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RECONSTRUCTION OF THE PRIMARY ORGANIC PRODUCTIVITY OF CAMPANO-MAASTRICHTIAN SHALES OF NKPORO AND APTIAN-ALBIAN AWI FORMATIONS, CALABAR FLANK USING PIGMENT YIELD INDEX

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ABSTRACT

Purpose. To derive the paleo organic productivity exemplified by the pigment yield index in order to resolve the controversy on the petroleum potentials of Nkporo and Awi Formations in Calabar Flank, Southeastern Nigeria.

Methods. Twenty outcrop samples each from Nkporo and Awi Formations in Calabar Flank, Southeastern Nigeria were analysed for total organic carbon, soluble organic matter and pigment richness by Walkley Black method, soxhlet extraction and column chromatography in order to derive the pigment yield index associated with paleo organic productivity which is needed to assess the petroleum potential.

Findings. The geochemical analysis results of the total organic carbon, soluble organic matter, pigment richness showed that the outcrop samples had organic matter and hydrocarbon above the threshold required for petroleum generation and that the samples were deposited in predominantly anoxic setting.

Originality. The results of the organic carbon (TOC) for Nkporo Formation – 1.51 to 4.30%; Awi Formation – 0.75 – 6.40%; soluble organic matter (SOM) Nkporo Formation – 110.5 to 4550.0 ppm and Awi Formation – 288.50 – 2664.25 ppm attest to the above threshold petroleum potential. Ni/Ni + VoP ratios for both Nkporo and Awi Formations < 0.5 to > 0.6 showed anoxic, paralic and oxic settings, though predominantly anoxic, excellent for organic matter preservation. Pigment yield indices of 5.88 and 5.58 equally depicts high petroleum potentials for the two Formations.

Practical implications. These results can be used to assess the paleo bioproductivity which is primary in petroleum generation, and in the resolution of controversies in the comparative assessment of two or more basins or formations.

Keywords: *porphyrins, pigment yield index, outcrops, paleo organic productivity, petroleum potential*

1. INTRODUCTION

The series of exposed sedimentary sequences from the Cretaceous to Tertiary basins of Southern Nigeria observed from road cuts or from sections exposed through gully erosion or quarries are characterized by diverse features which reflect changing depositional conditions and the complex interplay of sedimentation and tectonics (Essien, Ukpabio, Nyong, & Ibe, 2005). Thus, studies of these outcrops provide sufficient data for the evaluation of parameters that may be used for the geochemical interpretation of the conditions that prevailed before and during their deposition (Demaison & Moore, 1980).

Numerous studies have been carried out on these exposed sedimentary sequences from the Cretaceous to Tertiary basins in Calabar Flank, Southern Nigeria. For example, (Edet & Nyong, 1994) used palynoflora to reconstruct the paleobiogeography of Nkporo shales environment using microscopic examination of miospores. This may not have reflected the true paleobiogeography because of the possibility of the observed flora existing somewhere else and probably transported after death to the basin of deposition (allochthonous).

Consequently, the integration of some geochemical information into the geological framework provided a lot more information. Essien, Ukpabio, Nyong, & Ibe (2005); Ekpo et al. (2013) and Ibe & Osabor (2014) have employed the geochemical approach in the assessment of the paleoenvironment, petroleum potential and reconstruction of the sedimentation pattern of the sedimentary formations of the Calabar Flank. However, there appear to be some conflicting reports on the results of the assessment of the petroleum potentials of the Campano-Maastrichtian and Aptian-Albian Formations, Nkporo and Awi Formations. Essien, Ukpabio, Nyong, & Ibe (2005) reported Nkporo Formation as having a higher petroleum potential than Awi Formation while (Ekpo et al., 2005) reported Awi Formation as having a higher petroleum potential than Nkporo Formation. Which of the two has the higher petroleum potential is the object of controversy by the different workers.

We have attempted here to reconstruct and use the paleo organic productivity exemplified by the pigment yield index to resolve the controversy.

2. DESCRIPTION OF THE STUDY AREA

2.1. Location

The region known as the Calabar Flank was first recognized as the area covering Southern Nigeria sedimentary basin characterized by NW – SE trending crustal block faults (Murat, 1972). This was later modified and the structural location of the area was defined as being bounded by the Oban Massif in the north, the Calabar hinge line in the south and the Cameroun volcanics in the east. To the west, a NE – SW trending fault delineates the extent of the Calabar Flank and separates it from the Ikpe platform and adjacent basins of the southern Benue Trough (Fig. 1) (Nyong & Ramanathan, 1985).

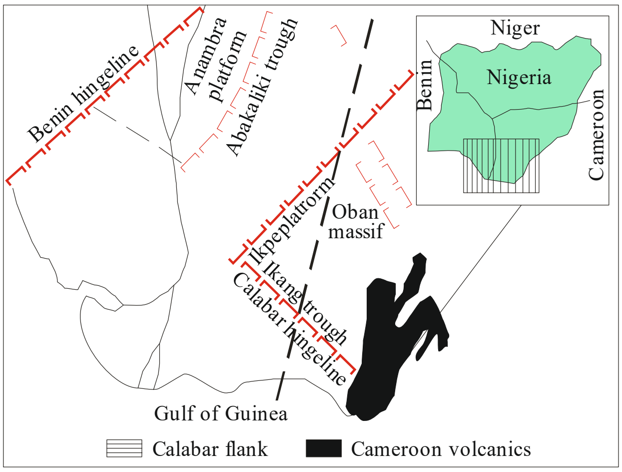

Figure 1. Structural and location map Calabar Flank and adjacent basins of the southern Benue Trough (adapted from Nyong & Ramanathan, 1985)

The Calabar Flank represents the northern most extension of a series of continental margin-sag basins, such as those of Cameroun, Gabon, Congo and Angola, which lie along the South Atlantic; possess similar NW – SE trending basement horst and graben structures (Reijers & Petters, 1987); which were initiated during the late Jurassic-Early Cretaceous rift phase prior to the final separation of Africa from South America (Reyment, 1980; Reyment & Dingle, 1987).

2.2. Sedimentation and stratigraphy

For most of the depositional history of the area, the Ikang Trough was an intracratonic mobile depression which accumulated mostly shales while the Ituk high, a relatively stable submarine platform, received predominantly limestone (Murat, 1972; Reijers & Petters, 1987).

According to (Nyong & Ramanathan, 1985), the Calabar Flank contains about 1000 m of Cretaceous sediments in outcropping sections. A comprehensive stratigraphic sequence (Fig. 2) compiled from (Adeleye & Fayose, 1978; Ramanathan & Kumaran, 1981; Petters, 1982), presents the following succession:

– Basal fluvio deltaic grits and sandstones of the Awi Formation (Aptian-Albian);

– Limestones and calcareous sandstones of the Mfamosing Formation (Mid-late Albian);

– Alternating limestone and shales of the Odukpani Formation (Cenomanian);

– Shales and marls of the Eze-Aku Formation (Turonian);

– Marls of the Awgu Formation (Coniacian);

– Carbonaceous shales of the Nkporo Formation (Campanian- Maastrichtian).

Chrono-stratigraphy	Litho-stratigraphy	
Maastrichtian	Nkporo shale	
Campanian		
Santonian		
Coniacian	Awgu formation	
Turonian	Eze-aku formation	
Cenomanian	Odukpani formation	
Albian	Asu rivers group	Miamosing Fm / Awi Fm
Aptian		

Figure 2. Lithostratigraphic succession on the Calabar flank

3. MATERIALS AND METHODS

The shales were collected from out-crops along Calabar-Odukpani Road and Calabar-Itu Highway as specifically indicated in the location map of the study area (Fig. 3). The weathered surfaces were dug out and samples collected at depths of 0.5 to 1.0 m with the help of a digger (Grosjean, Adam, Connan, & Albrecht, 2004). Precautions on the use of digger and spade were adopted. The samples were freeze dried before pulverizing and sievingto 200 mesh (0.04 µm).

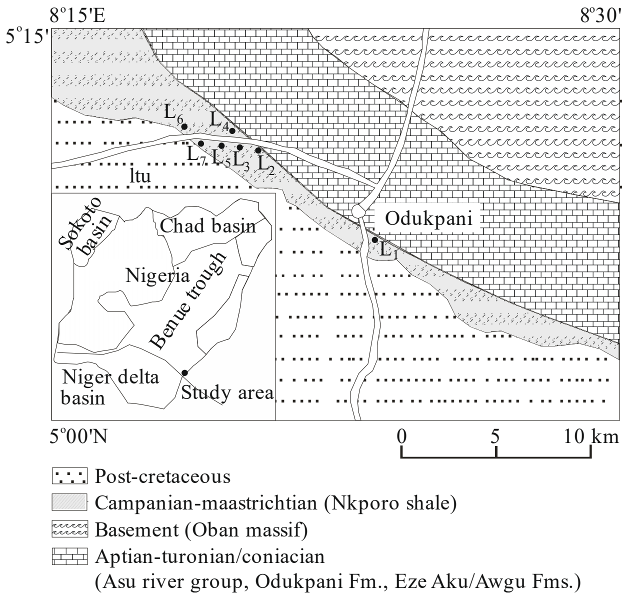

Figure 3. Geological map of the Calabar Flank showing sample location

3.1. Extraction of soluble organic matter

300 g of the sieved samples were placed in a thimble in a soxhlet extractor and extracted for 48 hours with re-distilled acetone and methanol (9:1 v/v) (Hodgson, Flores, & Baker, 1969). This process was precluded from sunlight with black curtains over the windows of the laboratory; essential precaution taken when dealing with porphyrins.

The extract was first quantified before been chromatographed on silica gel activated at 120°C for two hours. The nickel porphyrins were recovered through elution with 50% dichloromethane in hexane while the vanadyl porphyrins were recovered with 100% dichloromethane (Wignal & Twitchet, 2002). Purification was attained by repeated chromatography over neutral alumina developed with benzene in cyclohexane (Fractionation was carried out fairly rapidly to avoid the possibility of generating artifacts since deoxophylloerthroetioporphyrin (DPEP) undergoes hydroxylation at the isocyclic ring during chromatography on silica gel (Essien, Ukpabio, Nyong, & Ibe, 2005). The solvent for each fraction was carefully removed in a rotatory-evaporator.

Samples were dissolved in dichloromethane and analyzed using HACH DR 3000 SPECTROPHOTOMETER ULTRAVIOLET VISIBLE in a quartz cell (1 cm path length). Concentrations were calculated using the Beer-Lambert formula. Molar extinction coefficients of 26.14 and 34.82, at wavelengths of 571 and 551 nm for vanadyl and nickel porphyrins respectively (Moldowan, Sundararaman, & Schoell, 1986).

3.2. Determination of the total organic carbon (TOC)

The total organic carbon was evaluated by the method of (Walkley & Black, 1934). The crushed sample (1.0 g) was placed in a 500 ml flat bottomed flask and 1 N or 0.17 M potassium heptaoxodichromate (vi) (10 cm^3) was added to it followed by the addition of concentrated tetraoxosulphate (vi) acid (20 cm^3) through two different burrettes to oxidize the organic material. The mixture was swirled for about a minute for intimate mixing and allowed to stand for about 30 minutes.

After 30 minutes, the solution was diluted with distilled water (200 cm^3). 85% syrupy tetraoxophosphate (v) acid was added to prevent the iron (iii) ions, which are formed in the course of titration of iron (ii) salt from oxidizing the diphenylamine prematurely. Sodium fluoride was added immediately to bind the refractory metals like calcium, magnesium etc. The solution was titrated with 0.5 N (0.25 M) ferrous ammonium tetraoxosulphate (vi) solution after the addition of diphenylamine indicator. The colour changes were greenish brown to blue-black and then bright green at the end point.The percentage organic carbon was calculated using the equation below:

$$\%\ \text{organic carbon} = 10\left(1-\frac{T}{S}\right)\cdot F, \qquad (1)$$

where:

T – sample titration;

S – standard or blank titration;

F – factor derived as follows $F = \dfrac{1.0\cdot 12\cdot 1.72\cdot 100}{4000\,w}$,

where:

W – weight (in grams) of crushed sample used.

4. RESULTS AND DISCUSSION

The values of the total organic carbon content (% TOC), solube organic matter, nickel & vanadium porphyrin concentrations and the pigment yield indices are shown on Table 1 while the variation of pigment yield index with the depositional settings are shown on Figures 4 and 5.

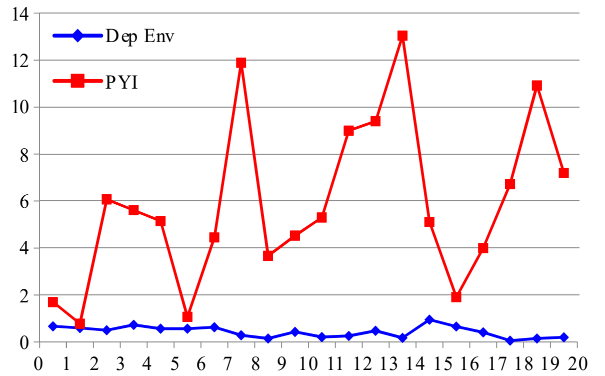

Figure 4. Pigment yield index versus depositional setting of Nkporo Formation

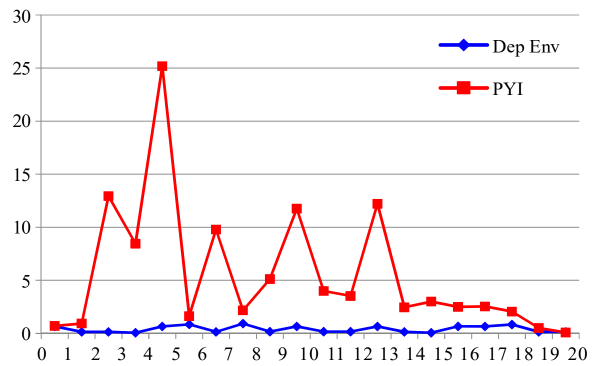

Figure 5. Pigment yield index versus depositional setting of Awi Formation

4.1. Organic richness and depositional setting

The % TOC values of 1.51 – 4.30% for Nkporo shale samples depicts a very good organic carbon accumulation and the % TOC values of 0.75 – 6.44% for Awi formation equally showed that it has very good potential for hydrocarbon generation. Both are very well above the lower limit of 0.5% by weight of organic matter which is equivalent to about 0.4% by weight of organic carbon generally believed to be required for petroleum generation before expulsion can be effected (Stein, Rullkotter, Kalkreuth, & Welte, 1987). It should be noted that low concentration of organic carbon favours dispersion rather than accumulation.

The soluble organic matter of Nkporo shales ranges from 110.5 – 4550 ppm. Most of the sampled shales of this formation are above the known minimum of 500 ppm required for commercial amount of hydrocarbon generation and have high SOM/TOC ratios. The rocks of Awi Formation have SOM values ranging from 288.50 – 2664.25 ppm equally having most of the samples above 500 ppm minimum earlier stated to be the requirement for commercial generation of petroleum; and have high SOM/TOC ratios as well, though the values of the ratios are comparatively lower than those of Nkporo formation.

Table 1. Results of the analyzed geochemical parameters

Sample name/No.	% TOC	SOM, ppm	SOM/TOC	VoP, ppm	NiP, ppm	NiP/ NiP + VoP	PYI
Nkporo 1	2.76	360.00	130.43	20.45	40.84	0.67	1.70
Nkporo 2	3.86	1070.00	277.20	15.34	23.50	0.60	0.78
Nkporo 3	3.01	825.00	274.09	100.32	101.36	0.50	6.07
Nkporo 4	2.94	927.00	315.31	111.20	79.73	0.73	5.61
Nkporo 5	2.74	2908.00	1061.31	80.57	107.49	0.57	5.15
Nkporo 6	1.51	576.00	381.46	30.52	40.65	0.57	1.07
Nkporo 7	3.23	1065.00	329.72	50.31	87.43	0.63	4.45
Nkporo 8	3.10	727.00	234.52	274.00	109.54	0.29	11.89
Nkporo 9	2.74	1672.00	610.22	114.23	19.62	0.15	3.67
Nkporo 10	3.23	2284.00	707.12	79.51	60.72	0.43	4.53
Nkporo 11	3.10	757.60	244.39	135.10	36.00	0.21	5.30
Nkporo 12	3.68	2850.80	774.67	180.93	62.86	0.26	9.00
Nkporo 13	4.30	573.00	133.26	114.30	103.57	0.48	9.40
Nkporo 14	2.79	842.50	301.97	380.50	86.35	0.18	13.04
Nkporo 15	2.37	303.30	127.97	10.10	205.62	0.95	5.11
Nkporo 16	3.11	110.50	35.53	20.89	40.45	0.66	1.91
Nkporo 17	1.78	4550.00	2500.00	131.69	92.04	0.41	4.00
Nkporo 18	2.60	4212.50	1620.19	243.62	14.78	0.06	6.72
Nkporo 19	3.34	1704.00	510.18	276.76	50.47	0.15	10.92
Nkporo 20	3.22	2277.00	707.14	179.00	44.62	0.20	7.20
Awi 1	1.45	288.50	198.97	16.44	32.60	0.66	0.71
Awi 2	1.99	589.00	295.95	40.05	7.07	0.15	0.94
Awi 3	6.44	2520.00	391.30	171.40	30.24	0.15	12.94
Awi 4	6.41	2164.50	337.68	123.37	8.66	0.07	8.46
Awi 5	6.12	2420.50	395.51	137.90	273.50	0.66	25.18
Awi 6	2.32	875.50	377.37	10.51	59.53	0.85	1.63
Awi 7	5.56	2200.50	395.77	149.63	26.41	0.15	9.79
Awi 8	2.33	1532.33	657.65	6.13	87.34	0.93	2.17
Awi 9	2.89	2569.50	889.10	146.43	30.83	0.17	5.12
Awi 10	4.60	1503.75	326.90	85.71	169.84	0.66	11.76
Awi 11	4.26	1361.90	319.70	77.63	16.34	0.17	4.00
Awi 12	1.92	2664.25	1387.63	151.84	31.97	0.17	3.53
Awi 13	4.40	1632.75	371.08	93.07	184.50	0.66	12.21
Awi 14	2.72	1129.67	415.32	76.82	13.56	0.15	2.46
Awi 15	2.32	2120.75	914.12	120.88	8.48	0.07	3.00
Awi 16	1.94	754.30	388.81	43.00	85.24	0.67	2.50
Awi 17	1.65	905.00	548.48	51.60	102.27	0.66	2.54
Awi 18	2.67	1001.50	375.09	12.02	65.10	0.84	2.06
Awi 19	1.35	468.00	346.67	31.82	5.60	0.15	0.51
Awi 20	0.75	150.35	200.47	10.22	1.80	0.15	0.09

The depositional environmental indicator, Ni/Ni + VoP ratios for both Nkporo and Awi formations show predominantly anoxic setting (< 0.5) with few paralic (≤ 0.6) and oxic (> 0.6) settings. The inferences from the above information are in agreement with the works of the previous authors already sited in the introduction.

4.2. Pigment yield index

The demise of tetrapyrrole pigment with increasing depth, or geologic age, within a profiled sediment apparently indicates a major pathway of chlorophyll diagenesis. In regions where the sediment-water interface is below the photic zone, the initial form of tetrapyrrole input is pheophytin. Loss of pheophytin, and its derivatives is apparently controlled by oxidation in the water column and at the sediment/water interface and by establishment of a negative Eh with burial (Baker & Louda, 1980).

Yield of tetrapyrrole pigment, as a component of the organic carbon fraction, is traced by the generation of a pigment yield index, PYI, obtained by normalising the pigment yield with the percent total organic carbon. Relating pigment yield to the organic carbon content of sediment, especially on a dry weight basis, now allows a much easier method in tracing trends and even rates of pigment loss from the fossil record.

Both the Nkporo and Awi Formations samples have sufficient pigment yield indices, greater than 4 which depicts sufficient tetrapyrolepreservation (Didyk, Simoneit, Brassel, & Eglinton, 1978). The shale samples of Nkporo Formation had mean pigment yield index, 5.88 ± 0.35 while the samples of Awi Formation had pigment yield index of 5.58 ± 0.30. This preservation may have been possible because of the sufficient anoxic settings (Table 1 and Figs. 4, 5). Therefore, from the point of view of tetrapyrole preservation both have almost equal potentials for the generation of petroleum with maturity.

However, rapid sedimentation, reducing conditions, and sediment stability lead to a reduction-thermal stress pathway wherein DPEP-porphyrins are generated and then thermally altered to yield transalkylatedmetallo-DPEP-etio mixtures. Conversely, sediment reworking, oxic conditions, and input of coarse-grained sediment leads to initial formation of oxidized tetrapyrroles, such as purpurins and the chlorin-*e* and -*p* series, followed by essentially complete removal from the fossil record by presently unknown mechanisms (Baker & Louda, 1980).

5. CONCLUSIONS

The Nkporo and Awi sedimentary samples analysed for this study revealed potential hydrocarbon pools from the results of basic geochemical parameters in agreement with previous workers. With the pigment yield indices of 5.88 ± 0.35 and 5.58 ± 0.30 for Nkporo and Awi formations respectively, both have sufficient paleo bioproductivity which placed them on almost equal status of petroleum generation potential.

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РЕКОНСТРУКЦІЯ ПЕРВИННОЇ ОРГАНІЧНОЇ ПРОДУКТИВНОСТІ КАМПАНО-МААСТРИХТСЬКИХ СЛАНЦІВ У НКПОРО ТА АПТСЬКО-АЛЬБСЬКИХ АВІ ФОРМАЦІЙ В КАЛАБАР ФЛАНК ІЗ ВИКОРИСТАННЯМ КОЕФІЦІЄНТА ВИДІЛЕННЯ ПІГМЕНТУ

К. Ібе, К. Огвуче

Мета. Оцінка палеоорганічної продуктивності кампан-маастрихтських сланців на основі коефіцієнта виділення пігменту для порівняння нафтового потенціалу формацій Нкпоро і Аві в Калабар Фланк (Південно-Східна Нігерія).

Методика. Двадцять зразків, відібраних з місця виходу породи на поверхню в районі формацій Нкпоро і Аві в Калабар Фланк, південно-східна Нігерія, були проаналізовані з точки зору вмісту загального вуглецю, розчинної органічної матерії, насиченості пігментом за допомогою методики Уолклі Блека, екстракції в апараті Сокслета, колоночної хромотографії з метою отримання коефіцієнта виділення пігменту. Даний показник пов'язаний з палеоорганічною продуктивністю та дозволяє оцінити нафтовий потенціал.

Результати. Геохімічними дослідженнями встановлено, що за процентним складом, зразки з формації Нкпоро містять органічного вуглецю (ОВ) від 1.51 до 4.30%; формації Аві 0.75 – 6.4%; розчинної органічної матерії (РОМ) в зразках з формації Нкпоро 110.5 – 4550 год/млн і формації Аві – 288.50 - 2664.25 год/млн, що свідчить про раніше згаданий потенціал утворення нафти. Визначено співвідношення Ni/Ni + VoP в зразках з формацій Нкпоро і Аві, яке склало < 0.5…> 0.6, що свідчить про безкисневих, паралічних та в меншій мірі, кисневих умовах, що дуже сприятливо для збереження органічної матерії. Величини коефіцієнта виділення пігменту 5.88 і 5.58 вказують на рівно високий потенціал утворення нафти в обох формаціях.

Наукова новизна. Вперше виявлено умови формування нафтового потенціалу формацій Нкпоро і Аві (Південно-Східна Нігерія) за вмістом загального вуглецю, розчинної органічної матерії, насиченості пігментом. Доведено, що зразки, взяті з місця виходу породи на поверхню, містять більше органічної матерії і вуглеводнів, ніж мінімальна кількість, яка необхідна для утворення нафти, і що вони залягали переважно в безкисневому середовищі.

Практична значимість. Результати даного дослідження можуть бути використані для оцінки палео біопродуктивності, головного показника утворення нафти, і при порівняльній оцінці двох або більше геологічних басейнів або формацій.

Ключові слова: *порфірини, коефіцієнт виділення пігменту, відслонення породи, палеоорганічна продуктивність, потенціал утворення нафти*

РЕКОНСТРУКЦИЯ ПЕРВИЧНОЙ ОРГАНИЧЕСКОЙ ПРОДУКТИВНОСТИ КАМПАНО-МААСТРИХТСКИХ СЛАНЦЕВ В НКПОРО И АПТСКО-АЛЬБСКИХ АВИ ФОРМАЦИЙ В КАЛАБАР ФЛАНК С ИСПОЛЬЗОВАНИЕМ КОЭФФИЦИЕНТА ВЫДЕЛЕНИЯ ПИГМЕНТА

К. Ибе, К. Огвуче

Цель. Оценка палеоорганической продуктивности кампан-маастрихтских сланцев на основе коэффициента выделения пигмента для сравнения нефтяного потенциала формаций Нкпоро и Ави в Калабар Фланк (Юго-Восточная Нигерия).

Методика. Двадцать образцов, взятых из места выхода породы на поверхность в районе формаций Нкпоро и Ави в Калабар Фланк, юго-восточная Нигерия, были проанализированы с точки зрения содержания общего углерода, растворимой органической материи, насыщенности пигментом при помощи методики Уолкли Блэка, экстракции в аппарате Сокслета, колоночной хромотографии с целью получения коэффициента выделения пигмента. Данный показатель связан с палеоорганической продуктивностью, и позволяет оценить нефтяной потенциал.

Результаты. Геохимическими исследованиями установлено, что по процентному составу, образцы из формации Нкпоро содержат органического углерода (ОУ) от 1.51 до 4.30%; формации Ави 0.75 – 6.4%; растворимой органической материи (РОМ) в образцах из формации Нкпоро 110.5 – 4550 ч/млн и формации Ави – 288.50 – 2664.25 ч/млн, что свидетельствует о ранее упомянутом потенциале образования нефти. Определено соотношение Ni/Ni + VoP в образцах из формаций Нкпоро и Ави, которое составило < 0.5…> 0.6, что свидетельствует о бескислородных, паралических и, в меньшей степени, кислородных условиях, что очень благоприятно для сохранения органической материи. Величины коэффициента выделения пигмента 5.88 и 5.58 указывают на равно высокий потенциал образования нефти в обеих формациях.

Научная новизна. Впервые выявлены условия формирования нефтяного потенциала формаций Нкпоро и Ави (Юго-Восточная Нигерия) по содержанию общего углерода, растворимой органической материи, насыщенности пигментом. Доказано, что образцы, взятые из места выхода породы на поверхность, содержат больше органической материи и углеводородов, чем минимальное количество, которое необходимо для образования нефти, и что они залегали преимущественно в бескислородной среде.

Практическая значимость. Результаты данного исследования могут быть использованы для оценки палео биопродуктивности, главного показателя образования нефти, и при сравнительной оценке двух или более геологических бассейнов или формаций.

Ключевые слова: *порфирины, коэффициент выделения пигмента, обнажения породы, палеоорганическая продуктивность, потенциал образования нефти*

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