**Terpane Characterization of Crude Oils from Niger Delta, Nigeria: A Geochemical Appraisal**

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**Abstract:** The geochemistry of crude oils from the Niger Delta, Nigeria, were evaluated using the characterization of C19 to C35 terpanes. Analyses of two representative crude oils from Western and Central Niger Delta showed abundances of C19 to C29 tricyclic terpanes constituted 12.09% and 29.74%, C24 tetracyclic terpane 0.31% and 0.15% and C27 to C35 pentacyclic terpanes 87.61% and 70.12%, respectively. Diagnostic ratios of terpanes indicate relatively low abundances of C23, C28 and C29 tricyclic terpanes, low abundances of homohopanes, a significantly high abundance of oleanane and suggested that the Niger Delta crude oils were derived from terrestrial organic matter source rocks deposited in an oxic environment during the Tertiary period. Multivariate oil-oil correlation plot showed the Western and Central Niger Delta crude oils are not distinct, but moderately related (genetically). However, diagnostic ratios of C24 tetracyclic terpane, which was high and moderate; gammacerane, which was low and high, revealed crude oils from Western Niger Delta were derived from predominantly terrestrial source and crude oils from Central Niger Delta were derived from terrestrial source with input from marine organic matter, respectively. Tricyclic terpanes/hopanes and isomerization ratios of C32 homohopanes indicated that the Niger Delta crude oils were generated at high maturity, at top of the oil generation window.

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**1. Introduction**

Crude oil originates from the chemical and geological transformation of biomolecules from prehistoric organisms of both marine and terrestrial sources buried deep under the earth's crust during sedimentary processes (Wang et al., 2006). Many of the geochemical compounds identified contain vital information about the fate of the crude oil in which they are found (Osuji et al., 2005; Mobarakabad et al., 2011). One such class of geochemical compounds found in crude oils is terpane, derived from terpenoid found in plants.

Terpanes found in crude oils and source rock extracts range from C19 tricyclic terpanes to C35 homohopanes and they are detected in a mixture of other types of petroleum hydrocarbons by gas chromatography-mass spectrometry (GC-MS) via monitoring the fragment ion at mass to charge (m/z) 191 (Killops and Killops, 2013). They have been used extensively to indicate organic matter's source, condition of the depositional environment, age of source rock from which the crude oil was derived and maturity at the time of oil generation, as well as correlation and/or differentiation of crude oils and source rocks (El-Gayar et al., 2002; Younes, 2001; Mohialdeen et al., 2013). The relative compositions of terpanes such as C19/C23, C23/C24, and C26/C25 tricyclic terpanes, as well as 18α/17α trisnorhopanes (Ts/Tm), C31 to C35 homohopanes, gammercerane and oleanane, are used as diagnostic parameters for evaluating crude oils (Volk et al., 2005).

The Niger Delta region in southern Nigeria is one of the world's most productive oil regions. As of early 2012, the region possessed an estimated 38 billion barrels of crude oil (Isumonah, 2013). Niger Delta crude oils have been evaluated with their bulk characteristics, light hydrocarbons, triterpanes, and aromatic hydrocarbons (Eneogwe et al., 2003; Onyema and Osuji 2015; Thomas, 1995; Sonibare et al., 2008; Onyema and Ajike 2010). This research utilized the distribution and characterization of C19 to C35 terpanes for correlation and geochemical appraisal of crude oils from two sub-regions of the Niger Delta, Nigeria.

**2. Material and Methods**

***2.1. Description of Study Area***

The Niger Delta region is located in southern Nigeria, between longitudes 5° and 8° E and latitudes 3° and 6° N, at the apex of the Gulf of Guinea. The region formed from the late Cretaceous into the Tertiary period with depositions of sediments south-west ward into the Gulf of Guinea in three sequentially arranged stratigraphic formations: Akata formation, Agbada formation and Benin formation. These formations are distinguished primarily based on the sand-shale ratios. Petroleum system in the region is identified as the Tertiary Niger Delta (Akata-Agbada) Petroleum System (Tuttle et al., 1999).

***2.2. Sample Collection***

Crude oil samples were obtained from oil flow stations: one each from Delta State (5°28’ N, 6°12’ E) and Rivers State (4°39’ N, 7°16’ E) in the Western and Central Niger Delta sub-regions, respectively. Each crude oil obtained, is a mixture of several producing oil wells flowing into the flow station and serve as a representative crude oil sample. With assistance of field technicians, the crude oils were obtained (1 liter each) and labelled sample-IRD and sample-AGR, respectively.

***2.3. Crude oil fractionation***

50 mg of each crude oil sample was weighed into labelled centrifuge tube. Excess pentane were added to the crude oil samples in the centrifuge tubes, allowed to stand for 3 hours and centrifuged to coalesce the precipitated asphaltenes. The pentane soluble fractions were decanted, concentrated with nitrogen gas at 40°C and each deasphalted crude oil sample transferred onto the top a glass column (30 cm x 1 cm) stuffed with glass wool at the bottom and packed with silica. *n*-hexane, dichloromethane and dichloromethane/methanol (1:1) mixture were poured into the packed columns to elute the saturates, aromatics, and resins, respectively. The eluents were concentrated using nitrogen gas at 40°C.

***2.4. Gas chromatography-mass spectrometry (GC-MS) analyses***

Agilent 7890A gas chromatograph (GC) system with a HP-5 silica capillary column (50 m x 320 m i.d. and 0.25 m film thickness) and an Agilent 5975 mass selective detector (MSD) was used to analyze the saturate hydrocarbon fractions of the crude oil samples. With the aid of G4513A automatic liquid sampler (ALS), 1 microliter of the saturate fraction of each sample was injected into the GC capillary column in splitless mode. The GC oven was set to an initial temperature of 80°C for 5 minutes (mins.), then ramped to 300°C at a rate of 4°C/min. and held at this temperature for 30 min. Terpanes were monitored using their characteristic fragment ion at mass to charge (m/z) 191. Peaks were identified by comparing their mass spectra to related literature. Quantification of the abundance of each peak was obtained by area integration, which was processed by Chemstation OPEN LAB CDS software.

**3. Results**

GC-MS analyses of the crude oil samples (-IRD and -AGR) monitored using the m/z 191 fragment ion showed well-resolved peaks (Figures 1 and 2). This fragment ion (m/z 191) is characteristic and indicate the occurrence of terpanes in Niger Delta crude oils (Volk et al., 2005; Eneogwe et al., 2002).

***3.1. Distribution of Terpanes***

Terpanes in samples -IRD and -AGR elute between 20 and 50 minutes on the m/z 191 mass chromatograms and ranged from C19 tricyclic terpane to C35 homohopanes (Figures 1 and 2; see Table 1 for peaks identifications). The most abundant terpanes in the Niger Delta crude oil samples were hopane (H30), oleanane (OL), and 30-norhopane (NH30), respectively. It was also observed that tricyclic terpanes, TR20a-d and TR21a-f (see Table 1 for peaks id.), were reduced in sample-IRD and prominent in sample-AGR (Figures 1 and 2).



Figure 1. m/z 191 mass chromatogram of crude oil sample-IRD from Western Niger Delta showing the distribution of terpanes



Figure 2.m/z 191 mass chromatogram of crude oil sample-AGR from Central Niger Delta showing the distribution of terpanes

Table 1.Identifications of terpane peaks on the m/z 191 mass chromatograms of the crude oil samples from Niger Delta, Nigeria

|  |  |  |  |
| --- | --- | --- | --- |
| Peak | Terpanes | Code | Empirical formula |
| 1 | C19 tricyclic terpane | TR19 | C19H34 |
| 2 | C20 tricyclic terpane (a) | TR20a | C20H36 |
| 3 | C20 tricyclic terpane (b) | TR20b | C20H36 |
| 4 | C20 tricyclic terpane (c) | TR20c | C20H36 |
| 5 | C20 tricyclic terpane (d) | TR20d | C20H36 |
| 6 | C21 tricyclic terpane (a) | TR21a | C21H38 |
| 7 | C21 tricyclic terpane (b) | TR21b | C21H38 |
| 8 | C21 tricyclic terpane (c) | TR21c | C21H38 |
| 9 | C21 tricyclic terpane (d) | TR21d | C21H38 |
| 10 | C21 tricyclic terpane (e) | TR21e | C21H38 |
| 11 | C21 tricyclic terpane (f) | TR21f | C21H38 |
| 12 | C22 tricyclic terpane | TR22 | C22H40 |
| 13 | C23 tricyclic terpane | TR23 | C23H42 |
| 14 | C24 tricyclic terpane | TR24 | C24H44 |
| 15 | C25 tricyclic terpane (a) | TR25a | C25H46 |
| 16 | C25 tricyclic terpane (b) | TR25b | C25H46 |
| 17 | C24 tetracyclic terpane | TET24 | C24H42 |
| 18 | C26 S tricyclic terpane (S) | TR26S | C26H49 |
| 19 | C26 tricyclic terpane (R) | TR26R | C26H50 |
| 20 | C28 tricyclic terpane (a) | TR28A | C28H52 |
| 21 | C28 tricyclic terpane (b) | TR28B | C28H52 |
| 22 | C29 tricyclic terpane (a) | TR29A | C29H54 |
| 23 | C29 tricyclic terpane (b) | TR29B | C29H54 |
| 24 | C27 18α trisnorhopane | Ts | C27H46 |
| 25 | C27 17α trisnorhopane | Tm | C27H46 |
| 26 | C28 bisnorhopane | H28 | C28H48 |
| 27 | C29 17α 25-norhopane | NH25a | C29H50 |
| 28 | C29 17β 25-norhopane | NH25b | C29H50 |
| 29 | C29 17α 30-norhopane | NH30 | C29H50 |
| 30 | C30 17α diahopane (Lupane) | LUP | C30H52 |
| 31 | C29 normoretane | M29 | C29H50 |
| 32 | Oleanane | OL | C30H52 |
| 33 | C30 hopane | H30 | C30H52 |
| 34 | C30 moretane | M30 | C30H52 |
| 35 | C31 22S homohopane | H31S | C31H54 |
| 36 | C31 22R homohopane | H31R | C31H54 |
| 37 | Gammacerane | GAM | C30H52 |
| 38 | C32 22S bishomohopane | H32S | C32H56 |
| 39 | C32 22R bishomohopane | H32R | C32H56 |
| 40 | C33 22S trishomohopane | H33S | C33H58 |
| 41 | C33 22R trishomohopane | H33R | C33H58 |
| 42 | C34 22S tetrakishomohopane | H34S | C34H60 |
| 43 | C34 22R tetrakishomohopane | H34R | C34H60 |
| 44 | C35 22S pentakishomohopane | H35S | C35H62 |
| 45 | C35 22R pentakishomohopane | H35R | C35H62 |

Terpanes detected by GC-MS analyses of the crude oil samples were of three groups: C19 to C29 tricyclic terpanes, C24 tetracyclic terpane and C27 to C35 pentacyclic terpanes. Figure 3 shows the profile of the three terpane groups detected in the crude oil samples. Total abundance of terpanes in the crude oils were high with the abundance in sample-IRD 1.52 times higher than sample-AGR (Figure 3). This indicate terpanes were more abundant in crude oils from Western Niger Delta than Central Niger Delta.

Pentacyclic terpanes (PENT) was the most abundant terpane group in samples -IRD and -AGR with compositions of 87.61% and 70.12%, while tetracyclic terpane (TET) was the least abundant with compositions of 0.31% and 0.15%, respectively. Abundances of tricyclic terpanes (TR) was low with compositions of 12.09% and 29.74%, respectively (Figure 3). According to Huang and Meinschein (1979), tricyclic terpanes are commonly in high abundances in marine-derived oils.

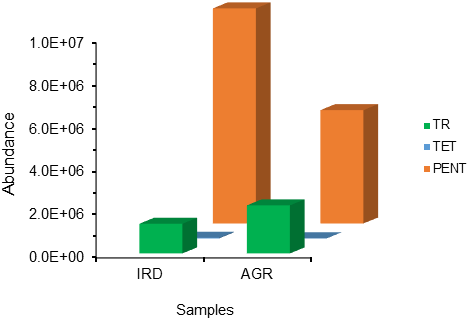




Figure 3.Profile of three terpane groups: tricyclic terpanes (TR), tetracyclic terpane (TET) and pentacyclic terpanes (PENT), in the crude oils (samples-IRD and -AGR) from Niger Delta, Nigeria

This distribution profile of terpane groups indicate the Niger Delta crude oils are characterized by high abundance of pentacyclic terpanes and mostly derived from non-marine/terrestrial organic matter sources. The abundance of tricyclic terpanes (TR) was more in sample-AGR (29.74%) than sample-IRD (12.09%) suggesting crude oils from Central Niger Delta received more marine organic matter input than those from Western Niger Delta.

***3.2. Characterization of Terpanes***

Geochemical characterization of crude oils utilize the abundances of terpanes to determine the source organic matter type, depositional environment and thermal maturity (Peters et al., 2005). From the abundances of terpanes in samples -IRD and -AGR, diagnostic ratios were calculated and used to characterize the Western and Central Niger Delta crude oils (Table 2). Crude oils from marine sources usually have high C23 tricyclic terpane to C30 17α(H)-hopane (TR23/H30) ratio with high abundances of C28 - C30 extended tricyclic terpanes associated with source rocks deposited in an anoxic environment during marine upwelling (Huang and Meinschein, 1979; Holba et al., 2003). Ratios of TR23/H30, TR28/H30 and TR29/H30 in samples -IRD and -AGR, which ranged from 0.02 to 0.05, were relatively low (Table 2). The absence of C30 extended tricyclic terpane and the relatively low abundances of C23, C28 and C29 tricyclic terpanes suggest that Niger Delta crude oils were generated from terrestrial organic matter deposited in an oxic environment.

Table 2.Diagnostic ratios of terpanes utilized for geochemical characterization of crude oil from Niger Delta, Nigeria



C24 tetracyclic terpane (TET24) is a biomarker that indicates terrestrial higher plant source (Disnar and Harouna, 1994). It was detected in both crude oil samples with abundances, as determined by TET24/TET24+TR26 ratio (see Table 1 for peak id), high (0.42) in sample-IRD and moderate (0.27) in sample-AGR (table 2). This suggest terrestrial organic matter source for the Niger Delta crude oils with the Western oil receiving a greater input than the Central oil. Oleanane is another biomarker indicating terrestrial higher plant (angiosperm) source, which appeared from the Cretaceous period (<130 million years) with growing abundance in the Tertiary period, but not found in older rocks and oils (Ekweozor and Udo, 1988; Riva et al., 1988; Murray et al., 1997; Hans et al., 2002). Alberdi and Lopez (2000) have used oleanane as a geochemical tool to assess crude oils from two sub-basins of the Venezuelan petroleum system. Oleanane abundances (OL/H30) in the crude oil samples were significantly high (0.90 and 0.95; table 2) and indicate the Niger Delta crude oils were predominantly derived from terrestrial organic matter source rocks deposited during the Tertiary period.

Gammacerane is a terpane biomarker used to determine the degree of salinity in the depositional environment of source rocks. Its abundance rises as the salinity of the depositional environment rises, from lacustrine to marine (Sinninghe Damste et al., 1995). Ratios of GAM/H30 and GAM/H31 (see Table 1 for peaks id), used to determine the abundance of gammacerane in crude oils, were low in sample-IRD (0.06; 0.13) and high in sample-AGR (0.16; 0.31), respectively (Table 2). This abundance of gammacerane suggest that the crude oils from Western Niger Delta (-IRD) were formed from source rocks deposited in a low salinity environment, whereas those from Central Niger Delta (-AGR) were formed from source rocks deposited in a higher salinity environment, typical of a marine habitat.

The relative abundances of C31 to C35 homohopanes and homohopane index (HHI), abundance of C35 homohopanes relative to total homohopanes, are suitable indicators for determining the reduction/oxidation (redox) condition of the depositional environment. Crude oils derived from source rocks deposited under highly reducing (anoxic) marine conditions, generally show increasing relative abundances of C31 to C35 homohopanes and high HHI, while those deposited under oxidizing (oxic) conditions show decreasing relative abundances and low HHI [18]. For samples -IRD and -AGR, the calculated relative abundances of homohopanes (HH/Σ H31-H35) progressively decreased from 0.40 to 0.07 and 0.35 to 0.06, while the HHI values were 0.07 and 0.06, respectively. These calculated ratio values suggest that Niger Delta oils were derived from source rocks deposited in an oxic environment.

Terpanes commonly used for assessment of crude oil thermal maturity are the homohopanes. This is based on the isomerization of the C-22 hydrogen atom of the C31 to C35 homohopanes from the biologically generated R-isomer to the thermodynamically more stable S-isomer until an equilibrium mixture is reached at maturity. Ratios of C32 homohopanes, H32S/H32R and H32S/H32S+H32R (see Table 1 for peaks id), were used to assess the maturity of the samples. For samples -IRD and -AGR, the calculated H32S/H32R ratio were 1.30 and 1.38, while for H32S/H32S+H32R ratio were 0.57 and 0.58, respectively (table 2). At about equilibrium, H32S/H32S+H32R ratio values ranges from 0.57 to 0.62 and indicated the crude oil was formed at top of the oil generation window (Peters and Moldowan, 1993). The isomerization ratio values indicate the thermodynamically stable H32S was more than the biologically generated H32R (i.e. slightly above equilibrium) and that Niger Delta crude oils were generated at peak of the oil generation window at a high maturity, with sample-IRD slightly less mature than sample-AGR. Similarly, tricyclic terpanes to hopanes (TR/Hop) ratio values of 0.18 and 0.54 suggest sample-IRD was less mature than sample-AGR, respectively. This is due to thermal cracking of hydrocarbons from high molecular weight to low molecular weight during crude oil maturation, resulting in the increased abundance of tricyclic terpanes in high-mature oils (Waples, 1985; Farrimond et al., 1999). From the geochemical characterization results of samples-IRD and -AGR, Niger Delta crude oils were generated at peak of the oil generation window at a high maturity, predominantly from terrestrial organic matter with source rocks deposited in an oxic environment during the Tertiary period. However, the abundance of gammacerane suggest that crude oils from Central Niger Delta received a greater marine input and were more mature than those from Western Niger Delta.

***3.3. Oil–oil Correlation***

Multivariate techniques was used for oil-oil correlation of the Western and Central Niger Delta crude oils. The technique compares hydrocarbon ratios of crude oils and put them in a multivariate plot (Ali et al., 2002; Onyema and Manilla, 2010). Nine (9) selected terpane ratios were determined (table 3) and used for multivariate correlation of oil samples -IRD and -AGR (Figure 4).

Table 3.Selected terpane ratios employed for multivariate correlation of the Niger Delta crude oil samples

|  |  |  |  |
| --- | --- | --- | --- |
| Axis | Terpane ratios | Sample  -IRD | Sample  -AGR |
| 1. | C19/(C19 + C23) | 0.41 | 0.70 |
| 2. | TR24/(TR24+TR23) | 0.46 | 0.40 |
| 3. | TET24/TR26 | 0.73 | 0.38 |
| 4. | Ts/Tm | 0.64 | 0.90 |
| 5. | GAM/H31(S+R) | 0.13 | 0.31 |
| 6. | H35/H34 | 0.68 | 0.69 |
| 7. | H35/(H35 + H34) | 0.40 | 0.41 |
| 8. | H30/M30 | 0.82 | 0.81 |
| 9. | H32 22S/(22S+22R) | 0.57 | 0.58 |

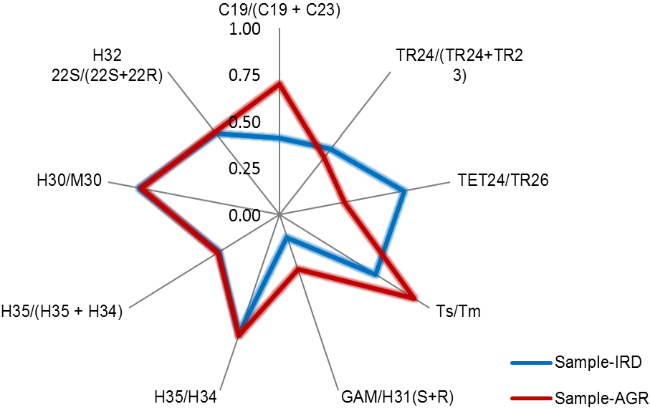


Figure 4.Plot of selected terpane ratios employed for multivariate correlation of oil samples -IRD and -AGR from Niger Delta, Nigeria

From figure 4, the pattern formed on the multivariate plot of selected terpane ratios by sample-IRD was comparable to sample-AGR. The multivariate plot of 9-axis (selected terpane ratios) showed samples-IRD and -AGR followed different paths on axis-1,-2, -3 and -4; similar path with slight deviation on axis-5 and same paths on axis-6, -7, -8 and -9 indicating the crude oil samples are not distinct, but moderately similar. Differences in paths followed by crude oils and source rocks on multivariate plots reflect differences in organic matter source, depositional environment, lithology and/or transformation process (Volk et al., 2005; Ali et al., 2002). This reveal the Western and Central Niger Delta crude oils are genetically related with input from a different organic matter source, depositional environment, lithology and/or transformation process to crude oils from one of the sub-regions.

**4. Conclusion**

Two representative crude oils from Western and Central Niger Delta were evaluated employing terpane characterization. Diagnostic ratios of C23, C28 and C29 tricyclic terpanes, C24 tetracyclic terpane, oleanane, gammacerane and homohopanes as well as multivariate correlation indicate crude oils from Western Niger Delta were derived from predominantly terrestrial source and Central Niger Delta, derived from mixed marine and terrestrial organic matter source. Source rocks of Niger Delta crude oils were deposited in an oxic environment during the Tertiary period. Ratios of tricyclic terpanes/hopanes and C32 homohopane isomerization indicate the Niger Delta crude oils were generated at high maturity, at top of the oil generation window with crude oils from the Central Niger Delta more mature than the Western Niger Delta. These terpanes can furthermore be used to characterize the Niger Delta petroleum system for continuity / compartmentalization and geochemical allocation of commingled crude oils from the region.

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