Exploration for Deeper Sources in Gas Window Regimes using Diamondoid, An Advanced Geochemical Tool

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ABSTRACT

Over the years, biomarkers such as steranes, hopanes, PAHs and their derivatives have been used in geochemistry for provenance identification amongst others. This is due to their non- biodegradability at high temperatures. However, their limitations are seen in the fact that they become heavily depleted beyond the oil window and may not appear at the gas window. Most petroleum systems are complex with different source contributions to reservoirs. A lot of sources and bypassed pay zones have been left unidentified due to non-detection. However with the development of nanotechnology, diamondoids have been identified, developed and used as marker to identify very deep sources where biomarkers are limited in application. Diamondoids are extremely thermally stable oil components. They are nanosized hydrogen terminators of Adamantane series. All liquid petroleum contain diamondoids. Diamondoid analyses gives information on gas window source contribution and deep gas sources where biomarker analyses is inadequate. Three methods have been developed; Quantitative Diamondoid Analyses, Quantitative Extended Diamondoid Analyses and Carbon Isotope Diamondoid Analyses. These methods have shown to be effective in mixed and cracked oil separations, source to oil correlation and gas window source detection. Stigmastane, C29ααα20R series is correlated with 3,4 dimethyldiamantane to give a clear distinct of oil sources and estimate diamondoid baseline concentration. Case studies on petroleum systems are discussed with emphases on how the technology has helped in unraveling deeper source contributions most importantly in the gas window regimes. Conclusively, integrating diamondoid geochemical tool in exploration activities would add value to understanding deeper source contribution in gas windows.

Keywords: Diamondoids, 3, 4 Dimethyldiamantane, Adamantane, Stigmastane, Gas window

INTRODUCTION

Background

Diamondoid technology is a recent and advanced geochemical tool developed to solve exploration problems and give clarity to complex petroleum systems. In Nigeria, the government saddled NNPC's Frontier Exploration Services department with the responsibility to explore for hydrocarbon in the frontier basins. Most of the frontier basins in Nigeria are quite rich in gas or condensates (Ubani, 2016). However, challenges in exploring for hydrocarbon from deep gas sources have created a paradigm shift towards advancing technological development in our exploratory tools.

In geochemical exploration, basic geochemical screening such as total organic carbon analysis, rock eval pyrolysis to get production index PI, S1, S2, S3 and Tmax, biomarker analysis using biomarkers such as pristane, phytane, steranes, hopanes, PAHs are used for source rock studies that extends to oil window and some part of the gas window (Peters *et al.,* 2005). However, deep gas sources cannot be unmasked using the prior mentioned techniques. Hence, turning to the use of diamondoid technology have shown positive solutions to challenges of unravelling the deep gas sources. Although a lot of geological studies including biomarker analyses have been done on most sedimentary basins in Nigeria, not much have been done on diamondoid analyses to explore for gas window sources.

This paper aims to give insight on diamondoid and its application as a geochemical marker to detect deeper sources especially those in gas window regime. In addition, some case studies of diamondoid analyses carried out on some Basins would be highlighted to show the usefulness of the types of Diamondoid Analyses (QDA, QEDA and CSIA-D) in solving exploration problems. The paper thus brings to light the necessity for the Nigerian Oil and Gas community to take up Diamondoid as a key geochemical tool in exploratory studies. This can be integrated into basin and petroleum systems modelling to help properly derisk our petroleum systems, give more insights into deeper sources especially gas window sources and overall increase the hydrocarbon reserves in Nigeria.

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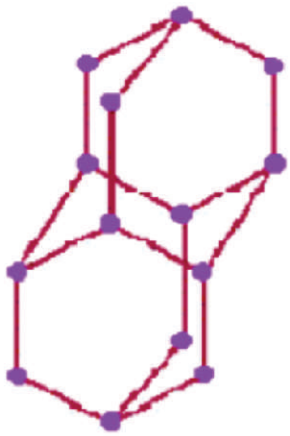
Nigerian Association of Petroleum Explorationists.

a)



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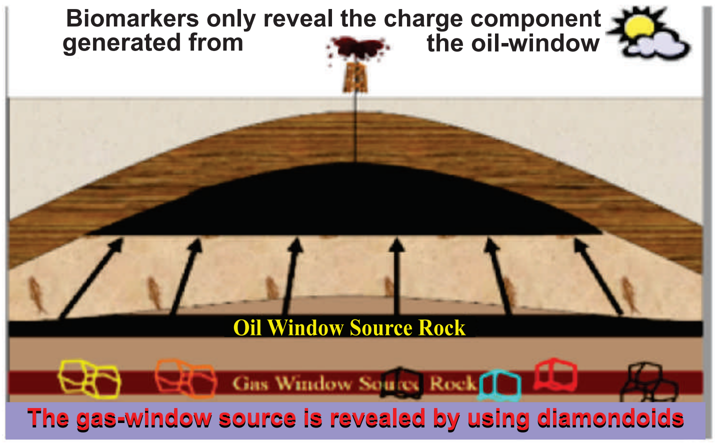

Figure 1a: Diamondoid Structure (Mansoori, 2005). **Figure 1b:** Diagram showing Diamondoid in a petroleum system (Moldowan, 2017).

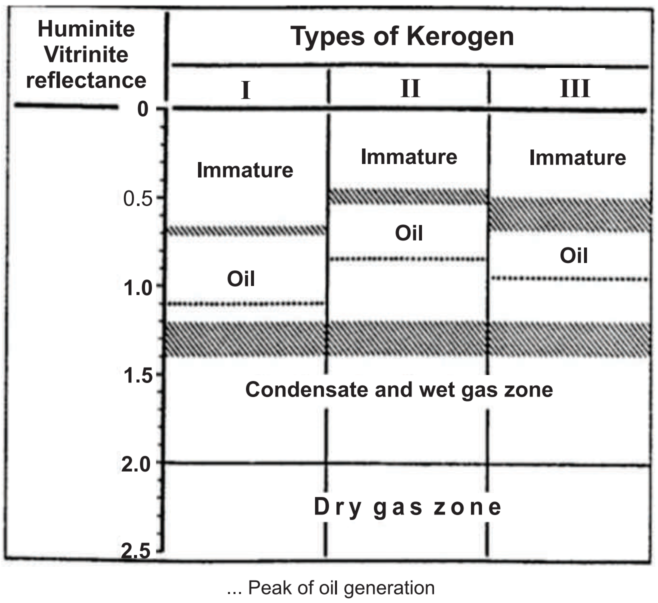

Figure 2: Diagram showing the Gas zones (regimes) from vitrinite reflectance profile (Franck d'Almeidaa et al., 2016). The gas regime starts from Vitrinite reflectance of 1.2Ro to beyond 2.5Ro. Diamondoid Analyses are useful in zones of very high vitrinite values.

Diamondoid and its Physicochemical Properties

The history of diamondoid dates back to 1933 when it was first isolated in Hodonin Oil fields in Czechoslovaskai in 1993 and was later synthesized by Lewis acid- catalysed rearrangement of hydrocarbon(Araujo *et al.,* 2012). A varying opinion is that diamondoid in petroleum was formed from enzymatically created lipids with subsequent structural rearrangement during the process of source rock maturation and oil generation(Mansoori, 2017). Because of this, diamondoid content of petroleum is applied to distinguish source rock facies.

Diamondoids are extremely thermally stable oil components present in liquid petroleum (Fang *et al.,* 2012). Diamondoids have high melting point, high vapour pressure and are illustrated by adamantane, diamantane, triamantane and methyl diamantane among others (Catherine *et al.,* 1991). Also, Mansoori (2007) stated that diamondoids will preferentially partition themselves at high temperature, high pressure and rather low boiling fractions of crude oil. The molecular structure of diamondoid shows cage like ultra-stable, saturated hydrocarbon with carbon-carbon structure repeating unit like in diamond lattice structure (Figure 1a). The unit of Adamantane is C4n+6 H4n+12 having only one isomer. However isomer increases in the polyadamantane. Adamantane crystallizes in a face centred cubic lattice unlike other organic compound making the molecule free from angle strain.

Diamondoid analyses gives information on gas window source contribution and deep gas sources where biomarker analyses is limited (Figure 1b). The distribution of higher diamondoids in oils is related to the oil sources. As a result, by accurately determining higher diamondoid distributions and relative concentrations, one can recognize oil families in much the same way biomarkers are used to group oils. However, whereas biomarkers are useful only up until the late oil window, higher diamondoid distributions can be used for correlations far into the gas window. If source-rock extracts or oils from known single source rocks are available, then the higher diamondoid distributions in oils and gas condensates of unknown origin can be correlated to their sources. Diamondoid concentration increases in the gas window (Wei *et al.,* 2006).

Application of Diamondoid Analysis

Diamondoids are used to recognize cracked oil and assess the degree of thermal alteration. Cracked oil often indicates active deep gas and condensate sources that are not recognized by using biomarkers. Diamondoid recognizes mixtures of cracked oil with normal "black" oil. Such mixtures often escape detection by geochemists. Diamondoids estimate the extent of deep source contribution to a reservoir containing mixed oil. This can also help to further evaluate gas potential and determine seal integrity throughout a basin. It determines the source of highly mature oil (and by inference, gas) by correlation using diamondoid isotope ratios and fingerprints. It is applicable to post mature and heavily degraded oil. Fingerprinting the diamondoids may be the best way to identify deep source since deep gas and condensate bubble through oil reservoirs and diamondoid from deep sources dissolve in oil (Moldowan, 2012, 2017; Moldowan *et al.,* 2015; Dahl *et al.,* 1999; Anlai, 2016). Diamondoid is also used in fingerprinting of light petroleum and gasoline(Stout&Douglas, 2010). Quantitative and related parameters of diamondoids can play an important role in determining the kerogen types, tracing oil origin in sedimentary basin subsurface, thermal cracking and thermal maturity(Dahl *et al.,* 1999; Wei et al., 2007; Dahl et al., 2003; Wang *et al.,* 2006; Huang *et al.,* 2011; Zhang & Huang, 2005).

Table 1: Differences between Biomarkers and Diamondoids.

Biomarkers	Diamondoids
Structure (cyclohexane& cyclopentane rings) $C_{30}H_{52}$, $C_{26}H_{36}$ etc	Condensed cyclohexane ring $C_{10}H_{16}$
Oil Window	Gas Window
Resistant to biodegradation	Highly resistant to biodegradation
High volatility	Low volatility
Boiling point 400 °C -500°C	Boiling point up to 1000°C
Vitrinite Reflectance 1.0Ro	2.0- 4.0 Ro
Low-Medium maturity	High maturity, cracked
Non Crystalline	Micro Crystalline

METHOD

Overview of Procedure for Diamondoid analysis

Diamondoid determination has been tried with different methods over the years. Dahl *et al.,* (1999) isolated the crystal of hexamentane or cyclohexamentane from distillate fraction. Le *et al.,* (2012) studied the characterization of diamondoid using GCXGC TOFMS and this method can detect trimethyl adamantane. GCXGC TOFMS can detect some diamondoids that cannot be seen by GCMS. Catherine *et al.,* (1991) recovered diamondoid from gas stream using oligomers. Zhang *et al.,* (2014) used a gas purge micro syringe extraction (GP-MSE) rather than column chromatography to prepare oil samples for diamondoid analysis. Anlai (2016) linked the ratio of methyl adamantane and methyldiamantane to determine the maturity of sources from condensate wells as they showed positive correlation with vitrinite reflectance (Ro).

The procedure for carrying out diamondoid analyses include sampling and sample administration, sample preparation, extraction, analysis and then interpretation of chromatograms. Depending on the analyte sought for as well as nature of sample to be analysed, various methods of extraction exist. Normally, column chromatography is carried out for extraction purpose just as in biomarker analysis.

Samples are spiked with the appropriate amounts of 5β-cholane and deuterated diamondoid internal standards for quantitation. Deuterated diamondoids in the spike should include D15-1-methyladamantane, D16-adamantane, D3-1-methyldiamantane, D4-diamantane, and D4-triamantane. The deuterated diamondoid standards (the "D"-compounds) are only known to be available to Biomarker Technologies, Inc., which has prepared them synthetically for their use in Quantitative Diamondoid Analyses (Anlai, 2106). Spiked samples are fractionated by sequential elution using a proprietary light hydrocarbon solvents and dichloromethane on silica gel columns to obtain saturate and aromatic fractions. Paraffins are removed from saturated hydrocarbon fractions using a proprietary light hydrocarbon solvent on zeolite columns. Adamantanes can be identified by characteristic fragment ions of m/z 136, 135, 149, 163, 177 and 191 when GCXGC TOFMS is used (Li *et al.,* 2012). Anlai (2016), Anlai (2018), Moldowan (2015) used GCMSMS for Diamondoid analyses using fragment ions of m/z 187, m/z 135 and biomarkers such as m/z217, m/z216, m/z191 are used to ensure biomarker-diamondoid correlation to determine extent of cracking and if the samples are co- sourced (Figure 5). Peaks generated during diamondoid analysis are given in Figure 3.

Most hydrocarbon in the frontier basins are condensate rich, hence likelihood of high maturity source which can be detected using the diamondoid technique. In gas regimes, from vitrinite reflectance Ro 1.3 – 2.5, some parameters have been set using methyl adamantane index(MAI) , methyl diamantane index (MDI). MAI= 1-

MA/(1-MA+2-MA) When Ro ⩾ 1.9, MDI(Chen et al.,1996; Nasir, 2013) .

Types of Diamondoid Analyses

Diamondiod analyses in geochemistry are grouped into three different techniques. These include Quantitative Diamondiod Analysis (QDA), Compound Specific Isotope Analysis-Diamondoid (CSIA-D) and Quantitative Extended Diamondoid Analysis (QEDA). QDA has been applied widely in basin analysis projects, often with some unexpected results from poorly-understood basins, and also in mature well-studied basins that were thought to be well- understood. QDA uncovers deep sources contributing cracked oil to liquid-oil accumulations in basins where applied. The distribution of higher diamondoids in oils is determined by QEDA. Larger diamondoids, including tetramantanes, pentamantanes and cyclohexamantane, could be isolated to the extent that quantitative analysis can be accomplished. Extremely biodegraded oil is also determined by QEDA. CSIA-D is a complementary analysis to QEDA, much like isotope analysis of individual biomarkers or alkanes, and subsequently, a complementary method accompanying biomarker fingerprints in correlation studies.

(1) D15 1-Methyladamantane (I.S.)
(2) 1-Methyladamantane
(3) 2-Methyladamantane
(4) 1-Ethyladamantane
(5) 2-Ethyladamantane
(6) Adamantane
(7) D16 Adamantane (I.S.)
(8) 1,3-Dimethyladamantane
(9) 1,4-Dimethyladamantane (1)
(10) 1,4-Dimethyladamantane (2)
(11) 1,2-Dimethyladamantane
(12) 4-Methyldiamantane
(13) D3 1-Methyldiamantane (I.S.)
(14) 1-Methyldiamantane
(15) 3-Methyldiamantane
(16) 4-Ethyldiamantane
(17) 1-Ethyldiamantane
(18) 3-Ethyldiamantane
(19) Diamantane
(20) D4 Diamantane (I.S.)
(22) Hopane
(23) 4,9-Dimethyldiamantane
(24) 1,4+2,4-Dimethyldiamantane
(25) 4,8-Dimethyldiamantane
(26) 3,4-Dimethyldiamantane
(27) Cholane (I.S.)
(28) C29 aaa 20R Sterane
(29) 9-Methyltriamantane
(30) Triamantane
(31) D4 Triamantane (I.S.)
(32) Tetramantane-1
(33) Tetramantane-2
(34) Tetramantane-3
(35) C27 diasterane 20S

Figure 3: Peaks generated during Diamondoid Analysis.

Case Studies

Success stories on the application of diamondoid analyses to several basins have been documented in various literatures and some are not yet published. A few of these basins are highlighted below.

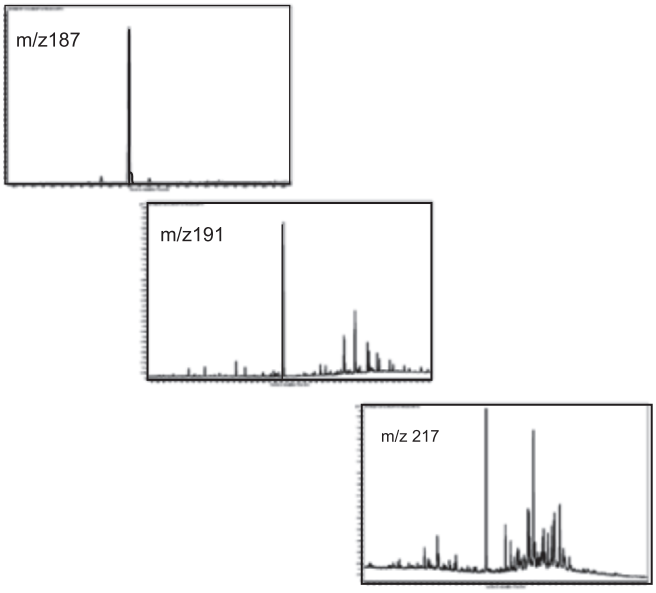

Figure 4: Typical Chromatograms with mass to charge ions used in diamondoid and biomarker analyses for correlation.

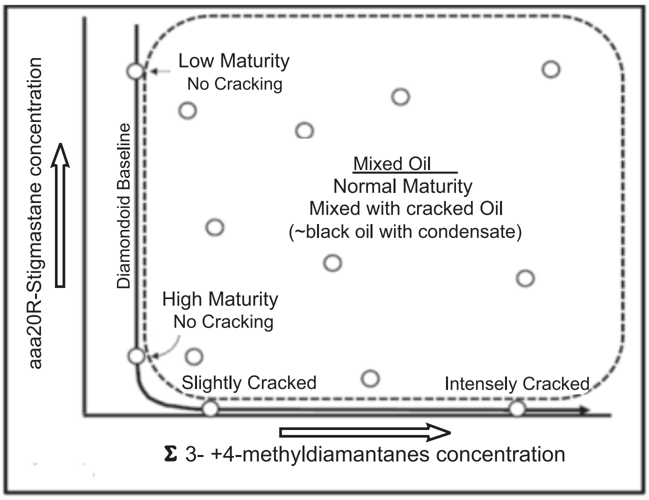

Figure 5: Diamondoid-Biomarker Correlation (Dahl *et al.,* 1999).

Niger Delta Basin

The Niger Delta basin is a prolific tertiary system in Nigeria consisting of kerogen type II and III source rock predominant in Akata formation and reservoir in Agbada formation (Reijers, 2011). The basin has sediment fill of 9-12km and total area of 300km2. Some studies have shown possibilities of cretaceous sources for the Niger Delta Basin along with the Akata Shale (Samuel *et al.,* 2009; Esugbue, 2016). Experiments/ Researches carried out on Niger Delta oil samples using diamondoid analysis have been able to delineate the cretaceous source contribution to the Niger Delta (Figure7). Abrakasa & Nwankwoala (2019) studied adamantane and methyl adamantine in thirteen oil samples from oil fields in Niger Delta using GC-MS m/z 136 and m/z 135. They further used the Methyl Adamantane Index (MAI) to assess maturity of the oil and correlation plot of 1-MA/nC11 to nC17+nC18/(Pr+Ph) to assess biodegradation

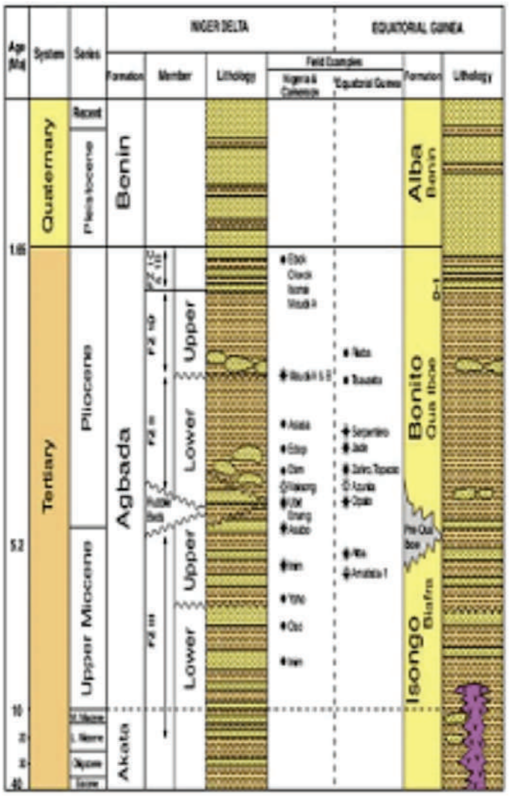

Figure 6: Stratigraphy of Niger Delta (Ndip et al., 2018).

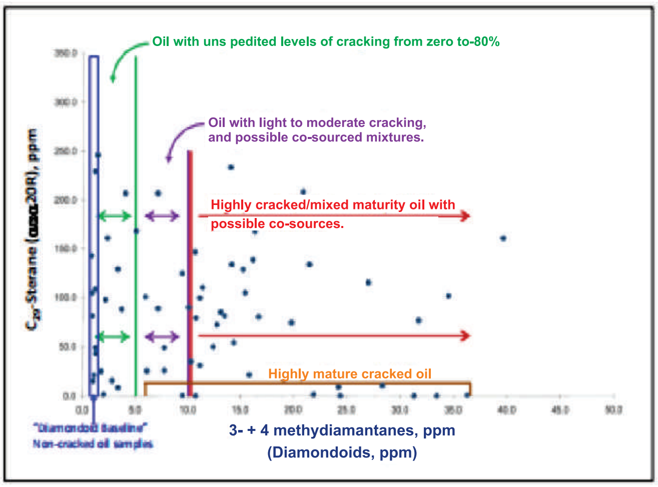

Figure 7: Quantitative Diamondoid Analyses on 63 Samples from Niger Delta, the highly mature cracked oil are from deep sources likely to be cretaceous (Moldowan, 2017).

Bornu Basin

Bornu Basin located in North East Nigeria is a large intracratonic basin, formed during the West and Central Africa Rifting System(WCARS), when the breakup of Gondwana and the opening of the South-Atlantic Ocean and the Indian Ocean occurred at about 120-130 Ma (Hamza, 2012). The basin covers one tenth of the Chad Basin and is filled with marine sediment of cretaceous origin (Olabode *et al.,* 2015). The source rock for the basin is suspected to be Fika Shale while the reservoir rock is likely the Bima sandstone.Some outcrop samples from Bornu Basin closer to the border with Benue Trough had been analysed using Quantitative Diamondoid Analysis and result showed possibilities of existence of deeper sources (FES, 2017).

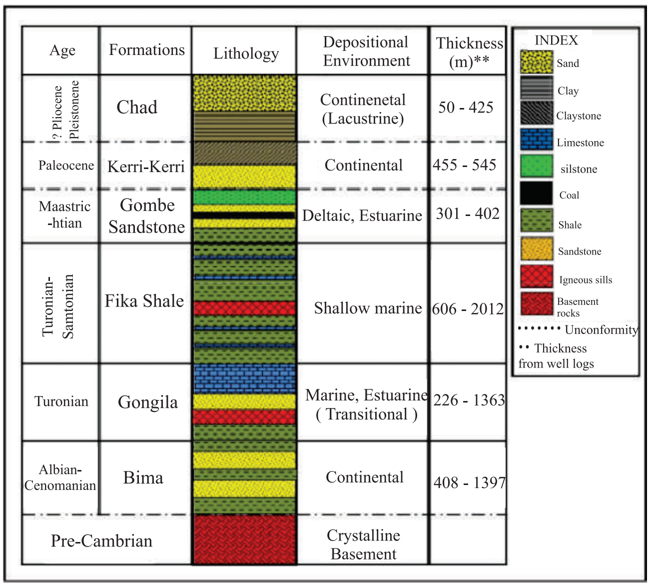

Figure 8: Stratigraphy of Bornu Basin (Adebanji et al., 2014).

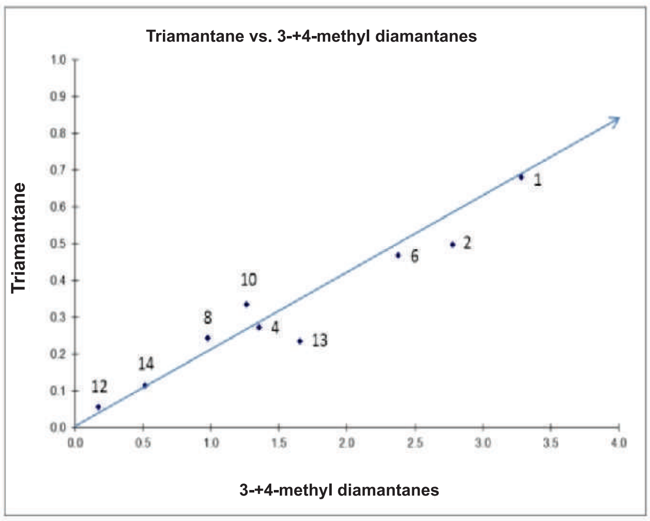

Figure 9: Diamondoid Source Correlation Plot of samples from Bornu Basin (FES, 2017).

Case Studies from other Countries

Tano Basin, Ghana

Tano basin, located in the south western part of Ghana is a cretaceous wrench which resides in between the Romanche and St Paul transform faults (Tetteh, 2016). There are three source rock plays in the Tano basin: Upper Albian source rock, Cenomanian source rock and Turonian source rock all made up of shales deposited due to the creation of the pull-apart basin. Daimondoid Analyses have been carried out on samples from Ghana and this was able to distinguish sources and correlate them. Figure 11 shows the result of Quantitative Extended Diamondoid Analyses (QEDA) that differentiated Saltpond Oil from Jubilee Oil and Onshore Tano Seeps. According to Moldowan (2017) the Jubilee oil has been correlated with marine Cretaceous source rocks; whereas, the Saltpond oils have been linked with Devonian source rocks. The Late Cretaceous (Turonian-Coniacian) sources suggested for the Jubilee Oil (Tetteh, 2016), if proven contributors in Nigeria, would have a profound effect on future exploration plays in the offshore. QEDA could be the key to determining such a "hidden" oil source that would best be expressed in the thermally recalcitrant diamondoids.

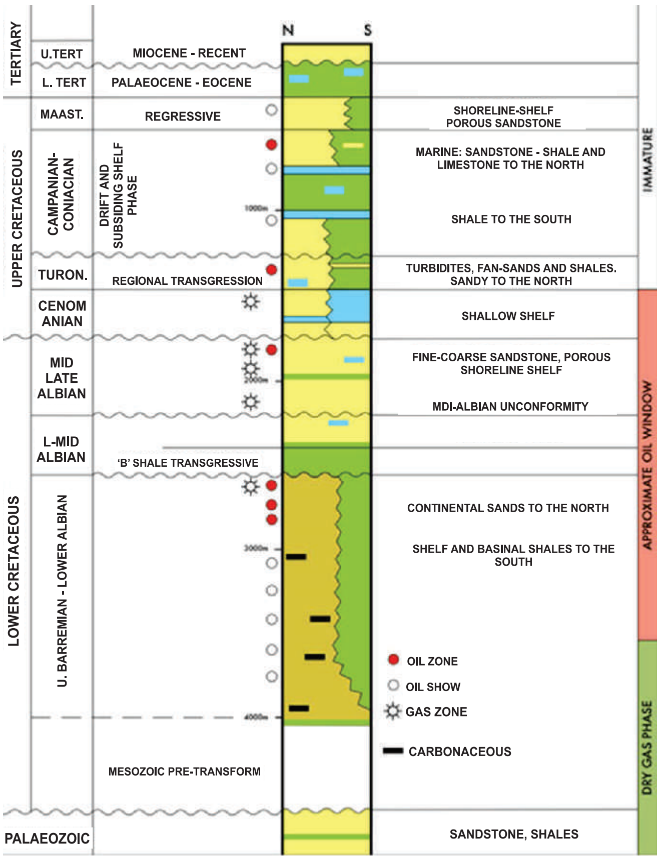

Figure 10: Stratigraphy of the Tano Basin (Amoo, 2014; Tetteh, 2016; Clontarf, 2019).

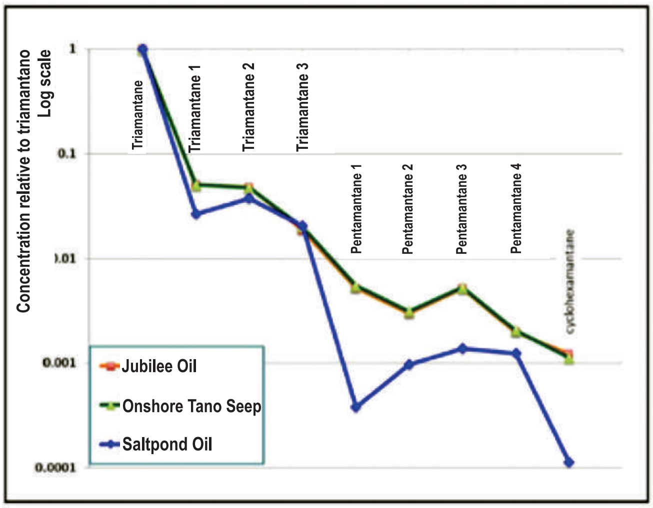

Figure 11: Quantitative Extended Diamondoid Analyses on samples from Ghana (Moldowan, 2017).

Williston Basin, North America

Williston basin is a prolific and large intracratonic basin that spans from East Montana through North and South Dakota to South Saskatchewan, Canada. Williston basin filling occurred predominantly in the Palaeozoic era with accumulation of sandstone, siltstone, shales, evaporite, limestone and dolomite. The Bakken formation have been investigated by Pallasto et al (2015) where they showed the formation to be rich in hydrocarbon. Part of the successes that led to more discoveries in the Williston Basin was due to the integration of Diamondoid Analysis for the detection of deeper sources and the correlation of reservoir with the deep source. Moldowan (2017) carried out CSIA-D on samples from the Canadian part of Williston Basin (Figure 13).

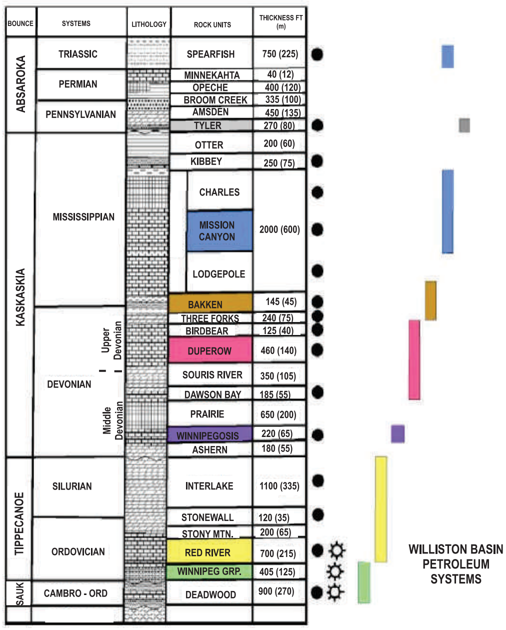

Figure 12: Stratigraphy of the Williston Basin (Pollastro, 2013).

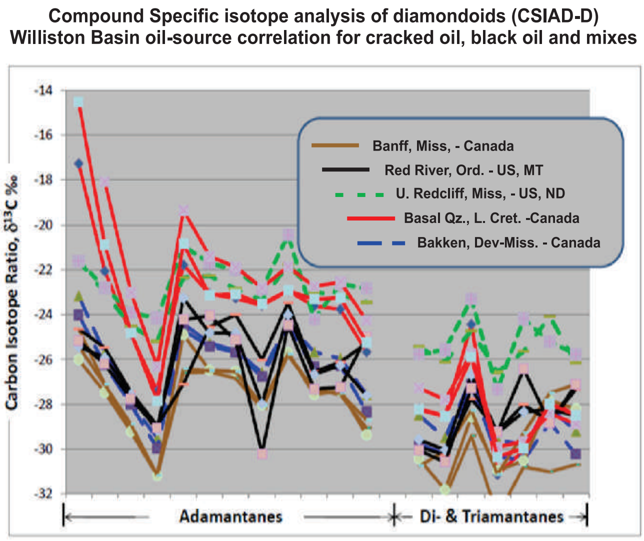

Figure 13: Result of Compound Specific Isotope Analysis – Diamondoid (CSIA-D) on samples from Williston Basin, Canada (Moldowan, 2017).

CONCLUSIONS

Diamondoid have indeed proven to be an indispensable geochemical tool in exploration based on the aforementioned successes recorded.

Diamondoid would help unravel the undiscovered deep source rocks and reservoirs in the frontier basins of Nigeria.

Overall, integrating diamondoid geochemical tool in exploration activities of all the frontier basins would help achieve value addition to Nigeria's hydrocarbon reserve.

RECOMMENDATION

It is highly recommended to consider using diamondoid technology as an important geochemical parameter for exploration activities.

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