Distribution of aliphatic and polycyclic aromatic hydrocarbons (PAHS) in coastal sediment from the Ngoua River (Douala, Cameroon)

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Abstract: This paper reports the identification and quantification of 15EPA-PAHs in the sediments samples collected in Ngoua River, Douala, Cameroon. PAH and *n*-alkanes were separated by using a chromatographic column, identified and quantified by using Gas Chromatography (GC/FID). The total concentration ranged from 140.42 µg/g to 229.47 µg/g dry matters during the rainy season and from 48.89 µg/g to 333.49 µg/g dry matters during the dry season. All samples showed the predominance of high Molecular Weight (HMW) PAHs due to industrial activities. At the upstream, the concentrations of PAHs were systematically lower than those found downstream indicating an influence of Nettoycam Company in the local pollution by PAHs. Principal component analysis (PCA) also showed remarkable differences of characteristics of samples collected upstream and downstream of Nettoycam Company, samples location and also of samples from rainy and dry seasons.

Keywords: Environmental Pollution, Hydrocarbons, Rivers, Sediment, Pahs

1. Introduction

Hydrocarbons compounds occurring are naturally and are important components of organic matter from marine coastal sediment. The composition and distribution are significantly affected by human activities, particularly in recent decades. They are an assemblage of substances coming from biogenic, diagenic, petrogenic and/or pyrogenic sources [23],[10] and have received much attention due to their ubiquitous distribution in marine and river sediments [21]. PAHs have environmental significance due to their potential toxicity to organisms. They are often hydrophobic and easily accumulated in sediment, organisms through food chains, and present a potential threat to aquatic ecosystems and even to human health [18].

Sediments are recognized as excellent sinks for pollutants such as hydrocarbons [16], and constitute an appropriate matrix for chemical analysis [12].

A pool of aliphatic diagnostic indices and more particular signatures can be used to identify biogenic terrestrial or marine or anthropogenic sources. PAHs distributions are also useful to differentiate the PAHs main sources, particularly to distinguish petrogenic from pyrogenic hydrocarbons [2].

In Douala (Cameroon) several coastal areas are suffering from the anthropogenic input due to the industrial and economic activity, such as the Ngoua River located in the industrial zone of Bassa (Douala) in the neighborhood of Nettoycam industry. This industry is specialized in treatment of hydrocarbons wastes. Many years ago, the principal economical activity of this River was fishing. But the intensification of industrial activity is responsible of the disappearance of fishes and shrimps [9]. Today the only activity of this River is wood selling and sand extraction by children especially during holydays in dry season.

A study carried out by Baok (2007) and by Rharrassin (2008) reported this river as the most polluted by microbiological and inorganic element (Cu, Ni, Pb). Nevertheless the organic pollution such as hydrocarbons is never study in this area. This work provides a more complete study of the industrial pollution of this River and the first to investigate the composition, origin and distribution of aliphatic and PAHs in

sediment in this River. The PAHs concentrations and distribution are discussed in terms of sampling location, seasonal variation, origin and sources.

2. Materials and Methods

2.1. Study Areas and Sampling Sites

Ngoua River is one of long river of Douala (Cameroon) located in the industrial zone of Bassa. This River has about 7 km of coast. Two series of sediment were used in the present study. The first series of four sites were taking in the dry season and the four others sites were taking in the raining season. In each sampling site, sediment were taken in eight adjacent point selected randomly, homogenized in a single sample (1kg), was kept in the laboratory for analysis. Samples of surface sediments were collected at stations along the coastal areas of Ngoua River as depicted in the map of the study area (Figure-1). The sampling campaign was conducted at 4 sites during August 2010 (raining season) and February 2011 (dry season). A global positioning system (GPS) was used to pinpoint sampling locations (Table 1).The sediments were dried in the open air, crushed and sieved and 2 mm diameter fraction was used for further analysis. They were subsequently kept in a refrigerator at -20^0C prior to analysis.

Table 1. *Location and characteristics of sampling sites.*

Names of station	Latitude	Longitude	Depth (m)
After Nettoycam industry (N1)	04°01.571'N	009°43.560'E	4
In the neighborhood of Nettoycam (N2)	04°00.878'N	009°44.104'E	8
Song-Mahop (N3)	04°00.557'N	009°44.304'E	5
Village-shell (N4)	04°00.535'N	009°44.223'E	6

2.2. Sample Extraction and Cleanup

The extraction procedure used for sediment samples is described in the literature [4]. Sediments were soxhlet extracted with 25 mL of methanol for 2h. The extract was concentrated and solvent-exchanged to hexane using a rotary evaporator. The hexane extract was subject to a 1:2 alumina/silica gel glass column for cleanup and fractionation. The column was eluted with 15 mL hexane and the eluted was discarded. The *n*-alkane fraction was eluted with 10 ml of hexane, the second fraction containing the mixture of *n*-alkane and PAH was eluted with 10 ml of hexane/ethyl acetate mixture (83:17, v/v) and the third fraction containing only the PAHs was eluted with 20 mL of hexane/ethyl acetate mixture (65:35, v/v).

2.3. Analysis for Hydrocarbons Identification and Quantification
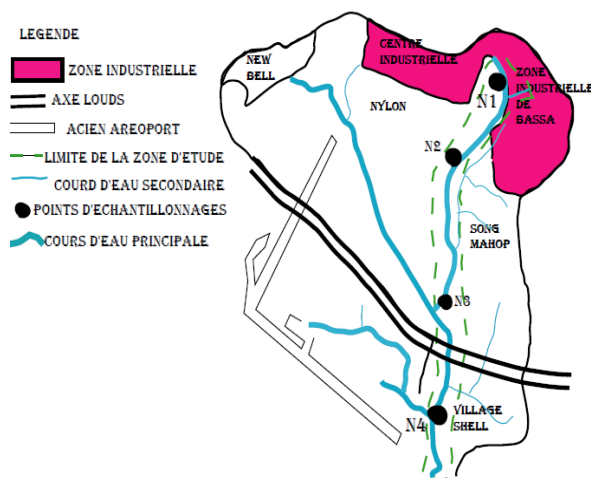

Fig 1. Samples location of sediments in Ngoua River

PAHs and *n*-alkanes concentrations were determined using the SHIMADZU GC-14B gas chromatograph (GC) equipped with a flame ionization detector (FID) and a Hewlett Packard integrator. The GC-FID was using a ZB-5 capillary column of 30m length, 0.32mm film thickness, and 0.25μm of internal diameter (US PATENT); Varian, Walnut Creek, CA, USA). Nitrogen (99.999%) was used as the GC carrier gas at a constant flow of 1.0 ml/min^{-1}. A microsyringe was used to inject 1-μL samples into the chromatograph. The column temperature was programmed from 40^0C (5min) at 320^0C (45min) at 6^0Cmin^{-1} and identification using standards with their Kovats Index (IK).

3. Results and Discussions

3.1. N-Alkane Distribution and Sources

Normal n-alkanes in the range C_9-C_{28} was found but only C_{12}-C_{28} was present in all samples. The total n-alkanes concentrations (TNA) ranged from 144,23 μgg^{-1} to 1567.21 μgg^{-1} in the rainy season and from 105.64 μg.g^{-1} to 2670.73 μg.g^{-1} in the dry season, with a highest concentration at N2 (2670.73 μgg^{-1}). The concentrations of all n-alkanes decreased from dry to rainy season (February to August) possibly due to the rainy period and to the consequent increase of river water flow and volume that led to n-alkanes dilution. The results of our study show the predominance of n-alkanes C_{19}-C_{28} in the rainy season and C_{13}-C_{28} in dry season with the predominance of C_{20}-C_{28}. This result was attributed to mixed origin of biogenic, bacteria and anthropogenic organic source inputs. For a better understanding of the principal sources of sediments contamination the determination of the carbon preference index (CPI) and the ratio of n-C_{17} / Pristane and n-C_{18} / Phytane was calculated. The CPI (carbon preference index), a measure of biologically synthesized n-alkanes[26, 27], indicates the relative contributions of n-alkanes from natural (biogenic/terrestrial; CPI > 1) compared to anthropogenic (CPI< 1) sources. The (CPI) was calculated according to [28] using the same odd-carbon and even-carbon number n-alkane

concentrations in the respective samples as follows:

$$CPI = (C23 + C25 + C27 + C29 + C31)/(C24 + C26 + C28 + C30)$$

Values of CPI ranged from 0.07-1.48 for the entire stations, with maximum value (1.48), indicating a relatively higher proportion of n-alkanes from anthropogenic (mostly petroleum) sources

3.2. PAHs Distribution and Sources

3.2.1. PAHs Distribution

The total concentration ranged from 140.42 $\mu g.g^{-1}$ to 229.47 $\mu g.g^{-1}$ dry weights during the rainy season and from 48.89 $\mu g.g^{-1}$ to 333.49 $\mu g.g^{-1}$ dry weight during the dry season, with a highest concentration at N2 (333.49 $\mu g.g^{-1}$).). Sites N2 and N3 were both located at the Nettoycam industry, present the highest level of PAHs during all season (Figure 2). This concentration is probably due to the recent deposition and accumulation of PAHs in the sediments. Compared to the low PAHs concentrations in sediment at site N1 and N3 a high level of PAHs was found at the site N2 and N4, which can be attributed to recent PAHs input around the area.

In the 16 PAHs repertory by European and American lists (U.S. Environmental Protection Agency, EPA), for their cancerogenicity and toxicity, 15 PAHs were found to be present in this study. For the individual distribution, the PAHs with high molecular weight are the predominate in the two season (raining and dry season).The PAHs distribution was mostly dominated by high molecular weight PAHs (4-, 5- and 6-rings) such as Fluoranthene, pyrene, Benzo[a]anthracene, Chrysene, Benzo[a]pyrene, Indeno[1,2,3-cd]pyrene, Benzo[ghi]perylene with the high concentration for Benzo[ghi]perylene (101,02 μgg^{-1}).

The higher concentration of Benzo[ghi]perylene (101,02 μgg^{-1}) is probably due the presence of the 6 rings in the structure. In the literature the biodegradability and bio-disponibility are function of the number of ring [23]. In this case, the PAHs with high molecular weight are not more hydrophilic like the 2-3 rings and the dissolution in the water is very difficult. This reason can be explain the concentration of this type of PAHs in the sediment.

The presence of the 2- and 3-ring at site N4 in the dry and rainy season is probably due to the presence of industrial activity such as petrol station located before of the site N4. These low PAHs concentration is because 2-and 3-rings PAHs are not strongly adsorb onto the surface of suspend and fine particles and they also have easily dissolution in water.
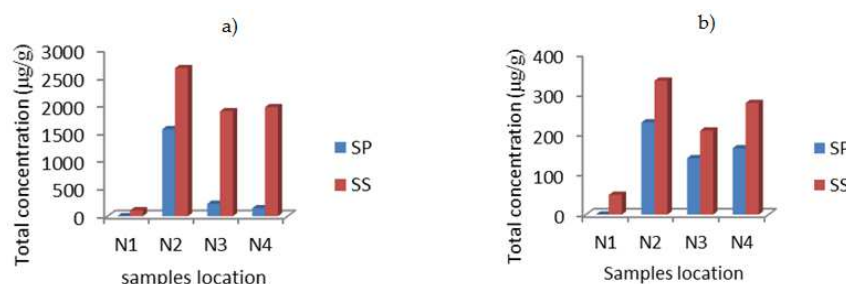

Fig 2. Total concentration of n-alkanes (a) and PAHs (b) in the Ngoua River sediment; SS: Dry season, SP: rainy season.

For the individual PAHs the highest concentration is from Dibenzo[agh]pyrelene (101,02 μgg^{-1}) at site N4 in dry season and the lowest concentration is from Acenaphtene (2,25 μgg^{-1}) in dry season. This result demonstrated that the HMW because of the composition have easily accumulation in the some matrix such as sediments, sands, suspend particles. Beyond this, some authors have also investigated the importance of high molecular weight PAHs [1],[15], [20] in many rivers likes Ushuaia Bay, Blanca Bay, and the Cienfuegos Bay (Cuba) (Table 2).

Table 2. Total concentration of PAHs in sediments of some rivers in the words.
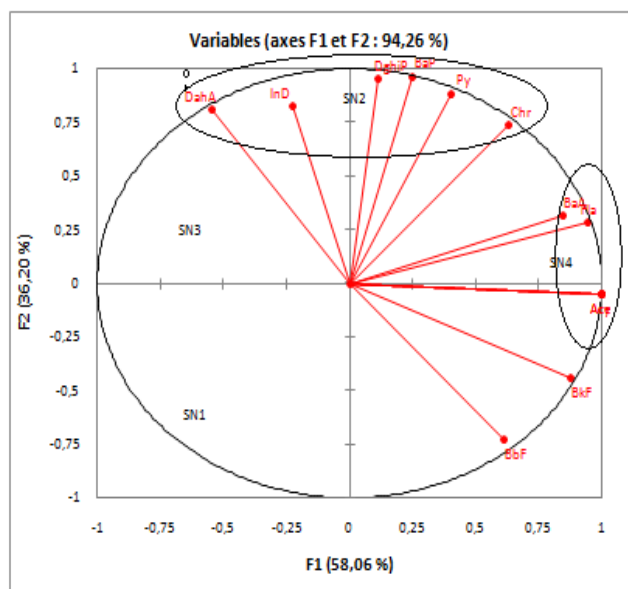
Location	Na	ΣPAHs (µg/g)	UCM (µg/g)	Reference
Ngoua River (Cameroun)	15	48.98 – 333.49	132.21- 2763,23	This study
Bahía Ushuaia, Patagonia	10	Nd - 0.360	4 -1050	Marta et al. (2012)
Bahía Blanca Estuary	24	0.015 - 0.010		Arias et al. (2010)
Marseille Bay (France)	11	0.034 - 1.826		Asia et al. (2009)
Daya Bay, South China	14	0.0425 - 0.156		Yan et al. (2009)
Abu Qir Bay, Egypt	20	<MDL- 2.660		Khairy et al. (2009)
Gulf of Aden, Yemen	17	0.022 - 0.604		Mostafa et al. (2009)
Cienfuegos bay, Cuba	17	0.450-0.010		Tolosa et al. (2009)
Dialo Estuary River China	16	0.272 -1.606		Bin et al. (2009)
Bahía Nueva, Patagonia	12	0.010 -7.690	0.01-8.53	Massara et al. (2008)
Eastern Aegean Sea, Izmir Bay	16	0.0025 - 0.113		Darilmaz and Kucuksezgin (2007)
Bonny River (Nigeria)	24	2.13-16.72(mg/kg)		Bassey et al. (2012)
Patagonia Coast	86	0.010 - 7.690	Nd-1195	Commendatore and Esteves (2007)
Pearl River Estuary	9	0.189 - 0.6367		Luo et al. (2006)

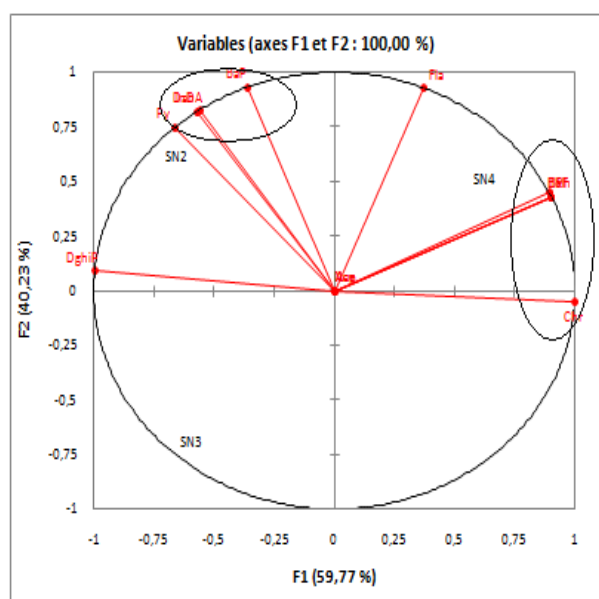Na: Number of PAH compounds analyzed in each study.

Nd: not detected by the methods.

According to Dzalla (2002) the pollution of this river especially by PAHs must be taken seriously otherwise the population going to be affected. Seven of the examined PAHs (i.e., ., Benzo[a]anthracene, Benzo[b]fluoranthene, Benzo[k]fluoranthene, Benzo[a]pyrene, Indeno[1,2,3-cd]pyrene, Dibenzo[ah]anthracene, and Benzo[ghi]perylene), which are known as carcinogens, were found in Ngoua River and represent 54 and 82% of the total concentration. This result can explain the disappearance of fishes and shrimps which represent the main revenue of the population of that area.

3.2.2. PAHs Sources

a) Ngoua dry season

b) Ngoua rainy season.

Fig 3. *PCA loading plot sediment from the Ngoua River,.*

Several molecular ratios of selected PAH compounds, such as the abundance ratio of 2–3-ring hydrocarbons to 4–6-ring hydrocarbons (LMW/HMW), Phen/Ant, Fl/Pyr, Fl/(Fl + Pyr), InP/(InP + BgP), have been developed to aid in the interpretation of PAH compositions and in their possible sources (Doong and Lin, 2004). A Fl/Fl + Pyr ratio <0.40 indicates petroleum, 0.40–0.50 indicates petroleum combustion, and >0.50 indicates the combustion of coal, grasses and wood (Doong and Lin, 2004). Value of Fl/Fl + Pyr (0.2) < 0.40 confirm petroleum pollution. PCA is a statistical tool that resets large amounts of data and facilitates visualization of similarities and differences between data sets [11]. In this research, PCA was used to better understand of PAHs sources in the Ngoua River and seasonal variation of concentration. PCA loading plots for sediment contamination and seasonal variation are shown in Figure 2. The two principal group of compounds was concentrated in site N2 and N4, which the mean concentration of N2. This group was constituted of PAHs (i.e., Benzo[a]anthracene, Benzo[b]fluoranthene, Benzo[k]fluoranthene, Benzo[a]pyrene, Indeno[1,2,3-cd]pyrene, Dibenzo[ah]anthracene, Benzo[ghi]perylene), which are known as carcinogens.

This demonstrates that sediments contaminants from the Ngoua River Estuary are a mixture of petrogenic (3-ring) and pyrolytic-derived (4- and 5-ring) PAHs. The high loading of site N2 indicates that the Nettoycam industry can be the main source of PAHs in sediment for the Ngoua River Estuary. Therefore, negative toxic effects exist for the Ngoua River, and they may be very significant.

4. Conclusion

This study has provided important data on parent PAHs levels in sediments of the Ngoua River, which is located in the industrial area in the neighborhood of the Nettoycam industry of Cameroon. The PAHs composition in the four sites examined was comprised mainly of 4- and 5-ring PAHs. This indicates a relatively recent local source and was attributed to the petrogenic source of PAHs. The PCA show the relation of sites location, sources and seasonal variation of this contamination. This result demonstrates that sediments were contaminated and this contamination was probability due to the Nettoycam industry. But to confirm this assessment, the samples most be collected inside the industry.

Acknowledgment

Table 3. *Concentration of n-alkanes in sediments (µg/g) from Ngoua River.*

Compounds	Ngoua sediments.							
	N1		N2		N3		N4	
	Rn	Dry	Rn	Dry	Rn	Dry	Rn	Dry
Nonane (C9)	nd	nd	nd	nd	nd	nd	nd	nd
Decane (C10)	nd	nd	nd	nd	nd	nd	nd	nd
Undecane (C11)	nd	nd	nd	nd	nd	nd	nd	nd
Dodecane (C12)	nd	nd	nd	11.46	nd	9.25	nd	10.45
Tridecane (C13)	nd	7.65	nd	18.56	nd	10.34	nd	11.96
Tétradecane (C14)	nd	8.72	nd	23.67	nd	15.13	nd	15.51
Pentadecane (15)	nd	6.91	nd	12.93	nd	10.38	0.02	12.21
Hexadecane (C16)	nd	nd	nd	19.52	nd	16.52	0.87	14.98
Heptadecane (C17)	nd	6.74	nd	13.23	4.09	9.05	4.48	10.67
Pristane	nd	4.07	nd	12.67	2.03	11.67	1.67	9.89
Octadecane (C18)	nd	nd	nd	14.03	8.23	12.23	9.74	9.67
Phytane	nd	nd	nd	16.89	9.56	14.33	3.67	11.67
C18/Phystane	nd	nd	nd	0.83	0.86	0.85	2.65	0.82
Nanodecane (C19)	nd	6.04	0.99	37.64	nd	12.43	2.44	21.21
Eicosane (C20)	nd	nd	45.67	39.01	13.43	20.67	22.63	24.42
Heneicosane (C21)	nd	nd	1.94	13.36	nd	9.94	nd	9.31
Docosane (C22)	nd	nd	3.61	14.85	nd	10.62	4.62	10.28
Tricosane (C23)	nd	nd	1.24	16.32	nd	10.23	4.89	10.58
Tetracosane (C24)	nd	nd	462	25.05	9.64	21.56	6.72	14.56
Pentacosane(C25)	nd	nd	6.38	41.97	8.27	10.34	9.11	14.29
Hexacosane (C26)	nd	6.71	8.67	10.94	9.75	13.78	12.12	9.67
Heptacosane (C27)	nd	8.67	18.94	11.35	9.91	10.56	10.61	12,38
Octocosane (C28)	nd	6.12	9.32	10.88	8.56	17.73	7.73	21.91
Σ of n-alkanes	nd	61.63	101.38	363.33	83.47	246.76	101.32	255.62
UCM	nd	105.64	1567.21	2670.73	223.34	1892.62	144.23	1967.65

Rn: Rainy season; Dry: Dry season; N: Ngoua sediments ; Nd : no detected
LMW / HMW, low molecular weight / high molecular weight; UCM: Unresolved Complex Mixture

Table 4. *Concentration of PAHs in sediments (µg/g) from Ngoua River.*

Compounds	Ngoua sediments							
	N1		N2		N3		N4	
	Rn	**Dry**	**Rn**	**Dry**	**Rn**	**Dry**	**Rn**	**Dry**
Naphtalene	nd	nd	nd	nd	nd	nd	nd	nd
Acénaphtylene	nd	nd	nd	nd	nd	nd	nd	2.25
Acénaphtene	nd	nd	nd	nd	nd	nd	nd	3.05
Fluorene	nd	nd	nd	nd	nd	nd	nd	3.65
Phenanthrene	nd	nd	nd	nd	nd	nd	8.67	19.02
Anthracene	nd	nd	nd	nd	nd	nd	nd	5.02
Σ of (LMW)	nd	nd	nd	nd	nd	nd	8.67	32.99
Fluoranthene	nd	5.45	17.85	21.17	9.91	10.32	21.34	47.15
Pyrene	nd	5.64	31.58	37.20	24.57	18.42	26.34	30.61
Benzo[a]anthracene	nd	nd	7.78	4.99	7.67	9.83	12.45	16.17
Chrysene	nd	nd	9.56	12.96	10.57	12.16	11.67	16.91
Benzo[b]fluoranthene	nd	4.25	nd	nd	nd	nd	3.10	4.89
Benzo[k]fluoranthene	nd	5.02	nd	nd	nd	nd	3.75	11.51
Benzo[a]pyrene	nd	5.61	14.56	36.71	9.62	22.76	12.42	27.15
Indeno[1,2,3-cd]pyrene	nd	11.23	39.40	56.45	16.74	15.31	24.88	17.28
Dibenzo[ah]anthracene	nd	11.29	28.96	62.99	10.67	30.41	17.43	4.67
Benzo[ghi]perylene	nd	nd	79.78	101.02	50.67	89.56	23.16	68.67
Σ of (LMW)	nd	48.89	229.47	333.49	140.42	208.77	156.54	244.86
LMH/LMW	nd	nd	nd	nd	nd	nd	0.05	0.13
UCM	nd	132.21	1984.32	2763.23	1056.49	1563.67	1342.11	2321.53
Σ of 16PAHs	nd	48.89	229.47	333.49	140.42	208.77	165.21	277.85

Rn: Rainy season; Dry: Dry season; N: Ngoua sediments ; Nd : no detected
LMW / HMW, low molecular weight / high molecular weight; UCM: Unresolved Complex Mixture

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