Insights into the Polycyclic Aromatic Hydrocarbons in Recent Saline Sediment Cores from Gabu Lake, Nigeria

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Abstract

In paleo-environmental studies, the direction of polycyclic aromatic hydrocarbon (PAH) distributions in sediment cores are commonly used markers of alterations in environmental, diagenetic, as well as paleo-climatic conditions. However, the factors contributing to the variability of these compounds are not fully understood particularly, in the Sub-Tropical African region. Here, the comparative evaluation of the recent saline sediment core (GS – obtained towards the shoreline and GC – towards the center) of Gabu Lake was undertaken to asses paleo-climatic changes that might have occurred during the period of geological time frame represented in the sediment intervals under study using gas chromatogram/mass spectrometry. Discrimination of the sources of anthropogenic polycyclic aromatic hydrocarbon to evaluate their petrogenic and pyrogenic origins could not be established due to near absences or non-detection of these PAH types in both GC and GS, reflecting a relatively pristine sedimentary environment most suitable for evaluation of allochthonous and autochthonous organic matter inputs, implying that the PAHs were predominantly from biogenic sources. This accession was founded on the discovery of high levels of isomers of lupines and chrysene with dominant members such as 3,4,7,12a-Tetramethyl-1,2,3,4,4a, 11,12,12a-octa hydro chrysene; 3,3,7,12a-Tetramethyl-1,2,3,4,4a, 11,12,12a-octa hydro chrysene; 3,4,7-Trimethyl-1,2,3,4-tetrahydrochryse-ne; and 3,3,7-Trimethyl-1,2,3,4-tetrahydrochrysene with m/z 292 ($C_{22}H_{28}$) small fragment ions following the loss of different aliphatic groups. Biogenic-derived PAH is formed via anaerobic oxidation of α and β amyrins or lupeol by a progressive aromatization process where the dominant types are found in both cores.

Keywords: *PAHs, Saline, Autochthonous, Allochthonous, paleo-environment, biogenic*

1. INTRODUCTION

Organic contamination of aquatic habitats is a major concern. Polycyclic aromatic hydrocarbons (PAHs) have received an enormous amount of interest as organic pollutants because of their widespread presence in marine sediments [1,2]. More importantly, concerns have been raised about PAHs' mutagenic and genotoxic potential in the environment, as well as their carcinogenic consequences on biological communities [3,4,5]. This is especially true in coastal areas with strong anthropogenic influence and several pollution sources.

Sediments act as storage environments for various organic matter sources and hydrocarbons, which can be utilized to measure environmental impacts over time. Marine-derived organic matter concentration in sediments is higher in locations with high levels of primary activity in surface waters [6].

Polycyclic aromatic hydrocarbons (PAHs) are traditional persistent organic pollutants (POPs). They reside in all forms of nature, including air, earth, rocks, vegetation, and human tissue [7, 8]. PAH deposition can be caused by human actions (timber, fossil fuels, and pasture burning, partial combustion of petroleum and coal, emissions from automobiles, and oil spillage leakage) and from the environment (forest wildfires, eruptions of volcanoes, and metabolism), can all contribute to the deposition of PAHs [9, 10]. Given their insoluble nature PAHs are readily deposited on tiny particles, allowing them to enter water bodies and deposit in sediments via sedimentation. Thus, lake sediments and organic matter (OM) are the principal destinations of PAHs [10, 11].

PAHs from combustion sources have a great affinity for soot fragments in natural sediments. They predominantly aggregate in cells fatty acids as they possess high lipophilic strength. Certain PAHs and petroleum-based hydrocarbons in saltwater and sediment have been found to affect water organisms, even at low quantities [12]. PAHs are a group of chemicals that may affect animals and humans through the food chain owing to their high solubility in lipids, low water solubility, and binding to marine particulates and sediments [13].

PAHs accumulate in sediments using processes by mechanisms identical to those that regulate surface soil deposits. PAHs bind to air particles in rural areas and can settle on the surfaces of lakes, streams, and oceans through dry or wet depositing. When particles disperse, surface of surface water. PAHs that have been adsorbed end up in freshwater or marine sediments. PAH is highly bonded to these sediments, which may act as a pollutant reservoir for PAH release under certain situations. As a result, species that dwell in sediments and filter water are particularly sensitive to pollution [14,15].

PAHs are relatively stationary once integrated into sediments as they are not soluble in water due to their low polarity. Nonetheless, PAHs, especially those with smaller molecular weights, are water-soluble. Due to this, modest levels of PAHs break down and enter the pore water, where they are soluble [16].

Lakes are important geographical features that provide links with hydrological systems on a local and transboundary scale, as well as supplying water sources, fisheries, livelihoods for many nearby communities, and several ecological services [17]. Lake bodies constantly acquire soluble or granular mineral and organic elements created in the water body of the lake (autochthonous) or carried from the catchment area (allochthonous). However, in addition to lake production, urban population growth and development near water bodies (lakes and rivers) add to the discharge and deposition of a variety of solid and nutrient materials on lake sediments [18].

Lake sediments are mostly made up of elastic material (silt, mud, and gravel particles), bio-detritus, precipitates, or a blend of each. The cumulative quantity of each is governed by the characteristics of the surrounding drainage reservoir, the weather, and the lake's overall age. Sediments accumulate at varying levels between aqua-habitats [19]. The accumulation level changes with place, but the average rate of sediment accumulation in fresh-water lake sediments is about 1 mm per year [6]. When a basin empties into a lake, accumulated materials can provide information on historic sedimentation rates, which can be used to assess the efficiency of previous erosion control initiatives [20].

Biological matter is also part of the paleolimnological record retained in lake sediments [21]. The diverse types of organisms that live in a lake and its drainage area generate organic matter with different biochemical properties. Variations in the habitat of these biotas induce various changes in the volumes and types of organic matter accumulated during a lake's life. Hence, organic matter is an active biogeochemical factor in sediments [22]. The composition or grain-size distribution, pH, salinity, DO_2, mineral contents, organic matter, and redox potentials are the essential influncers of a lake's chemical-sediment relationship [23].

Organic pollutants are one of the major environmental glitches resulting from their large-scale uses in transportation, industrial, agricultural, and other sectors, a variety of aliphatic and aromatic hydrocarbons are extensively used as energy sources, and the contamination by organic pollutants in aquatic environments has adverse effects. Previous reports from recent sediment of Gabu Lake indicated the complexity inherent in using molecular proxies for organic matter source characterization [4]. In the present report, the concentration and sources of PAH in the lake will be presented.

2 MATERIALS AND METHODS

Gabu Lake is a small enclosed saline basin of relatively high altitude (69 m above sea level-asl) adjacent to a semi-enclosed freshwater lake (44m asl), located in Gabu community in Yala, Local Government Area of Cross River State, Southeast Nigeria. It has a surface area of about $600m^2$ and a maximum depth of ca. 5 m at the center with underground spring water constituting the main source of water to the lake [4]. The volume of the lake water is ca. $3000m^3$. The lake lies at the intersection of longitude 8.45^0E of the meridian and latitude 6.25ºN of the equator in a sub-humid climate (Fig.1). The vegetation is primarily mangroves, merging with the rainforest belt, and consists of sparse trees, low-covering plants, and grasses. It can be categorized into grassland and rainforest vegetation.

The study area is drained by four major rivers and their tributaries namely Konshisha, Oruaba, Okpauku, and Kpa. These major rivers and their tributaries show dendritic and occasional trellis drainage patterns and empty into the Cross River drainage system. Whereas some of the streams are perennial, others are ephemeral and usually dry up during the dry season. They commonly originate as surface flows from springs that indent the relatively higher elevations from the north towards the southern part of the area. These rivers and their tributaries expectedly influence the groundwater dynamics around the study area [4]. The occupations of the people are mainly subsistence mixed cropping and mining of barite by the youth which is the major solid mineral that dominates the area. The indigenes and strangers are forbidden from drinking, bathing, fishing making salt from the lake water by evaporation process.

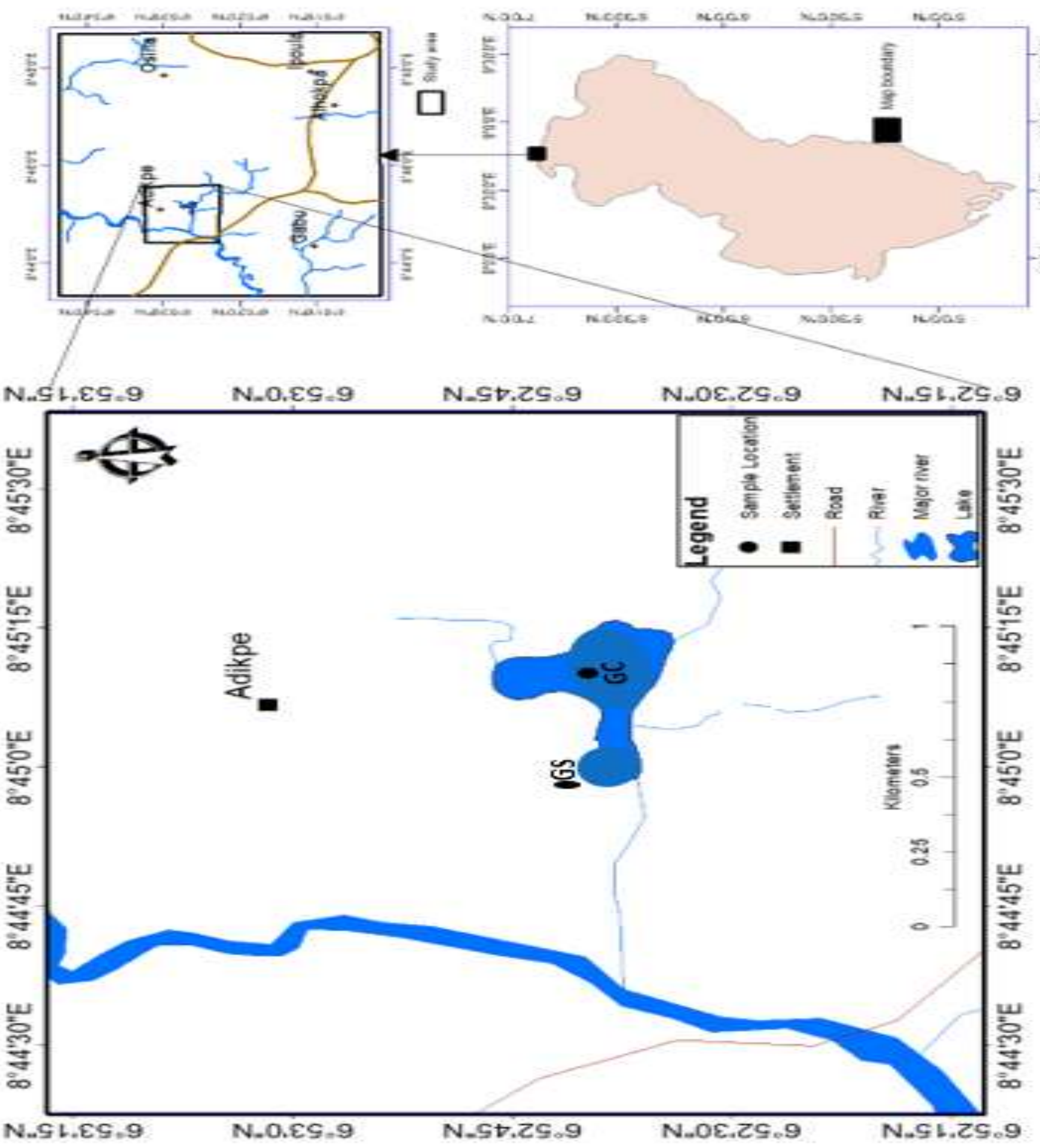


FIG. 1: Map of the study area indicating the sampling points

2.1 Sample collections, preparation, and preservation

Two sediment cores (80 cm long each) were collected from two different locations of the Gabu Lake (one at the center labelled GC and the other shoreline GS) for PAHS characterization using a calibrated PVC corer. Each core was cut into twenty sections of 4cm, thus making a total of 20 subsamples in all. Sediment samples were wrapped with aluminum foil, labeled, and placed in a cooler containing ice (at – 4ºC) to prevent the activity of microorganisms on the samples and transported to the laboratory for further protocols. The samples were air–dried, crushed, sieved to pass through <65µm mesh and kept in a refrigerator (-20ºC) before the extraction process.

2.2 Extraction and Purification

To reduce sample contamination to the barest minimum, all glassware was washed with soap and water and properly rinsed with *n*-hexane and acetone before being placed in an oven regulated at 300ºC to combust any organic carbon that may have adhered to the wall of the glassware and then rinsed again with acetone. Similarly, solvents to be used were properly distilled to ensure purity. Extraction of total lipid extract was done as described by Oyo-Ita *et al,* 2013 [6].

Briefly, 2 g of sieved sediment sample was placed in a test tube. An appropriate solvent mixture of dichloromethane and methanol in the ratio of 2:1 was added to the content of the test tube. Extraction was carried out with the aid of an ultrasonic agitator. The test tube containing the sample was placed in a 250cm^{3}beaker containing little water and the beaker was placed in the agitator for 30 minutes after which the test tube was removed and shaken for 1 minute before returning it to the ultrasonic agitator. The procedure was thus repeated three times to ensure effective extraction. The extracts were centrifuged (3000rpm), decanted into beakers and vacuum evaporated to about 2ml. The extract was saponified with 30 ml KOH (6%) in MeOH for 2 H at 75ºC, the neutral components were recovered by extraction with 3 x 30 ml *n*-hexane. The alkaline mixture was acidified to pH 2 with 5 ml HCl and the acidic compounds recovered by extraction with 3 x 30 ml acetone. Aliquots of the total polar fractions were converted to trimethylsilyl derivatives by reaction with N, O-bis-(trimethylsilyl)trifluoroacetamide (BSTFA) and pyridine for 3 H at 70 °C. This involves the conversion of hydroxylated compounds such as sterols, Fatty-acid and alcohols to the TMSi-esters.

2.3 Gas chromatography-mass spectrometry analysis

Gas chromatography-mass spectrometry and Geochemical analyses were carried out in the laboratories of GFZ German Research Centre for Geosciences, Potsdam, Germany according to Oyo-ita *et al* [3].

2.4 Statistical analysis

Descriptive Statistics such as mean, range, and standard deviation were deployed for this work. The characteristic geochemical proxies and PCA-MLR method were combined to determine the source distribution of OM in this research.

3 RESULTS AND DISCUSSION

The distributions of total polycyclic aromatic hydrocarbon in GC and GS are presented in Fig. 2, the distributions of the individual PAHs are presented in Fig. 3, the representative mass chromatogram is shown in Fig. 4 and their respective structures are shown in Fig 5. The concentration of PAHs was generally higher at the shoreline than at the centre core (Fig. 2). This is because the shoreline area is closer to humans such as mineral mining and sewage disposal which can lead to higher PAHs levels. For the GC, 3,4,7-Trimethyl-1,2,3,4-tetrahydrochrysene exhibited the least concentration (2.91 ng/g dw) at 72 - 76 cm and des (-A)- 17,26-bisnorlupa-5,7,9,11,13-pentaene had the highest concentration (509.61 ng/g dw) at 48 – 52 cm (Fig. 3).

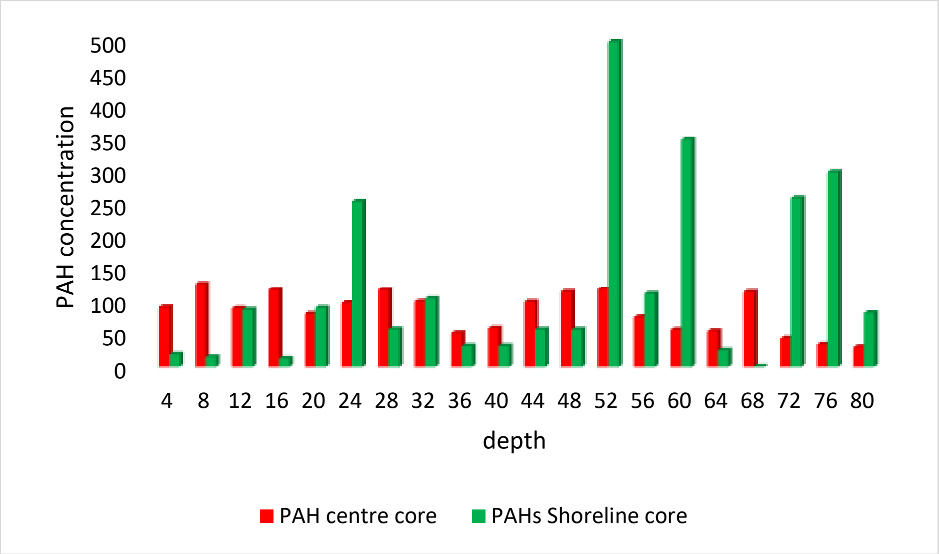


Fig. 2: Distribution of total PAHs (ng/g) at centre and shoreline core of Gabu lake

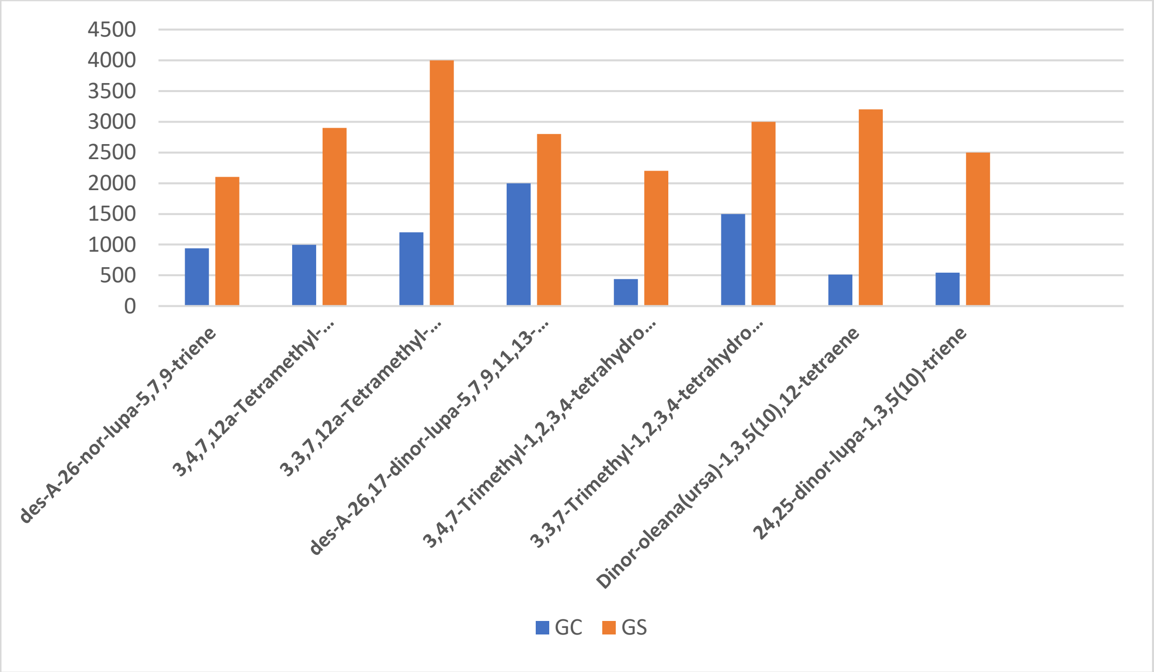


FIG. 3: individual PAH concentrations at centre and shoreline core of Gabu lake

In the case of GS, three (3) PAH compounds namely; des-(A)-26-norlupa-5,7,9-triene, 3,4,7-Trimethyl-1,2,3,4-tetrahydrochrysene and 24,25-bisnorlupa-1,3,5(10)-triene exhibited the least concentration (0.10 ng/g dw) at different sediment layers and the highest concentration (2,359,047 ng/g dw) was found in 3,3,7,12a-Tetramethyl-1,2,3,4,4a,11,12,12a-octahydrochrysene at 48 - 52 cm (Fig. 3).

Discrimination of the sources of anthropogenic polycyclic aromatic hydrocarbon (PAH) to evaluate their petrogenic and pyrogenic origins could not be established due to near absences or no*n*-detection of these PAH types in both GC and GS cores, reflecting a relatively pristine sedimentary environment most suitable for the evaluation of allochthonous and autochthonous organic matter inputs (i.e. natural biogenic types). (Fig. 4). To evaluate the inputs of biogenic /diagenetically derived PAH (i.e. from vascular plant and degradation product), the distribution of mono, di, aromatic compounds and characteristic isomeric series of four, or five-ring PAH were examined down cores (Table 1). These compounds were considered to be produced by microbial and oxidative degradation or transformation of pentacyclic triterpenoids present in vascular plant waxes and woody tissues (Simoneit 1998; Jaffe *et al.* 2006; Oyo-ita *et al.* 2013). Four isomers of the four rings of octahydrochrysene and mono-di aromatic lupine derivatives were dictated down the core.

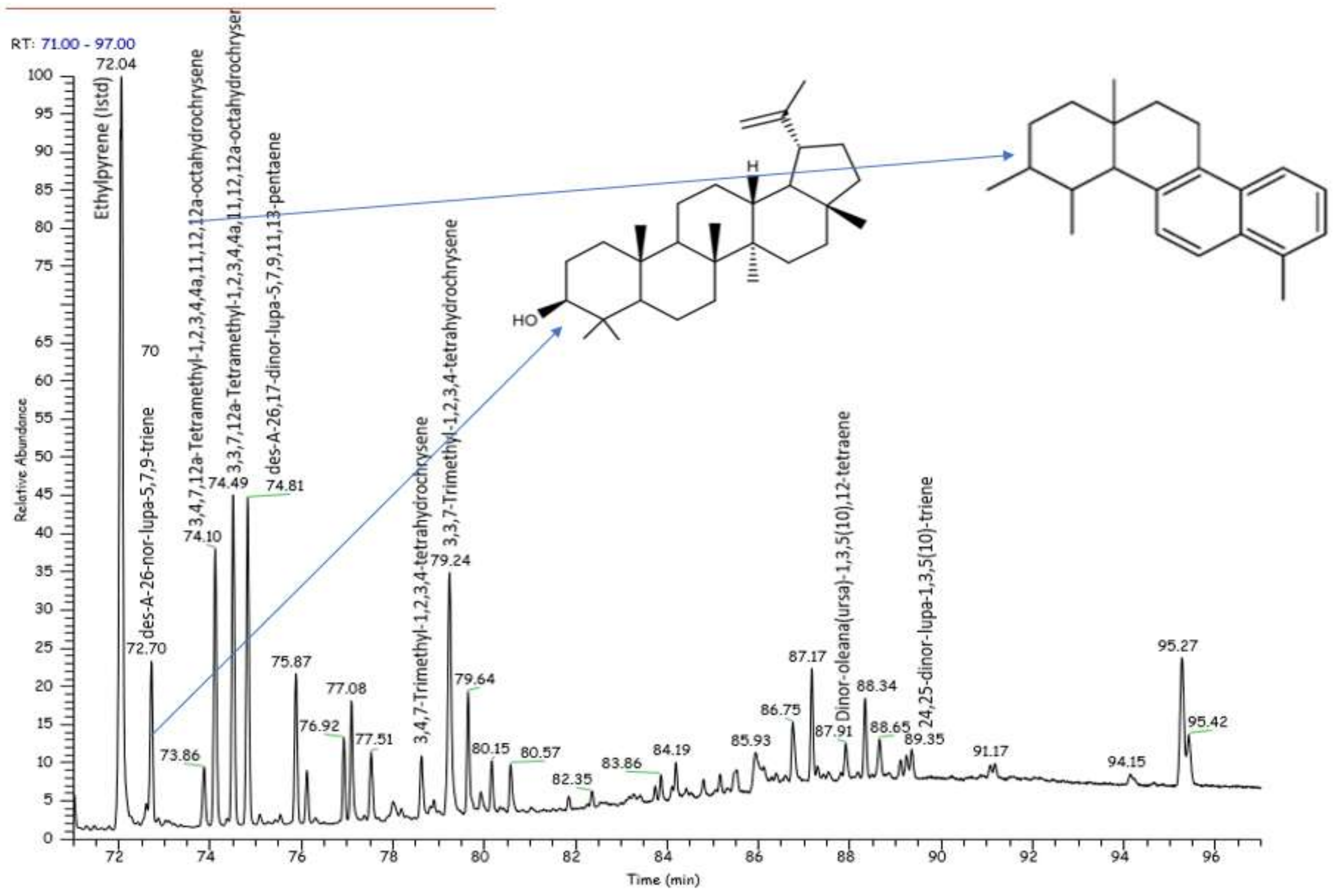
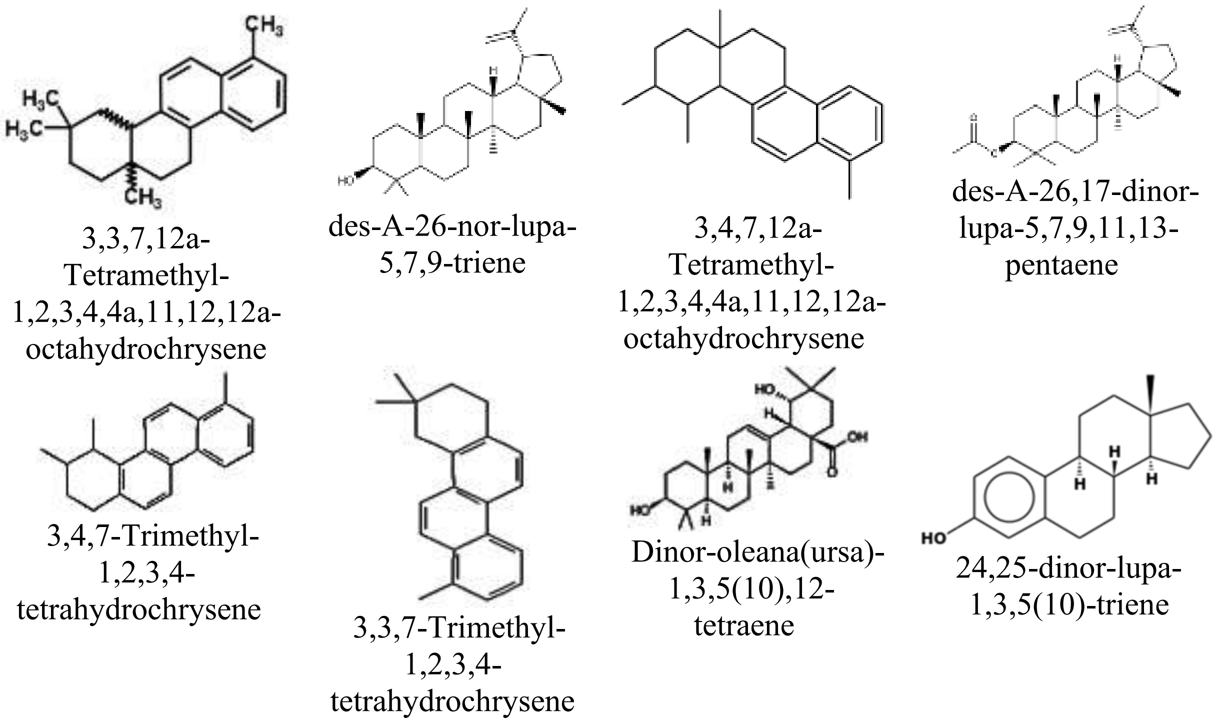


FIG. 4: representative mass chromatogram of biogenic derived polycyclic aromatic hydrocarbons in Gabu lake sediment

The mass chromatogram of tricyclic and tetracyclic derivatives are similar with dominant members such as 3,4,7,12a-Tetramethyl-1,2,3,4,4a, 11,12,12a-octa hydro chrysene; 3,3,7,12a-Tetramethyl-1,2,3,4,4a, 11,12,12a-octa hydro chrysene; 3,4,7-Trimethyl-1,2,3,4-tetrahydrochryse-ne; and 3,3,7-Trimethyl-1,2,3,4-tetrahydrochrysene with m/z 292 (Fig. 4 and 5) ($C_{22}H_{28}$) with minor fragment ions from loss of various aliphatic moieties. The other isomers are equivalent derivatives from ursesne and lupine precursors. The vertical variations of these naturally derived PAH in the 2 cores may reflect variations in source input, differences in sediment, and extent of aerobic or anaerobic microbial activities. In other words, the relatively higher level of natural and diagenetically formed PAH in the GS than in GC may be related to the proximity of the former to the various types of vascular plant (angiosperm vs gymnosperm) stands along the lake catchment.



3,3,7,12a-Tetramethyl-1,2,3,4,4a,11,12,12a-octahydrochrysene

des-A-26-nor-lupa-5,7,9-triene

3,4,7,12a-Tetramethyl-1,2,3,4,4a,11,12,12a-octahydrochrysene

des-A-26,17-dinor-lupa-5,7,9,11,13-pentaene

3,4,7-Trimethyl-1,2,3,4-tetrahydrochrysene

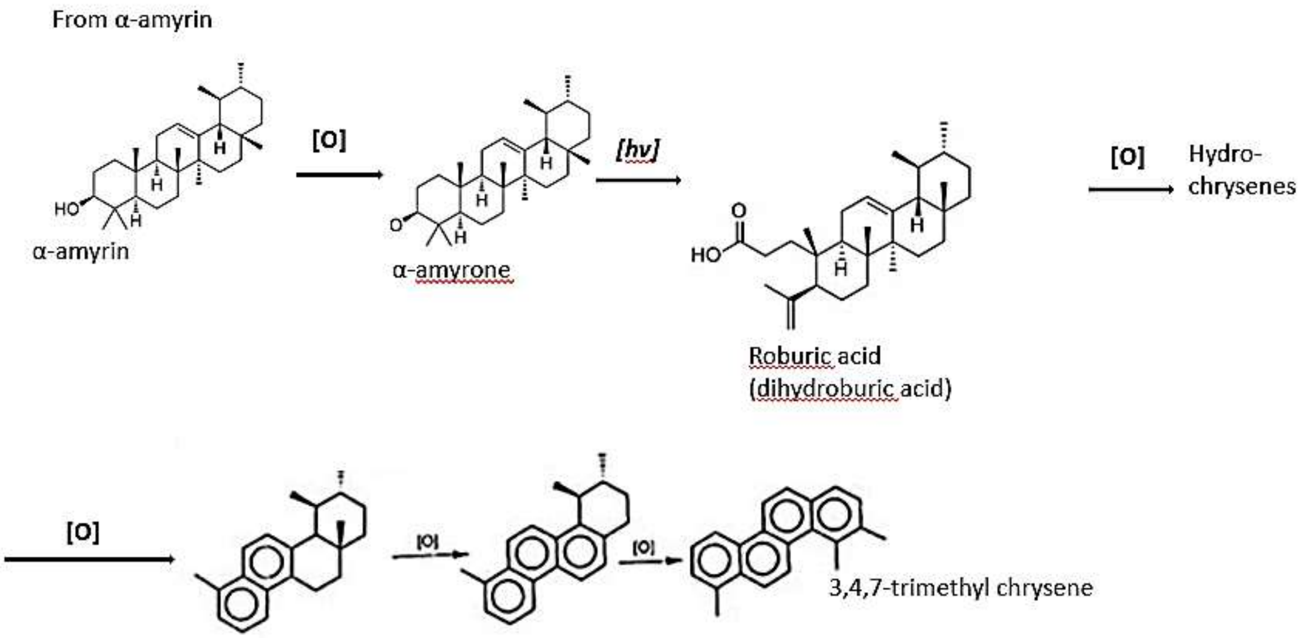
3,3,7-Trimethyl-1,2,3,4-tetrahydrochrysene

Dinor-oleana(ursa)-1,3,5(10),12-tetraene

24,25-dinor-lupa-1,3,5(10)-triene

FIG 5: Structures of PAHs identified in Gabu lake sediment

The vascular plant-derived PAH are considered to be formed during early diagenesis and are generally not related to anthropogenic PAH input [24-27]. The isomeric chrysene derivatives are usually considered to form by progressive aromatization of pentacyclic triterpenoids such as the α and β amyrins or lupeol (natural product precursor) during early diagenesis [28,29]. This aromatization of pentacyclic triterpenoids proceeds from ring A to ring E in the case of pentacyclic picene (not dictated in the studied sample [6, 30]), and from ring B to ring D with initial loss of ring A in the case of the tetracyclic chrysene derivatives as found in the present study (Fig. 6) also, des-A-26,17-dinor-lupa-5,7,9,11,13-pentaene are considered to be derived from lupeol by progressive aromatization of ring A, B, and C with initial loss of ring A via formation of des-A-26-norlupa-5,7,9-triene.



α-amyrin

CONCLUSION

The study investigated the molecular distribution of polycyclic aromatic hydrocarbons in recent sediment cores in Gabu Lake, Yala LGA of Cross River State to delineate their sources with the view to assessing digenetic and paleo-climatic changes. Discrimination of the sources of anthropogenic polycyclic aromatic hydrocarbon to evaluate their petrogenic and pyrogenic origins could not be established due to near absences or no*n*-detection of these PAH types in both GC and GS, reflecting a relatively pristine sedimentary environment most suitable for evaluation of allochthonous and autochthonous organic matter inputs, implying that the PAHs were predominantly from biogenic sources. This accession was based on the detection of high levels of isomers of lupines and chrysene with dominant members such as 3,4,7,12a-Tetramethyl-1,2,3,4,4a, 11,12,12a-octa hydro chrysene; 3,3,7,12a-Tetramethyl-1,2,3,4,4a, 11,12,12a-octa hydro chrysene; 3,4,7-Trimethyl-1,2,3,4-tetrahydrochryse-ne; and 3,3,7-Trimethyl-1,2,3,4-tetrahydrochrysene formed by the progressive anaerobic oxidative aromatization of pentacyclic triterpenoids eg.α and β amylin or lupeol). These digenetic processes involved aromatization of ring B to ring D with initial loss of ring A in the case of the tetracyclic chrysene derivatives and from lupeol by progressive aromatization of ring A, B, and C with initial loss of ring A to formed des-A-26-norlupa-5,7,9-triene.

Disclosure of interest

The authors declared no competing interest.

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