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Abstract

The crude oils from the Northern and offshore Niger Delta basin, Nigeria have been characterized by gas chromatography-mass spectrometry (GC-MS) in terms of their origins, depositional environments and thermal maturity based on the distribution of triaromatic steroids and aromatic dinosteroids. The oil samples from ADL and MJI oilfields were characterized by higher abundance of C_{26} 20R + C_{27} 20S TAS, C_{28} 20S and C₂₈ 20R triaromatic steroids while OKN, MJO and WZB oil samples were characterized by the predominance of C_{26} 20R + C_{27} 20S TAS peak. Among the compounds identified in m/z 245 mass chromatograms, C₂₁methyltriaromatic steroids, C₂₂methyltriaromatic steroids, C₂₇, 4-methyltriaromatic steroid, C₂₇, 3-Methyltriaromatic steroid + C₂₈, 3,24-Dimethyltriaromatic steroid and C_{27} , 4-methyltriaromatic steroid + C_{29} , 4-methyl-, 24ethyltriaromatic steroid were the dominant compounds in the oil samples from ADL and MJI oilfields while the oil samples from OKN, MJO and WZB oilfields were characterized high abundance of all the compounds identified in m/z 245 mass chromatograms of the aromatic fractions of the oils. The crude oils were found to be formed from mixed origin (terrestrial and marine) but with significant contribution of dinoflagellates to the organic matter and deposited in freshwater-brackish/saline lacustrine environment. The oil samples were found to have early oil window maturity status based on the distributions and abundance of triaromatic steroids in the crude oils and this was further supported by well-established maturity parameters based on the saturate and aromatic biomarkers. This study showed that the abundance and distribution of triaromatic steroids and triaromaticdinosteroids can be used to assess the origin, depositional environments and thermal maturity of crude oils in the Niger DeltaBasin.

Keywords: Triaromatic steroids, Aromatic dinosteroids, crude oils, Niger Delta.

Introduction	crude oils, are ro
Biological markers, or "molecular fossils", which can be identifiedin	organicfacies, depo andthermalhistory
extracts of ancient sediments and in	in the case of petro

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rock(s). Particular classes of molecular in petroleum have fossils found increasing application as age diagnostic markers (Peters et al., 2005). Steranes with 27, 28 and 29 carbons and conventional side chains are important biomarkers for eukaryotic life. Furthermore, those with unconventional side chains offer opportunities for making more specific genetic links between families of organisms and mature sediments and oils (Moldowan et al.,1991).

Triaromatic steroids (TAS) may originate by aromatization and loss of a methyl group $(-CH_3)$ from steroids monoaromatic (Rioloand Albrecht, 1985; Riolo et al., 1985; Peters et al., 2005). The C_{26} , C_{27} , C_{28} triaromatic steroids potentially retain genetic information about petroleum and source rocks similar to C_{27} , C_{28} and C_{29} regular steranes. 2005). The cross plot of C_{26}/C_{28} 20S versus C_{27}/C_{28} 20R triaromatic steroid ratios have been used to distinguish petroleum systems (Picha and Peters, 1998; Peters et al., 2005). Zhang et al. (2000, 2002) and Mi et al. (2007) relatively higher reported

abundances of C_{26} 20S, C_{26} 20R + C_{27} 20S and C_{27} 20R TAS in oils derived from Cambrian source rocks, whereas C_{28} 20S and C_{28} 20R TAS are relatively abundant in oils which originated from Middle–Upper Ordovician carbonate source rocks in the Lunnan oil field, Tarim Basin. Also, triaromatic steroids have been successfully applied to thermal maturity assessment of crude oils and source rocks (Xiangchun et al., 2011; Asif and Fazeelat, 2012).

Triaromaticdinosteroids are important molecular fossil groups derived from dinosterols, compounds known to be the almost exclusive, widely occurring natural products of dinoflagellates (Robinson et al., 1984; Moldowan et al., 1996; Moldowan and Talyzina, 1998). The highest concentrations of these compounds are typically found in strata deposited since the beginning of the Mesozoic. However, Moldowan and (1998)concluded Talyzina that dinoflagellate ancestors may date back to the Early Cambrian on the basis of biomarker evidence. Zhang et al. (2002) reported the wide occurrence of 24norcholestane, dinosteranesand

triaromaticdinosteroids with relatively high abundance in extracts from organic sediments in rich Cambrian and Precambrian (Sinian) rocks of the Tarim Basin. Biogeochemical evidence for dinoflagellate ancestors were reported in the Early Cambrian (Withers, 1987) and molecular evidence has provided a link of cyst-forming dinoflagellates with pre-Triassic ancestors (Moldowanand Talyzina, 1998). These studies suggest that planktonic algae suchas

Geological and Stratigraphic Setting

Niger delta is a sedimentary basin situated in the re-entrant of the Gulf of Guinea, West Africa. The sub-aerial portion of the Niger Delta covers approximately 75,000 km² and stretches about 200 km from apex to mouth. The total sedimentary prism encompasses km^2 , 140000 with a maximum stratigraphic thickness of about 12 km (Whiteman, 1982). The stratigraphy of the thick sedimentary sequence is divided into three lithostratigraphic units, namely the Akata, Agbada and Benin Formations (Short and Stauble, 1967).

dinoflagellates and diatoms may have originated earlier than the Mesozoic.

However, there is limited reports on triaromatic steroids in Niger Delta crude oils while aromatic dinosteroids have been not reported or studied in Niger Delta crude oils. This study is conducted to investigate the distributions and geochemical significance of aromatic steroids and aromatic dinosteroids in Niger Delta source rocks.

The uppermost unit, the Benin Formation which ranges from Oligocene to recent in age, comprises continental/fluviatile sands, gravels, and backswamp deposits up to 2500 m thick. These are underlain by the Agbada Formation of paralic, brackish to marine, coastal and fluvio-marine deposits. These are mainly interbedded sandstones and shale with minor lignite organized into coarsening upward 'offlap' cycles. Underlying this unit is the Akata Formation, ranging in age from Paleocene to Miocene consists of mainly of overpressure shales deposited under fully marineconditions.-

The depobelts are partitioned into 6-7east-west bound blocks corresponding discrete periods of the deltas to evolutionary history starting from the oldest in the north, northern delta to theyoungest, offshore in the south (Doust and Omatsola, 1990). It is believed that each depobelt constitutes a more or less autonomous unit with respect to sedimentation, structural deformation hydrocarbon and generation and accumulation (Evamy et al., 1978). Available source rocks in the basinexist

Materials andMethods

Samples

Forty-one crude oil samples from five wells in five fields were collected and analyzed.

Fractionation and Analysis

The crude oils were separated into saturated and aromatic hydrocarbon fractions using silica gel/alumina chromatography columns eluted with nhexane and dichloromethane:n-hexane (2:1, v:v), respectively (Li et al., 2012). The GC-MS analyses of the saturate and aromatic fractions were performed on an agilent 5975i gas chromatography (GC) equipped with an HP-5MS (5%)

mainly in the lower parts of the paralic (Agbada Formation) sequence and uppermost strata of the continuous marine shale (Akata Formation; Evamyet al., 1978; Ekweozor and Daukoru, 1994). The hydrocarbon habitat of the Niger Delta is mostly the sandstone reservoir of the Agbada Formation where oil and gas are usually trapped in rollover anticlines associated with growthfaults.

phenylmethylpolysiloxane) fused silica capillary column (60m x 0.25mm i.d., x 0.25µm film thickness) coupled to an agilent 5975i mass spectrometry (MS). The GC operating conditions are as follows: the oven temperature was held isothermally at 80°C for 1 min, ramped to 310°C at 3°C/min and held isothermal for 16 min (Li et al., 2012). Helium was used as the carrier gas with constant flow rate of 1.2 mL/min. The MS was operated in the electron impact (EI) mode at 70eV, an ion source temperature of 250 °C and injector temperature of 285°C. The identification and elution order of fluorene and its derivativeswere

determined by comparison of their mass spectra and relative retention times in the corresponding mass chromatograms with

Results and Discussion

Distribution characteristics of Triaromatic Steroids and Aromatic Dinosteroids in Crude Oils from Niger DeltaBasin

m/z 231 245 The and mass chromatograms showing the distributions of triaromaticsteroids (TAS) and aromatic dinosteroids in the oil samples are shown in Figure 1. The peak identities are shown in Table 1. The crude oils from ADL and MJI oilfields are characterized by higher abundance of $C_{26} 20R + C_{27} 20S$, $C_{28} 20S$ and $C_{28} 20R$ triaromatic steroids while C₂₆ 20S occur as least (Fig. 1). This pattern of distribution has been reported in a freshwater lacustrine crude oils from LinnanSubsag, Shandong Province, China (Xiangchun et al., 2011) and crude oils from Ordovician reservoirs from the Tahe oil field, Tarimbasin, China (Li et al., 2012). OKN, MJO and WZB oil samples are characterized by the predominance of C_{26} 20R + C_{27} 20S TASpeakswhileC₂₈20SandC₂₈20R

those reported in literature (Zhang et al., 2000, 2002; Wang et al., 2008; Li et al., 2012).

TAS and C₂₆ 20S occurred relatively low (Fig. 1). Crude oils and source rocks from saline and brackish lacustrine environments have been reported to contain low abundance of C₂₈ TAS (Xiangchun al., 2011). et Also, Cambrian and Lower Ordovician source rocks have been shown to be characterized by lower C_{28} 20S and C_{28} 20R TAS and higher C_{26} 20S, C_{27} 20R and C_{26} 20R + C_{27} 20S TAS peaks, which is similar to those reported in the literature (Zhang et al., 2002; Mi et al., 2007; Li et al., 2012). There are appreciable quantities of C_{20} and C₂₁triaromatic steroids in all the oil samples studied in this work (Fig. 1). Among the methyltriaromatic steroids, dimethyltriaromatic steroids, aromatic dinosteroids and C₂₉, 3-Methyl-, 24ethyltriaromatic steroids identified in chromatograms, m/z 245 mass C₂₁methyltriaromatic steroids, C₂₂methyltriaromatic steroids, C₂₇, 4steroid, methyltriaromatic 3-C₂₇. Methyltriaromatic steroid $+ C_{28}, 3, 24$ -

Dimethyltriaromatic steroid and C_{27} , 4methyltriaromatic steroid + C_{29} , 4methyl-, 24-ethyltriaromatic steroid are the dominant compounds in the oil samples from ADL and MJI oilfields while one of the peaks of aromatic dinosteroids occurs as the least (Fig. 2). The samples from OKN, MJO and WZB oilfields are characterized by high abundance of all the compounds

Geochemical Significance of Triaromatic Steroids and Aromatic Dinosteroids in Crude Oils from Niger DeltaBasin.

The geochemical parameters computed from triaromatic steroids, aromatic dinosteroids and other geochemical parameters are shown in Table 2. The geochemical parameters used are determine the origin and maturity status of the Niger Delta crude oils.Occurrences of aromatic steroids are influenced by multiple factors and thus can be used as indicator of different depositional source inputs and environments. For example, organic matter formed in fresh water environment is abundant with C₂₈-TAS while in saline and brackish water

identified in the m/z 245 mass chromatograms of the aromatic fractions of the crude oils (Fig. 2). The significant of aromatic dinosteroids amounts observed in the oil samples indicates contributions significant of dinoflagellates into the organic matter that formed the oils (Li et al., 2012; Zhang et al., 2002; Wang et al., 2008).

environments it is abundant with C₂₆-TAS (Xiangchun et al., 2011). The $C_{26}/C_{28} - 20S$ TAS and C_{27}/C_{28} - 20R TAS ratios for the rock samples range from 0.21 to 0.50 and 0.43 to 0.84, respectively (Table 2). The low values of $C_{26}/C_{28} - 20S$ TAS recorded in some oil samples indicate oils formed from a freshwater environment while higher values recorded in some samples indicate a lake environment of brackishsaline water (Xiangchun et al., 2011; Li et al., 2012). These results show that the crude oils studied are formed from source rocks of mixed origin (terrestrial and marine) deposited in freshwaterbrackish/saline lacustrine environments. The cross plots of $C_{26}/C_{28} - 20S$ TAS versus C₂₇/C₂₈- 20R TAS showedthat

the oil samples from the same field received similar organic material (Fig. 3). The triaromaticdinosteroids are assumed to originate from dinosterol and related sterols. which characterize modern marine dinoflagellates (Moldowan and Talyzina, 1998; Peters et al., 2005). This association suggests that triaromaticdinosteroids useful are biomarkers for improving the recognition dinoflagellates. of ancient The triaromaticdinosteroid hydrocarbon index (TDSI) could be used to reflect the contribution of organic matter from dinoflagellates in the source rocks and crude oils. This parameter has been used indicate contribution to the of dinoflagellates to the oils and source rocks of the Tarim Basin, NW China (Zhang et al., 2000, Li et al. 2012) and Paleogene lacustrine sediments from Bohai Bay Basin, China (Wang et al., 2008). The TDSI values for the rock samples range from 0.52 to 0.71 (Table 2). These values indicate significant contributions of dinoflagellates to the organic matter that formed the source rocks. Progressively evidence suggested

thatdinoflagellates or theirancestors

has

more

appeared during the Early Cambrian (about 520 Ma. Moldowan and Talyzina, 1998) or even Precambrian (Summons et al., 1992; Moldowan et al., 2000; Zhang et al., 2002). In this study, the presence of aromatized dinosteroids with extremely high relative concentrations indicates dinoflagellates that or their lived during ancestors the Cambrian and Early Ordovician in the seas of Niger Delta.

Ratios of C₂₆-TAS 20S/ (20S+20R) and C_{28} -TAS 20S/(20S+20R) are mostly used to evaluate thermal maturity (Xiangchun et al., 2011; Asif and Fazeelat, 2012). In the oils crude studied. theratiosare within the range of 0.22 to 0.28 and 0.52 to 0.59 respectively (Table 2), suggesting early mature characteristics. The values also vary over a small range, indicating similar maturity status for the oils. The Methylphenanthrene index (MPI-1) and 20S/(20S+20R)C₂₉steranes for the rock samples range from 0.69 to 0.96 and 0.28 to 0.47, respectively (Table

Radke, 1988) already inferred from the

2), supporting that the oils are within oil

window (Seifert and Moldowan, 1981;

from the triaromatic steroids based maturity parameters. These findings are consistent with the previous report on

4.0 Conclusion

The crude oils from the Northern and offshore Niger Delta basin, Nigeria have characterized been by gas chromatography-mass spectrometry (GC-MS) in terms of their origins, depositional environments and thermal maturity based on the distribution of triaromatic steroids and aromatic dinosteroids. The oil samples from ADL and MJI oilfields were characterized by higher abundance of C_{26} 20R + C_{27} 20S TAS, C₂₈ 20S and C₂₈ 20R triaromatic steroids while OKN, MJO and WZB oil samples were characterized by the predominance of C_{26} 20R + C_{27} 20STAS peak. Among the compounds identified in m/z 245 mass chromatograms, C₂₁methyltriaromatic steroids, C₂₂methyltriaromatic steroids, C₂₇, 4methyltriaromatic steroid, C_{27} , 3-Methyltriaromatic steroid + C_{28} , 3,24-Dimethyltriaromatic steroid and C₂₇, 4methyltriaromatic steroid + C₂₉, 4methyl-, 24-ethyltriaromatic steroid were the dominant compounds in theoil

maturity status of Niger Delta crude oils (Sonibare et al., 2008; Faboya et al, 2015).

samples from ADL and MJI oilfields while the oil samples from OKN, MJO and WZB oilfields were characterized high abundance of all the compounds identified in m/z 245 mass chromatograms of the aromatic fractions of the oils. The crude oils were found to be formed from mixed origin (terrestrial and marine) but with significant contribution of dinoflagellates to the organic matter and deposited in freshwater-brackish/saline lacustrine environment. The oil samples were found to have early oil window maturity status based on the distributions and abundance of triaromatic steroids in the crude oils and this was further supported by well-established maturity parameters based on the saturate and aromatic biomarkers.

This study showed that the abundance and distribution oftriaromatic steroids and triaromaticdinosteroids can be used to assess the origin, depositional environments and

thermal maturity of crude oilsin the

Niger DeltaBasin.

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m/z 245: Methyltriaromatic steroids and triaromatic dinosteroids



Time (min)



Fig. 1: m/z 231 Mass chromatograms showing the distributions of

I triaromatic steroids in the crude oils from the Niger Delta. Oil samples.

Chromatograms of aromatic fractions of Niger Delta rock samples.					
PEAK NUMBER	COMPOUND				
1	C ₂₁ Methyltriaromatic steroid				
2	C ₂₂ Methyltriaromatic steroid				
3	C ₂₇ ,3-Methyltriaromatic steroid				
4	C ₂₇ ,4-Methyltriaromatic steroid				
	C ₂₇ ,3-Methyltriaromatic steroid+C ₂₈ ,3,24-Dimethyltriaromatic				
3+8	steroid				
5	C ₂₉ Triaromatic dinoflagellatessterane				
	C ₂₇ ,4-Methyltriaromatic steroid+C ₂₉ ,4-Methyl-, 24-				
4+7	Ehytriaromatic steroid				
6	C ₂₉ ,3-Methyl-, 24-Ethyltriaromatic steroid				
7	C ₂₉ ,4-Methyl-,24-Ethyltriaromatic steroid				
8	C ₂₈ ,3,24-Dimethyltriaromatic steroid				

Table 1: The peak identities of the compounds identified in m/z 245	mass
Chromatograms of aromatic fractions of Niger Delta rock sam	ples.

Table 2a: Source and thermal maturity parameters computed from triaromatic steroids, aromaticdinosteroids and related compounds in Niger Delta crudeoils

		C ₂₇ /C ₂₈	C ₂₆ /C ₂₈		20S/(20S+20R)	20S/(20S+20R)	20S/20S+20R	
Sample	Depth(m)	20R TAS	20S TAS	TDSI	C ₂₆ TAS	C ₂₈ TAS	C ₂₉	MPI^{-1}
ADL1	2602-2607	0.48	0.21	0.59	0.23	0.55	0.41	0.73
ADL2	2602-2607	0.48	0.21	0.57	0.23	0.56	0.40	0.71
ADL3	2702-2704	0.50	0.32	0.57	0.23	0.52	0.40	0.69
ADL4	2718-2720	0.51	0.23	0.52	0.22	0.59	0.44	0.73
ADL5	2759-2763	0.52	0.24	0.58	0.25	0.55	0.43	0.70
ADL6	2766-2770	0.45	0.23	0.58	0.24	0.55	0.45	0.78
ADL7	2905-2908	0.52	0.22	0.56	0.23	0.54	0.45	0.79
ADL8	2964-2967	0.45	0.23	0.58	0.24	0.56	0.44	0.82
ADL9	3064-3052	0.43	0.22	0.59	0.24	0.54	0.47	0.84
OKN1	1749-1750	0.71	0.45	0.65	0.28	0.55	0.36	0.95
OKN2	1892-1895	0.77	0.49	0.68	0.27	0.53	0.40	0.91
OKN3	1905-1907	0.77	0.48	0.68	0.27	0.54	0.40	0.91
OKN4	1952-1955	0.69	0.45	0.65	0.27	0.54	0.33	0.93
OKN5	2050-2059	0.76	0.49	0.68	0.27	0.53	0.41	0.90
OKN6	2369-2555	0.75	0.49	0.68	0.27	0.52	0.39	0.95
OKN7	2377-2672	0.75	0.49	0.68	0.27	0.53	0.43	0.90
OKN8	2469-2782	0.75	0.48	0.67	0.27	0.53	0.39	0.89
OKN9	2485-2793	0.78	0.48	0.68	0.27	0.54	0.41	0.91

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OKN10	2489-2491	0.70	0.46	0.66	0.27	0.54	0.36	0.76
OKN11	2521-2523	0.72	0.46	0.66	0.27	0.55	0.33	0.70

Table 2b: Source and thermal maturity parameters computed from triaromatic steroids, aromaticdinosteroids and related compounds in Niger Delta crudeoils

		C ₂₇ /C ₂₈	C_{26}/C_{28}		20S/(20S+20R)	20S/(20S+20R)	20S/20S+20R	
Sample	Depth(m)	20R TAS	20S TAS	TDSI	C ₂₆ TAS	C ₂₈ TAS	C ₂₉	MPI^{-1}
OKN12	2530-2537	0.70	0.45	0.65	0.27	0.54	0.37	0.76
OKN13	2566-2568	0.69	0.45	0.64	0.28	0.55	0.35	0.94
OKN14	2677-2683	0.68	0.45	0.62	0.28	0.54	0.41	0.62
OKN15	3148-3154	0.69	0.48	0.62	0.28	0.54	0.38	0.65
OKN16	3593-3605	0.66	0.43	0.61	0.27	0.53	0.41	0.70
MJO1	2207-2216	0.70	0.44	0.69	0.27	0.52	0.45	0.77
MJO2	2070-2081	0.70	0.44	0.69	0.27	0.53	0.46	0.77
MJO3	2091-2104	0.70	0.45	0.68	0.27	0.53	0.45	0.78
MJO4	2096-2101	0.71	0.44	0.68	0.27	0.53	0.45	0.78
MJI1	1607-1611	0.59	0.35	0.64	0.26	0.55	0.28	0.76
MJI2	1777-1779	0.56	0.32	0.63	0.26	0.56	0.40	0.84
MJI3	1795-1797	0.56	0.32	0.63	0.26	0.55	0.36	0.83
MJI4	1920-1921	0.55	0.32	0.64	0.26	0.55	0.37	0.96
MJI5	1936-2342	0.59	0.36	0.64	0.26	0.55	0.35	0.78
MJI6	1944-1947	0.54	0.33	0.64	0.26	0.54	0.34	0.82
MJI7	1948-1950	0.54	0.31	0.62	0.26	0.56	0.39	0.86
MJI8	1979-2398	0.59	0.34	0.63	0.26	0.55	0.36	0.82
MJI9	2442-2444	0.53	0.34	0.62	0.26	0.55	0.38	0.83
MJI10	3030-3036	0.59	0.37	0.62	0.27	0.53	0.38	0.83
WZB1	1610-2647	0.84	0.50	0.71	0.24	0.52	0.42	0.86
WZB2	1811-1957	0.75	0.46	0.69	0.26	0.52	0.37	0.86

 C_{26}/C_{28} 20S TAS = triaromatic steroids ratio.

 C_{27}/C_{28} 20R TAS = triaromatic steroids ratio

TDSI = Triaromaticdinosteroids/(triaromaticdinosteroids + 3-methyl-24-ethyl triaromatic steroids) index

 C_{26} TAS S/(S+R) = C_{26} triaromatic steroids maturity parameters

 C_{28} TAS S/(S+R) = C_{28} triaromatic steroids maturity parameters

MPI-1 = methylphenanthrene index 1

20S/(20S+20R) C₂₉ = C₂₉steranes maturity parameters.