-300 mesh) and Cu powder (>230 mesh) were purchased from VWR international and Merck (Darmstadt, Germany).

2.3 Extraction and Separation

Precleaned thimble cellulose extraction was prepared and filled 10 g accurately weighed of freeze-dried sediment then extracted by six-term apparatus for 3 h with a 150 mL mixture of HEPT/DCM (1/1; v/v). Before extraction, 0.5 mL in ternal standard (15 mg/L) was added into sample. The extract

Figure 1. Site description of Jakarta Bay, Indonesia
or Extractible Organic Material (EOM) was evaporated under gentle nitrogen stream and weighed (microbalance Perkin-Elmer AD2Z) part of the EOM was dissolved in HEPT and applied to a 50% silica/50% alumina (8 g of each, both deactivated with 5% H₂O) with chromatography column adsorption, borosilicate glass column (30 m x 1.0cm). The saturated fraction (n-alkanes) was eluted with 30 mL of HEPT and 20 mL of HEPT/DCM (90/10; v/v) on the column then fraction was evaporated on a rotary evaporator under a stream of nitrogen, and dry residues were quantified gravimetrically (microbalance Perkin-Elmer AD2Z, France). Cu powder which had been washed with diluted HCl (1/4; v/v) was added and mixed with extract to remove their sulphur interference. Cu powder residue was removed, and extract solution was diluted in HEPT to 1 mL. The n-alkanes fraction were analyzed by Agilent 7000 A GC/ QQQ-MS (triple quadrupole Gas Chromatography-Mass Spectrometry system, Agilent Technologies, USA) with DB-5MS (30 m x 0.25 mm x 0.25μm) capillary column, nitrogen as carrier gas, temperature programmed from 80°C (2 min) then raised to 300°C (12°C min⁻¹). The mass spectrometer was operated in electron ionization (EI) mode (70 eV) and simultaneously scanned in both Full Scan and Selected Ion Monitoring (SIFI mode). We have analyzed variety of n-alkanes (n-C13-C33) at m/z 71. The n-alkanes were identified by comparison of mass spectra with NB75 mass spectra library and matching their retention times against corresponding individual standard solution (1-ecicosane).

2.4. Quality control
All data were subject to quality and control procedures. Blank HEPT and matrix duplicate samples were analyzed as quality control procedures. One internal standard (n-nonadecane-d₅₀) was added to each sample and matrix blank prior to extraction as surrogates to assess the recovery. n-alkane surrogate recovery was 112%. Individual n-alkanes were quantified using deuterated internal standard (n-alkane d₄₀ m/z 66) with 12 variation concentrations between 0.375-15,000 pg.μL⁻¹ (data not shown).

3. Result and Discussion
The gravimetric data for each fraction are mainly composed of saturated hydrocarbons and the extractable organic matter (EOM) content varied from 980-2,380 sed. dry weight mg.kg⁻¹. The fraction masses were ranged from 680-1,440 sed. dry weight mg.kg⁻¹sed. dry weight. Station E has a large amount of EOM and aliphatic hydrocarbon fractions because physically it is mud sediment and plant. Gravimetric data the extract of aliphatic hydrocarbons have showed in Table 1.

| Table 1. Gravimetric data (mg.kg⁻¹ sed. dry weight) |
|-----------------------------------------------|
|   | St.A | St.B | St.C | St.D | St.E | St.F | St.G | St.H |
| EOM | 1.060 | 1.870 | 1.630 | 1.150 | 2.380 | 980 | 1.683 | 2.240 |
| F1  | 700  | 1.280 | 1.120 | 680  | 1.440 | 620 | 980  | 990  |

EOM = Extractible Organic Materials; F1 = aliphatic hydrocarbons fraction

The distribution spatial of total n-alkanes (n-C₁₃-n-C₃₃) in aliphatic hydrocarbon fraction ranged from 480 to 1934 sed. dry weight μg.kg⁻¹(Figure 2, and Table 2). The highest concentration was station H, which site near from urban area and from two main watershed inputs (Figure 1). Contamination levels found in sediments are lower than those measured in previous studies such as in Gulf of Fos (French Mediterranean Cost), (Σn-C₁₄-₃₂: 548-4794 μg.kg⁻¹ sed.dw) [10] Arc River and BerreLagoon, Western Mediterranean Sea (Σn-C₁₃-₄₄: 563-5068 μg.kg⁻¹ sed.dw)[4], Huveaune river, France (Σn-C₁₄-3₄: 183.6-236780.4 μg.kg⁻¹ sed.dw) [15] Cretan island, Greece, Eastern Mediterranean Sea (Σn-C₁₃-₄₄: 326-3758 μg.kg⁻¹ sed.dw) [5] and coast of Aliaga, Turkey (Σn-C₁₁-₃₄: 340-56480μg.kg⁻¹ sed.dw) [16]. In America, fishing village-Z3. Patos lagoon, Pelotas region, Brazil (Σn-C₁₄-₃₄ 337.6-5868.9 μg.kg⁻¹ sed.dw) [17] In Asia, yellow sea (Huanghai) Northwestpacific ocean, China (Σn-C₁₄-
33:700-15800 μg.kg\(^{-1}\) sed.dw) [18] and Segara Anakan lagoon, Cilacap, Indonesia, total of \(n\)-alkanes (\(\sum n-C_{13-34}\) ranged from 2786 to 129027 μg.kg\(^{-1}\) sed.dw) [9].

3.1 The possible source
To assess the possible source of saturated hydrocarbons or \(n\)-alkanes in the marine environment are complex and of diverse origins. Several criteria are used to determine the probability and to assess the possibility of their origins (anthropogenic or biogenic) in reserve sediment [15 and 17]. We calculated 9 ratios: CPI (\(n-C_{24-n-C_{33}}\), \(n-C_{17}/Pr\), \(n-C_{18}/Phy\), Pr/Phy, \(n-C_{25}/n-C_{17}\), Terrigenous/aquatic ratio (TAR)[3,5], Natural Alkane Ratio (NAR) [3,5,9,10,15 and 17], \(\sum n\)-alkanes/\(n-C_{16}\), TMD (Terrestrial Marine Discriminant) and Proxy ratio (Paq) [9,14]. Proxy ratio (Paq) indicate the presence of macrophyte inputs from terrestrial plants [9,13]. Values < 0.1 proposed non-emergent plant input characteristic. The values are between 0.1 to 0.4 indicate emergent macrophytes. In the other hand, those from 0.4 to 1.0 indicate submerged/ floating macrophytes. The result showed that 5 stations (C,D,E,G,H) were characterized by emergent macrophytes input while 3 other stations (A,B and F) were submerged/ floating macrophyte input. This ratio is characteristics for mangroves vegetation and their association with coastal ecosystems [9].

The Carbon Preference Index (CPI) which indicate a predominance of odd numbered \(n\)-alkanes in the range \(n-C_{24}\) to \(n-C_{32}\) or range \(n-C_{25}\) to \(n-C_{33}\) are characteristics of terrestrial higher plant debris to the shelf sediments [3,17]. This ratio also was used to assess the relative inputs from biogenic and petroleum-related sources [7]. CPI values close to one also indicate from marine microorganisms and/or recycle organic matter, another hand these values also indicate \(n\)-alkanes source from fossil fuels, including petroleum sources \(n\)-alkanes from biogenic sources (land plant material) have CPI close to 5-10 (station H). All station except station H were significantly higher than 1 (from 1.26 to 3.24) which were terrigenous inputs [11,12]. Our finding is in accordance with the previous study that showed Jakarta Bay was affected by the contribution of petrogenic sources and these petrogenic inputs might be related to the extensive use of fossil fuels to increase of industry and motor vehicle in Jakarta [1]. The presence of short odd alkanes particularly \(n-C_{17}\) is indicative of biogenic marine organisms (algae, plankton, marine animals, etc.) \(n-C_{17}/Pr\) and \(n-C_{18}/Phy\) ratios: these ratios are used to evaluate the presence of oil and the relative biodegradation of \(n\)-alkanes [9,10]. Pristane (Pr) and Phytane (Phy)
are indices of biogenic origin and consist of naturally hydrocarbons present in zooplankton, algae or bacteria. However, the presence of oil can be either biogenic or diagenetic in origin [5,6]. $n$-$C_{18}$/Phy ratio ($>1$) found in all stations that confirm oil input and lower than one confirm such selective biodegradation phenomena [5] $n$-$C_{29}$/n-$C_{17}$ ratio: $n$-$C_{29}$ is abundant in land plants, and $n$-$C_{17}$ is prominent in marine organisms, the ratio of these two alkanes reflects the relative contributions [4].

The NAR (Natural n-Alkane Ratio) represents the proportions between natural and petroleum long chain n-alkanes [3,4,9,10]. This ratio is close to zero for petroleum hydrocarbons and crude oils terrestrial plants or marine plants source for the value close to one [2,4]. For all stations, this ratio ranges between 1.45 and 1.93 indicate from terrestrial inputs. The TAR (Terrigenous Aquatic Ratio) represented a crude indicator of the relative strength of terrestrial versus aquatic sources [3,5,9] this ratio between the concentration of long-chain n-alkanes ($n$-$C_{27}+n$-$C_{29}+n$-$C_{31}$) to short-chain n-alkanes ($n$-$C_{15}+n$-$C_{17}+n$-$C_{19}$). This ratio was found below 1 for most of the stations except for stations G (1.66) and H (6.96) which can be assessed as predominant terrestrial input. (Table 2). The LMW/HMW (Low molecular weight (sum of $n$-$C_{14}$ to $n$-$C_{20}$) / High molecular weight (sum of $n$-$C_{21}$ to $n$-$C_{34}$) ratios is indicated n-alkanes from petrogenic and plankton sources, if this value close to 1 and < 1 usually represent biogenic source which produced by plants, marine animals and sedimentary bacteria [4,14]. This ratio also indicates the presence of fresh oil in sediment for the value >2 [13].

For Jakarta bay, the station A,B,C,D,E,F are higher than 1 confirm essentially from anmix between natural and petrogenic source. Then for station G and H confirm the biogenic source. $\sum n$-alkanes/n-$C_{16}$ ratio indicated a biogenic input for value >50 and whereas values < 15 indicate petroleum-contaminated samples [9]. Two stations (Station A and H) characterized mix between biogenic and petroleum input. TMD (Terrestrial Marine Discriminant) is indicated from terrestrial and marine sources with a value 1. A value $>1$ a dominant terrestrial input (Stations G and H), whereas a value <0.5 confirm dominant marine contribution (Stations A,B,E and F). A value between 0.5 and 1 indicate mixed terrestrial and marine input (Stations C and D). Moreover, TMD confirmed the n-alkane from vascular plants and phytoplankton, same function like TAR index [9,14].

**Table 2.** Concentration ($\mu$g.kg$^{-1}$ sed.dry weight) of n-alkanes, pristane and phytane in sediment samples from Jakarta Bay (Stations A to H)

| Composes                   | St.A | St.B | St.C | St.D | St.E | St.F | St.G | St.H |
|----------------------------|------|------|------|------|------|------|------|------|
| $\sum n$-alkanes ($n$-$C_{13}$-$C_{33}$) | 479.97 | 564.75 | 752.30 | 572.35 | 635.39 | 602.75 | 1178.86 | 1934.52 |
| $n$-$C_{17}$/Pr             | 0.78  | 1.09  | 0.72  | 0.97  | 1.10  | 1.04  | 1.06  | 1.08  |
| $n$-$C_{19}$/Phy            | 1.09  | 1.29  | 1.16  | 1.50  | 1.24  | 1.95  | 1.24  | 1.30  |
| CPI$_{34-32}$              | 1.78  | 1.26  | 2.08  | 1.87  | 1.88  | 1.96  | 3.24  | 5.01  |
| $n$-$C_{29}$/n-$C_{17}$     | 2.74  | 5.71  | 1.81  | 1.93  | 3.89  | 2.56  | 0.82  | 0.21  |
| Pr/Phy                    | 0.91  | 0.82  | 0.81  | 0.93  | 0.96  | 0.83  | 1.09  | 0.93  |
| NAR                       | 1.83  | 1.87  | 1.78  | 1.93  | 1.73  | 1.82  | 1.61  | 1.45  |
| TAR                       | 0.50  | 0.27  | 0.77  | 0.95  | 0.39  | 0.47  | 1.66  | 6.96  |
| Pr/Phy                    | 0.91  | 0.82  | 0.81  | 0.93  | 0.96  | 0.83  | 1.09  | 0.93  |
| TMD                       | 0.46  | 0.30  | 0.76  | 0.94  | 0.42  | 0.46  | 1.57  | 5.78  |
| Paq                       | 0.44  | 0.51  | 0.35  | 0.30  | 0.29  | 0.44  | 0.21  | 0.12  |
| $\sum n$-alkanes/n-$C_{16}$ | 22.20 | 16.55 | 14.11 | 10.30 | 15.41 | 11.25 | 28.61 | 52.87 |
| LMW/HMW                   | 1.45  | 1.98  | 1.18  | 1.29  | 1.84  | 1.71  | 0.59  | 0.20  |

Pr=Pristane, Phy=Phytane, CPI (Carbon Preference Index) as $2^*(n$-$C_{25}+n$-$C_{27}+n$-$C_{29})/(n$-$C_{27}+2^*(n$-$C_{29}+n$-$C_{31}+n$-$C_{33}))$, NAR (Natural n-Alkanes Ratio) calculated as ($\sum (n$-$C_{19,33})$)-$2^*$($\sum (n$-$C_{20,32})$)/$\sum (n$-$C_{19,33})$, TAR (The terrigenous/aquatic ratio) calculated as ($n$-$C_{27}+n$-$C_{29}+n$-$C_{31})/(n$-$C_{25}+n$-$C_{27}+n$-$C_{31}+n$-$C_{19}$), LMW/HMW ($\sum (n$-$C_{14}$ to $n$-$C_{20}$)/ $\sum (n$-$C_{21}$ to $n$-$C_{34}$)), TMD (Terrigenous Marine Discriminant) : ($n$-$C_{25}+n$-$C_{27}+n$-$C_{29}+n$-$C_{31}+n$-$C_{19}$) / ($n$-$C_{15}+n$-$C_{17}+n$-$C_{19}+n$-$C_{21}+n$-$C_{23}$), Paq (Proxy ratio) : ($n$-$C_{23}$+$n$-$C_{25}$)/($n$-$C_{25}+n$-$C_{27}+n$-$C_{29}$+$n$-$C_{31}$).
GC chromatograms showed the series of resolved compounds as a sign of petroleum input and/or the presence of biodegraded complex mixture of hydrocarbons, mainly \( n \)-alkanes and some branched alkanes called Unresolved Complex Mixture (UCM). It is amount coelution of structurally complex isomers and homologues of branched and cyclic hydrocarbons. Figure 2 was showed the concentration of \( n \)-alkanes in surface sediment from Jakarta Bay. The UCM ranging from \( n\)-C\(_{13}\) to \( n\)-C\(_{34}\) were observed in bimodal pattern in the most station except for station H. Such bimodal UCMs indicated the presence coexistence between a recent and an old pollution.

**Figure 2.** Capillary column gas chromatograms of \( n \)-alkanes fractions in station A (above) and Station H (under), Jakarta Bay, Indonesia
Capillary column gas chromatograms profiles (Figure 2) showed Station A indicate the profile n-alkanes from petroleum input with dominant compounds found in the whole sediments are n-C_{17}, n-C_{18}, Pr, Phy, n-C_{19} and n-C_{20}. On the other hand, station H has predominant intensity for n-C_{27}, n-C_{29}, n-C_{31} and n-C_{33} which can be assessed as terrestrial input. Interestingly, we clearly demonstrated the important extent of n-C_{33} that refers mangroves leaf fingerprint [9].

4. Conclusion
The total n-alkanes (n-C_{13}-nC_{33}) concentrations ranged from 480 to 1,934 μg.kg^{-1} sediment dry weight (dw). The ratios of specific n-alkanes(n-C_{17}/Pr, n-C_{18}/Phy,n-C_{17}/n-C_{29}, Pr/Phy, TAR, \sum n-alcanes/n-C_{16}, NAR, LMW/HMW, CPI_{27-34}, Paq and TMD index) showed that the hydrocarbons source in the sediments were generally petrogenic and terrigenous input (in the case of G and H). These data can use baseline reference and evaluating recent environmental condition in this site for future efficient environmental management.

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