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Total and Polyaromatic Hydrocarbons in Water, Sediment, Fin and Shellfishes from Badagry Creek and Ologe Lagoon, Lagos, Nigeria

¹ADERINOLA, OJ; ^{2*}MEKULEYI, GO; ²WHENU, OO

¹Department of Zoology and Environmental Biology, ²Department of Fisheries, Lagos State University, Nigeria *Correspondence:gabrielmekuleyi@gmail.com; Toyinakinmoorin@gmail.com

ABSTRACT: The concentrations of eleven (11) polyaromatic hydrocarbons (PAHs) and total hydrocarbons (THC) were investigated in the water, sediment, *Chrysichthys nigrodigitatus* and *Macrobrachium macrobrachion* from Badagry Creek and Ologe Lagoon in Lagos, Nigeria using EPA and Gas Chromatography (GC) methods. All the samples(except water measured in mg/l) were measured in $\mu g/g$. PAHs were not significantly different(p>0.05)across the two stations and were within WHO recommended limit. The highest concentration of THC from Badagry Creek was recorded in *M. macrobrachion* (219.565±171.891 $\mu g/g$) and this value exceeded WHO recommended limit. Similarly, the highest THC (211.565±127.923 $\mu g/g$) recorded in *M. macrobrachion* above WHO limit. The study has shown that the polyaromatic hydrocarbons were below the risk level, which indicated no risk status from the consumption of the fish species studied. However, *C. nigrodigitatus* and *M. macrobrachion* studied from these water bodies are highly contaminated with THC. Therefore, persistent monitoring and strict adherence to responsible waste discharge should be upheld by all industries near these waters in order to avoid deleterious effects on the biota as well as ensuring safety of the consumers.

DOI: https://dx.doi.org/10.4314/jasem.v22i5.10

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Dates: Received: 04 February 2018; Revised: 10 April: 2018; Accepted: 22 April 2018

Keywords: Concentrations, Hydrocarbons, Fish, Safety

Polycyclic aromatic hydrocarbons (PAHs) are a class of ubiquitous organic compounds consisting of two or more fused aromatic rings. They are mostly hydrophobic and are capable of bio-accumulating in animal and human tissues (Zheng et al., 2007). Polycyclic aromatic hydrocarbons (PAHs) are classified as persistent organic pollutants commonly occurring in the environment and are considered to be one of the most difficult organic contaminants to treat (Edwards, 1983; Cerniglia, 1992; Weissenfels et al., 1992; Zheng et al., 2007). Due to their toxic, mutagenic and carcinogenic properties, they pose a significant environmental risk to public health (Chen and Liao, 2006). Sixteen (16) PAHs have been identified as priority pollutants by both the US Environment Protection Agency (USEPA) and the European Union (USEPA, 1977; European Union, Commission Recommendation, 2005). Generally, the presence of PAHs in the environment has increased over the last 100 years; however, global concentrations may have stabilized due to recent air and water quality regulations (Fernandez et al., 2000). PAHs concentrations in the environment are often closely related to local and regional sources, although remote areas can be sites of PAHs deposition through atmospheric processes and long range transport.

Ologe Lagoon and Badagry Creek are very important water bodies in Nigeria. Also, *Chrysichthys nigrodigitatus* and *Macrobrachium macrobrachion* are important components of the diet of the people of Lagos and its environs because they are available all year round. Therefore, the main aim of this study was to assess the level of polyaromatic hydrocarbons and total hydrocarbons in the water, sediment and fish species from Ologe Lagoon and Badagry Creek with regards to human health.

MATERIALS AND METHODS

Study Area: Badagry Creek and Ologe Lagoon are part of the continuous system of Lagoon and creeks along the coast of Nigeria. Badagry creek, the larger of the two sampling water bodies lies within longitude2°42'E and 3°42'E and stretches between latitude 6°22'N and 6°42' N, sharing boundary with Republic of Benin., River Yewa, Bawa and Doforo creeks also empties into it. It is about 60km long and 3km wide. Its depth ranges between 1-3m. Ologe Lagoon on the other hand is situated adjacent to Badagry creek between latitude 6°26'N and longitude 3001'E to 3° 07' E. It has a surface area of 9-42km² and is the smallest of the Lagoons in south western Nigeria. It receives water from Rivers Owo, Ore and Opomu, and waste waters

from Agbara industrial and residential area.

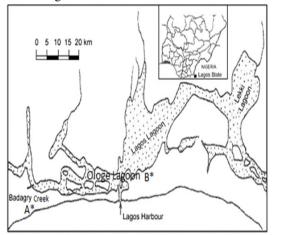


Fig 1: Map of Badagry Creek (A*) and Ologe Lagoon (B*) in Lagos State, Nigeria

*Samples Collection:*Samples of water, sediments, fish and shell fish were collected from two different sampling points at Badagry Creek and Ologe Lagoon between August and October 2016, and subsequently treated following the methods of the American Public Health Association (APHA, 2005).

Extraction and fractionation of PAHs in Water Samples: 250ml each of the water samples was transferred into a separating funnel. The pH was adjusted to < pH 2. The solution was then extracted twice with 15ml methylene chloride. The extract was dried with 5g anhydrous sodium sulfate and concentrated to 1ml in rotary evaporator. 50ml of hexane was added and the sample extracted again down to 1ml. The concentrate was fractioned, first eluted with 10ml hexane, and collected as aliphatic fraction, followed by elution with 15ml methylene chloride, and collected as aromatic fraction. Both fractions were concentrated to 1ml and stored capped in GC vials.

Extraction and fractionation of Sediment Samples: 10g of the sediment sample was blended with 10g of anhydrous sodium sulfate. The mixture was placed in an extraction thimble and refluxed for 4 hours with 50ml methylene chloride. Thereafter the solution was cooled, dried with 5g anhydrous sodium sulfate, and concentrated to 1ml in a rotary evaporator. The concentrate was fractionated over silica gel column, first eluted with 10ml hexane and collected as aliphatic fraction, and then with 15ml methylene chloride, and collected as aromatic fraction.

Extraction and fractionation of Fish Samples: The fish sample was homogenized with a blender. A 2g portion of the homogenate saponified with 200ml

methanol/KOH(12% KOH in 95% methanol) solution in an ultrasonic bath at 60 °C, for 30min. The sample was cooled and filtered through glass cool into separatory funnel. The filtrate was extracted twice with 100ml hexane. The extract was washed with methanol/water (4:1) mixture, and then concentrated to 1ml with a rotary evaporator. The concentrate was fractionated through a silica gel column, first eluted with 10ml hexane to collect the aliphatic hydrocarbon fraction, and then with 15ml methylene chloride to collect the aromatic hydrocarbon fraction. Both fractions were concentrated to 1ml and stored capped in GC vials.

Determination of PAHs in the Solid Samples: 5g of the sample was weighed into an extraction bottle and 20ml of dichloromethane was added and sonicated in an ultrasonic sonicator for 2 hrs. The extract was concentrated to 20cm³ in a rotary evaporator. 20ml of 0.5 KOH in 100ml of methanol was added and the mixture was refluxed for 1hr in a water bath at 60°C. 20cm³deionized water was added and extracted with hexane (20cm³). The extract was dried over anhydrous sodium sulphate and the extract was concentrated at 60°C in a rotary evaporator to 20cm³. The extract was passed through silica gel column (DB 5 MS (30m x 0.25mm x 0.5µm) which had been pre-conditioned with hexane. The extract was eluted with 20cm³ of hexane for aliphatic fractions. To some column, 20cm³ dichloromethane was added for the elution of PAHs and the eluent was concentrated to 1cm³ and solvent exchanged with 1cm3 of actonitrile. 1µL of the extract was injected into a pre-programmed GC vials (HP 6890A). The concentration of the PAHs was calculated from the peak area of the calibration standards. 1µL of each of the fractions was injected into the GC, set-up for the quantification of PAHs and the petroleum hydrocarbons (WHO, 2003; APHA, 2005).

GC Operating Conditions for PAHs: Initial oven temp-40°C; Initial hold time-2 min; Ramp – 12° C/min 40 to 300°C at 12°C/min to 300°C for 10 min; Final oven temp- 300°C; Detector temp- FID 350° C; Injector temp- 350° C; Carrier gas- Hydrogen,4 ml/min; constant flow; Injection volume- 1 μ L, splitless, (hold 2 min)

Determination of THC: 3.0g wet weight of each of the fish samples were homogenized in a mortar to paste using a pestle. The homogenized fish samples were freeze-dried for 48hr. The samples were then extracted in a soxhlet apparatus for 8hr using a mixture of nhexane and dichloromethane according to WHO (2003). The total hydrocarbons in the extract were determined using UV spectrophotometer model DREL 3000 at a wavelength of 450 nm against blank of nhexane. Total hydrocarbon content was determined by acidifying samples with concentrated sulphuric acid, and then extracting thrice with 25 ml diethyl ether. The ethereal extract is evaporated and the residue cooled, and weighed. The THC is calculated as:THC (mg/kg) = (weight of residue (mg) x 1000)/ sample weight taken (g)

GC Set-up Conditions for THC: GC: HP 6890A, Column: DB 5 MS (30m x 0.5mm x 0.15µm), Oven: 60°C (hold 2 min), 60 to 300°C at 12°C/min to 300°C for 10 min, Detector: FID 250°C, Carrier Gas: Helium, 48.5cml/s; Make-up: nitrogen at 30 ml/min, Injector: 350°C, Injection volume: 1 µL, splitless,

GC Calibration Standards: THC Mix 1 (2000 mg/L) Diesel Fuel Standard (C_{10} - C_{28}); EPA method 610 PAH standard mix, Calibrate the GC using working standards (5-100 mg/L). Run samples and quantitate the components.

RESULTS AND DISCUSSION

The summary of the concentrations of various PAHs detected in water, sediment, *C. nigrodigitatus* and *M. macrobrachion* from Badagry Creek and Ologe Lagoon are shown in Table 1.

The same concentrations $(0.000 \pm 0.000 \text{ mg/l})$ of naphthalene, acenaphthene, fluoranthene, Pyrene, chrysene, benzo (a) pyrene, rhenanthrene and acenaphthylene respectively were obtained in water samples from both Badagry Creek and Ologe Lagoon. On the other hand, concentrations of anthracene in surface water from Badagry creek and Ologe Lagoon are $0.000 \pm 0.000 \text{ mg/l}$ and $0.001 \pm 0.001 \text{ mg/l}$ respectively. The concentration of fluorine in the surface water at Badagry creek was $0.001 \pm 0.001 \text{ mg/l}$ and $0.005 \pm 0.003 \text{ mg/l}$ from Ologe Lagoon. Benzo (a) anthracene concentration in surface water at Badagry creek was $0.00 \pm 0.000 \text{ mg/l}$ while $0.015 \pm 0.007 \text{ mg/l}$ was obtained from Ologe Lagoon.

Naphthalene values $(0.007 \pm 0.001 \ \mu g/g)$ and $(0.007 \pm 0.005 \ \mu g/g)$ were recorded in sediment from Badagry creek and Ologe Lagoon respectively. The concentration of acenaphthene in bottom sediment from Badagry creek was $0.002 \pm 0.001 \ \mu g/g$ while $0.002 \pm 0.002 \ \mu g/g$ was obtained in Ologe Lagoon. The concentration $(0.003 \pm 0.002 \ \mu g/g)$ of anthracene was recorded in bottom sediments from Badagry creek while at Ologe Lagoon, $0.005 \pm 0.004 \ \mu g/g$ was detected in the sediment. The concentration of fluorine obtained in the sediments was $0.008 \pm 0.009 \ \mu g/g$ and

 $0.002 \pm 0.001 \mu g/g$ from Badagry creek and Ologe Lagoon respectively. The concentration of fluoranthene in bottom sediments was higher in Badagry creek (0.006 \pm 0.008 μ g/g) than in Ologe Lagoon (0.000 \pm 0.000 μ g/g). The concentration of benzo (a) anthracene in bottom sediments was lower in Badagry creek $(0.001 \pm 0.001 \mu g/g)$ when compare to Ologe lagoon $(0.005 \pm 0.006 \ \mu g/g)$. The concentration of pyrene in bottom sediments from both Badagry creek and Ologe lagoon respectively are $(0.004 \pm 0.004 \ \mu g/g)$ and $(0.004 \pm 0.003 \ \mu g/g)$. The concentration of chrysene in bottom sediments was lower in Badagry creek $(0.011 \pm 0.03 \mu g/g)$ than in Ologe Lagoon $(0.024 \pm 0.010 \ \mu g/g)$. The concentration of benzo (a) pyrene $(0.000 \pm 0.000 \,\mu g/g)$ were obtained in bottom sediments from both Badagry creek and Ologe Lagoon. The concentration of rhenanthrene in bottom sediments was lower in Badagry creek (0.002 \pm 0.002 μ g/g) in comparison with that in Ologe lagoon (0.005 \pm 0.007 μ g/g). The concentration of acenaphthylene in bottom sediments was also lower in Badagry creek $(0.000 \pm 0.000 \,\mu g/g)$ than in Ologe Lagoon $(0.001 \pm 0.001 \,\mu g/g)$.

concentrations (0.000) $\pm 0.000 \mu g/g) of$ Similar naphthalene were obtained in C. nigrodigitatus and M. macrobrachion at Ologe Lagoon and Badagry Creek. The concentration of acenaphthene(0.000 ± 0.000 $\mu g/g$) were recorded in C. nigrodigitatus and M. macrobrachion from Badagry creek, while 0.001± $0.000 \mu g/g$ and $0.002 \pm 0.003 \mu g/g$ concentration of acenaphthene were recorded respectively in C. nigrodigitatus and M. macrobrachion at Ologe Lagoon. The same concentration of $0.000 \pm 0.000 \,\mu g/g$ of fluorine was obtained in C. nigrodigitatus and M. macrobrachion at both Badagry creek and Ologe Lagoon. The concentration of anthracene in fin fishes was lower in Badagry creek $(0.000 \pm 0.000 \mu g/g)$ than in Ologe Lagoon $(0.003 \pm 0.004 \mu g/g)$. The concentration of anthracene in shell fishes was lower in Badagry creek $(0.00 \pm 0.00 \mu g/g)$ than in Ologe Lagoon $(0.001 \pm 0.001 \mu g/g)$. The concentrations of fluoranthene in fin fishes were low both in Badagry creek (0.00 \pm 0.00 µg/g) and Ologe Lagoon (0.00 \pm $0.00 \mu g/g$). The concentration of fluoranthene in shell fishes was low both in Badagry creek (0.00 \pm $0.00\mu g/g$) and Ologe Lagoon $(0.00 \pm 0.00\mu g/g)$. The concentration of benzo (a) anthracene in fin fishes was lower in Badagry creek $(0.00 \pm 0.00 \mu g/g)$ than those in Ologe Lagoon $(0.002 \pm 0.003 \mu g/g)$. The concentration of benzo (a) anthracene in shell fish was lower in Badagry creek $(0.00 \pm 0.00 \mu g/g)$ as compared to Ologe Lagoon $(0.002 \pm 0.003 \mu g/g)$.

	Badagry creekTest				Ologe Lagoon Test			
	Surface water	Sediment	C.nigrodigi tatus	M.macrobr achion	Surface Water	Sedimen t	C.nigrodi gitatus	M.macrob rachion
Naphthalene ($\mu g/g$)	0.000 ± 0.000^{a}	0.007 ±	0.000±	0.000±	0.000±	0.007±	0.000±	0.000±
		0.001 ^a	0.000^{a}	0.000^{a}	0.000^{a}	0.005^{a}	0.000^{a}	0.000^{a}
Acenapthene $(\mu g/g)$	0.000±	0.002±	0.000±	0.000±	$0.000 \pm$	0.002±	0.001±	0.002±
	0.000^{a}	0.001 ^a	0.000^{a}	0.000^{a}	0.000^{a}	0.002^{a}	0.001 ^a	0.001 ^a
Fluorine (µg/g)	0.001±	$0.008 \pm$	0.000±	$0.000 \pm$	$0.005 \pm$	0.002±	$0.000 \pm$	$0.000 \pm$
	0.001 ^a	0.009^{a}	0.000^{a}	0.000^{a}	0.003 ^a	0.001 ^a	0.000^{a}	0.000^{a}
Anthracene (µg/g)	0.000±	0.003±	0.000±	$0.000 \pm$	0.001±	0.005±	0.003±	0.001±
	0.000^{a}	0.002^{a}	0.000^{a}	0.000^{a}	0.001 ^a	0.004^{a}	0.004^{a}	0.001 ^a
Fluoranthene $(\mu g/g)$	0.000±	$0.008 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$
	0.000^{a}	0.006^{a}	0.000^{a}	0.000^{a}	0.000^{a}	0.000^{a}	0.000^{a}	0.000^{a}
$Benzo(a)anthracene(\mu g/g)$	0.000±	0.001±	$0.000 \pm$	$0.000 \pm$	0.015±	0.006±	0.003±	0.003±
	0.000^{a}	0.001 ^a	0.000^{a}	0.000^{a}	0.007 ^a	0.005^{a}	0.002 ^a	0.002^{a}
Pyrene (µg/g)	0.000±	0.004±	0.001±	0.003±	$0.000 \pm$	0.004±	0.001±	0.001±
	0.000^{a}	0.004 ^a	0.001 ^a	0.002 ^a	0.000 ^a	0.003 ^a	0.001 ^a	0.000^{a}
Chrysene (µg/g)	0.000±	0.011±	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	0.024±	$0.000 \pm$	0.001±
	0.000^{a}	0.003 ^a	0.000 ^a	0.000^{a}	0.000 ^a	0.010^{a}	0.000 ^a	0.000^{a}
Benzo(a)pyrene (µg/g)	0.000±	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$	$0.000 \pm$
	0.000^{a}	0.000^{a}	0.000 ^a	0.000^{a}	0.000 ^a	0.000^{a}	0.000 ^a	0.000^{a}
Rhenanthrene $(\mu g/g)$	$0.000 \pm$	$0.002\pm$	0.000±	0.000 ± 0.0	$0.000 \pm$	0.007±	$0.000 \pm$	$0.000 \pm$
	0.000^{a}	0.001 ^a	0.000 ^a	00^{a}	0.000 ^a	0.005 ^a	0.000 ^a	0.000^{a}
Acenaphthylene $(\mu g/g)$	0.000±	$0.000 \pm$	0.000±	$0.000 \pm$	$0.000 \pm$	0.001±	$0.000 \pm$	$0.000 \pm$
	0.000^{a}	0.000 ^a	0.000 ^a	0.000 ^a	0.000 ^a	0.001 ^a	0.000 ^a	0.000^{a}
Total hydrocarbon	0.495±	12.090±	27.210±	219.565±1	0.695±	38.57±9.	104.505±	211.565±
Content $(\mu g/g)$	0.035 ^a	8.641 ^b	15.429 ^{bc}	71.891°	0.417 ^a	249 ^{ab}	23.342 ^{bd}	27.923 ^{cd}

Table 1: Mean concentration ±Standard deviation of PAHs and THC in surface water, bottom sediment, C. nigrodigitatus and M. macrobrachion from Badagry creek and Ologe Lagoon.

Mean values with different superscript in the rows are significantly different

However, the concentrations $(0.001 \pm 0.001 \ \mu g/g)$ of pyrene were recorded in fin fish from both water bodies. The concentration of pyrene in shell fish was higher in Badagry creek $(0.002 \pm 0.003 \ \mu g/g)$ than in Ologe Lagoon $(0.001 \pm 0.000 \ \mu g/g)$. The concentration $(0.000 \pm 0.000 \ \mu g/g)$ of chrysene in fin fish were recorded from both Badagry creek and Ologe Lagoon. The concentration of chrysene in shell fish was lower in Badagry creek $(0.000 \pm 0.000 \ \mu g/g)$ and higher in Ologe lagoon $(0.001 \pm 0.000 \ \mu g/g)$.

The concentration $(0.000 \pm 0.000 \mu g/g)$ of benzo (a) pyrene were recorded in fin fish from both Badagry creek and Ologe Lagoon. Similarly, the concentration $(0.000 \pm 0.000 \mu g/g)$ of benzo (a) pyrene in shell fish were recorded from both Badagry creek and Ologe Lagoon. Also, same concentration $(0.000 \pm 0.000 \mu g/g)$ of rhenanthrene were recorded in fin fish from Badagry creek and Ologe Lagoon respectively. The concentration of rhenanthrene in shell fish at both stations was $0.000 \pm 0.000 \mu g/g$. The concentration of acenaphthylene in both fin fish and shell fish respectively was $0.000 \pm 0.000 \mu g/g$ at both water bodies. There were no significant (p<0.05) different in all the PAHs examined in the samples collected from the two stations.

The mean concentration of the total hydrocarbon content in water, sediment, *C. nigrodigitatus* and *M. macrobrachion* respectively recorded in Badagry Creek were $(0.495\pm0.035\text{mg/l}, 12.090\pm8.641\mu\text{g/g}, 27.210\pm15.429 \mu\text{g/g} \text{ and } 219.565\pm171.891\mu\text{g/g})$ while $(0.695\pm0.417\text{mg/l}, 38.57\pm9.249 \mu\text{g/g},$

104.505±23.342 µg/g and 211.565±127.923 µg/g) were obtained in Ologe Lagoon respectively (Table1). The highest concentration of the total hydrocarbon content (219.565±171.891 µg/g) from Badagry Creek was recorded in *M.macrobrachion* while the least (0.495±0.035mg/l) was obtained in the water sample .Similarly, the highest THC (211.565±127.923 µg/g) was recorded in *M.macrobrachion* at Ologe Lagoon while the water sample had the least THC value of 0.695±0.417mg/l. There were significant differences (p<0.05) in the mean value of THC in sediment, *C. nigrodigitatus* and *M. macrobrachion* across the two stations.

The concentrations of all the PAHs examined in water, sediment, C.nigrodigitatus and M. macrobrachion from Badagry Creek and Ologe Lagoon were below the WHO maximum permissible limit of 10mg/l in water, 0.01 µg/g in sediment, 0.001µg/g in fish and shell fish respectively. The PAHs values in this study were lower than PAHs reported by Al-Busaidi et al., (2013) in marine Clam, Liochoncha ornata collected from the Omani sea; in rivers and estuaries of Malaysia (Zakaria et al., 2002); Asuquo and Udoh (1999) in Ethmalosa fimbriata and C. nigrodigitatus from Nigerian rivers; and Kpobari et al., (2013) in two Tilapia queneesis and Liza falcipinis from Ogoni land in Nigeria. Similarly ,the PAHs detected in this study are lower than those reported in stock fishes (Gardus morhua and Molva molva) by Eze and Ogbuehi (2015); Ayejuiyo, et al., (2012) and Liang et al., (2011) .The significant differences recorded in THC of sediment, C. nigrodigitatus and M. macrobrachion

from the two sites was similar to the findings of Ibigoni *et al.*,(2009).However, the THC recorded in this study were lower than total hydrocarbon levels reported in the surface waters, sediments and biota in an oil polluted mangrove wetland in the Niger Delta (Ibigoni *et al.*,2009).

The higher total hydrocarbon concentrations recorded in the stations that have no oil formations could imply input result from other sources like domestic wastes, discharge of sewage, drifts from polluted areas and other activities (Ahmed, 1983). The sediment loads of THC all through the sampling periods and sites were consistently higher than that of the surface water indicating that it was a better indicator of pollution in the Lagoon system even after the sources of pollution has been removed. The hydrocarbon content of fish is mainly associated with the level of organic matter in the environment (Kucuksezin et al., 2006). These levels of total hydrocarbons suggest that the fish species might have fed on high amounts of petroleum or organic matter containing waste material from the industries and runoff from the urban areas (Kucuksezin et al., 2006). High hydrocarbon content causes oxygen deterioration by reduction in gaseous diffusion through the surface film of oil with far reaching implications for the flora and fauna of the affected area (Osuji et al., 2004).

The results also showed that the total hydrocarbon concentrations in the fish species studied were lower than those detected in Mytilus edulis from North Sea (Viddowet al., 1982) and higher than that reported by Asuquo et al., (2001) for total hydrocarbon in Mytilus edulis and Ethmalosa fimbriata from Cross River. The result also revealed that the total hydrocarbon in the fish species studied were all above the WHO permissible limit of heavy metals for seafood of 0.002 µg/g (WHO, 1985). The THC detected in the study was higher than those obtained in Sphyrena afra, Oreochromis niloticus and Elops lacerta from Calabar (Edem et al., 2008) It was also higher than those found in Macura reptantia, Penaeus notialis and Ocypoda africanus from Bight of Bonny, Niger Delta in Nigeria (Nsikak et al., 2007) and David-Oku et al., (2006) in M. vollenhovenii from south eastern Nigeria.

Conclusion: The study has shown that the polyaromatic hydrocarbons were below the risk level, which indicated no risk status from the consumption of the fish species studied. On the contrary, the fin and shell fishes studied from these water bodies are highly contaminated with THC. Therefore, persistent monitoring and strict adherence to responsible waste discharge should be upheld by all industries near these waters in order to avoid deleterious effect of the

biodiversity in these water bodies as well as ensuring safety of the consumers.

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