18. ORGANIC GEOCHEMISTRY OF CRETACEOUS SHALES FROM DSDP SITE 398, LEG 47B, EASTERN NORTH ATLANTIC

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ABSTRACT

Cretaceous, organic-rich, dark-colored shales and mudstones were drilled in the Vigo Seamount area on DSDP Leg 47B. Despite their relative richness, their organic matter is predominantly derived from detrital material of continental origin. Some contribution of marine organic matter is apparent in the middle to upper Albian and Cenomanian sediments. An immature stage of evolution can be assigned to all the samples.

INTRODUCTION

The purpose of this study was to geochemically examine the organic matter of the Cenomanian-Aptian shales and siltstones cored on DSDP Leg 47B (Figure 1).

A pyrolysis assay on raw samples was performed, followed by a chloroform extraction. Elemental composition of kerogen and humic compounds fractionation were carried out to complete the description.

SAMPLING

Dark shales interbedded with siltstones were encountered in the Barremian-Aptian series (Unit 4C). They were overlain by other dark laminated to bioturbated shales (Unit 4B) and dark mudstones to claystones (Unit 4A) of Albian and Cenomanian ages (Figure 2).

Sixty-five samples were selected from the Aptian-Cenomanian interval (Cores 127 to 56). They correspond to an approximate spacing of one sample per core.

ANALYTICAL METHODS

The samples were freeze-dried, then ground in an Aurec pulverizer (grain size \cong 90 μ m). The succession of processes are schematized in Figure 3. Organic carbon was analyzed with a LECO apparatus. A pyrolysis assay was conducted on raw samples as a method of rapid source rock and maturation characterization (Espitalié et al., 1977). Equivalent parts of ground samples were then extracted with chloroform, followed by a thin-layer chromatography fractionation of the extract (Huc et al., 1976). From the insoluble residue of the rock, the total humic compounds were extracted and humic acids separated from fulvic acids (Huc et al., 1978). The hydrolyzable fraction of the residual insoluble part corresponds to humin and its non-hydrolyzable fraction to kerogen. Organic carbon content from total humic compounds and from fulvic fraction was analyzed with a Carmhograph Wosthoff apparatus. El-



Figure 1. Location of DSDP Site 398 in the Vigo Seamount area.

emental analysis of C, H, O, N, S, and Fe plus ash content were measured on kerogen, with values obtained by calculation on a mineral-free basis.

DETAILED RESULTS

Mineral Carbon

Most of the Albian and Aptian samples have a low content (<1.20 wt.%) of mineral carbon (Table 1). The interval between Section 56-4 and Core 60, of Cenomanian and Albian ages is richer, with 2.80 to 5.60 per cent of mineral carbon.



Figure 2. Stratigraphic position of cores from Holes 398A, B, and D.

Organic Carbon

The variation of organic carbon content from the Aptian-Cenomanian series (Table 1) corresponds to the results obtained from shipboard analysis.

From base to top, Aptian Cores 127 to 119 and 116 correspond to the richest interval (1.03 to 1.80 wt.%). The organic content is low in Aptian Cores 118 to 104 and 102 (0.31 to 0.87%), and increases again (0.93 to 1.57%) for Albian Cores 103 and 101 to 95. The values are rather low for the overlying Albian in the interval between Cores 93 to 82 (0.53 to 1.12%), and low again in Cores 80 to 60 (0.28 to 0.83%). The relatively carbonate-rich Albian-Cenomanian (Cores 60 to 56) shows the lowest content (0.24 to 0.44%), whereas the uppermost Section 56-1 (Senonian) show the largest organic content (5.89 and 8.72%) of the whole section.

Pyrolysis Assay and Organic Matter Characterization

As explained previously in the paper for Leg 47A (Deroo et al., Volume 47, Part 1), the method of pyrolysis described in Espitalié et al. (1977) allows the definition of three types of kerogen considered by Tissot et al. (1974) which can be used as references for organic matter characterization.

The organic-rich samples from Section 56-1 are located close to the evolution path of kerogen Type II (Figure 4), i.e., organic matter mostly derived from marine planktonic material. The underlying Cenomanian Sections 56-2 to 56-5, which are devoid of carbonates, and the carbonate-containing Albian Section 59-3 are located between the evolution paths of kerogen Types II and III.

This location suggests a variety of altered material of Type II or, more probably, a mixture of Types II and III as suggested by its relatively high oxygen index. The other samples (from Sections 56-4 and 57-2 to 59-2) have the largest oxygen index (250 to 500) and a low hydrogen index. The remaining part of the Early Cretaceous (Cores 60 to 127) also shows a high oxygen index (50 to 300) and a low hydrogen index (<50). All these samples can be related to Type III kerogen of continental origin. Moreover, their very low hydrogen index suggests an advanced stage of alteration due to reworking and/or oxidation.

Organic carbon and hydrogen indexes, expressed per weight unit of rock, were plotted in a column and compared to the geological section (Figure 5). The column allows the subdivision of the stratigraphic section into four main intervals, although organic matter of Type III is found throughout.

Interval 1 (Cores 127 to 116), with a content of one per cent of organic carbon, is the most representative of a Type III material (see Figure 4).

Interval 2 (Cores 118 to 102), in addition to a low organic carbon content of 0.50 per cent and less, shows the poorest hydrogen indexes of the series. They are characteristic of altered organic material, possibly by oxidation prior to deposition.

Interval 3 (Cores 103 to 80) is comparable to Interval 1 for Cores 103 to 96, 91, 97, and 81. Between Cores 92 and 80, a more altered material is also present which recalls Interval 2.

Interval 4 (Core 79 to Section 56-4), despite the same poor content of organic carbon (generally 0.50% or less), shows comparatively higher hydrogen indexes than Intervals 2 and 3 (Cores 92 to 80). It can be explained by some contribution of marine Type II material in addition to the main Type III.

In Sections 56-3 and 56-2 again a contribution of Type II must be present. Indexes comparable to those of Interval 1 are found in these sections, although the organic carbon content is lower. The same characteristics are reinforced for the uppermost samples from Sec-



Figure 3. Schematic analytical processes.

tion 56-1 where the contribution of Type II is slightly more important.

These intervals can be considered in the paleoenvironment frame defined in this volume by Sigal, de Graciansky, and Chamley et al. (Figure 5).

The first interval (1) refers to the IIa-b subdivision of Sigal and is of early to late Aptian age. These sediments are clastic series rich in higher plants fragments and mainly derived from detrital or redeposited material. The illite and mixed layers versus smectite ratios characterize the clay composition.

The second interval (2), of late Aptian age (IIc-d) where higher plant detritus is still abundant, would be less clastic than the first interval; the samples studied belong to hemipelagic material. Smectite predominates over illite and mixed layers.

The third interval (3) corresponds to the IIIa-b subdivisions, of early to middle Albian age, and contains dark to black shales with a rich content of higher plant detritus. Most of the samples again are hemipelagic. Illite and mixed layers become important and attapulgite is present.

The fourth interval (4) joins together the IV and V subdivisions of middle Albian to middle Cenomanian age. Their content of higher plant detritus is low. A significant content of carbonates (Figure 5) characterized the Vb subdivision where detrital or redeposited facies are also characteristic. Attapulgite is largely present in Cores 78 to 74; illite and mixed layers are less important compared to the third interval.

The intervals defined on the basis of pyrolysis were also compared with the palynological units defined by Habib (this volume) from palynology facies analysis. There are close relationships between our Interval 1 and the exinitic palynology facies of Unit II, as defined by Habib, and also between his Unit III, micrinitic pal-

Sample (Interval in cm)	Sub-Bottom Depth (m)	Mineral Carbon (wt. %)	Organic Carbon (wt. %)	Hydrogen Index (mg HC per g org. C)	Oxygen Index (mg CO ₂ per g org. C)	Hydrogen Index (mg HC per g rock)	Oxygen Index (mg CO ₂ per g rock)	Pyrolysis Temperature (°C)
56-1, 120-139 56-1, 120-139 56-2, 120-130 56-3, 0-10 56-3, 0-10	946.70 946.70 948.20 948.50 948.50	0.40 0.40 0.08 0.0 0.0	5.89 8.72 0.61 0.84 0.95	381. 303. 77. 132. 211.	29. 21. 66. 61. 38.	22.45 26.40 0.47 1.11 2.00	1.68 1.82 0.40 0.51 0.36	417. 413. 408. 394. 423.
56-3, 0-10 56-4, 34-60 56-5 57-2, 12-99 57-4, 25-43	948.50 950.34 951.50 957.22 961.25	0.0 2.80 0.24 3.92 4.12	1.61 0.20 0.19 0.44 0.24	185. 61. 152. 16. 30.	63. 450. 74. 241. 500.	2.97 0.12 0.29 0.07 0.07	1.01 0.90 0.14 1.06 1.20	398. 444. 439.
58-2, 80-85 59-2, 120-150 59-3, 135-146 60-2, 0-10 61-2, 0-10	966.80 976.70 978.35 985.00 994.50	5.60 3.56 4.52 4.24 2.52	0.42 0.26 1.70 0.83 0.36	30. 18. 143. 54. 29.	333. 419. 121. 149. 289.	$0.12 \\ 0.05 \\ 2.43 \\ 0.45 \\ 0.11$	1.40 1.09 2.05 1.24 1.04	423. 428. 430. 444.
62-1, 0-33 63-4, 98-135 65-1, 68-105 67-3, 20-40 68-3, 6-16	1002.50 1017.48 1031.68 1053.20 1062.56	1.20 0.96 1.20 0.88 0.60	0.39 0.42 0.28 0.50 0.56	24. 22. 48. 45. 33.	259. 271. 421. 258. 173.	0.09 0.09 0.14 0.23 0.18	1.01 1.14 1.18 1.29 0.97	439. 432.
69-2, 19-29 70-3, 70-80 71-2, 35-45 72-5, 35-45 73-5, 82-114	1070.69 1082.20 1089.85 1113.35 1123.32	0.48 0.44 0.52 0.44 0.80	0.48 0.53 0.46 0.54 0.45	35. 28. 29. 34. 18.	160. 206. 217. 172. 222.	0.17 0.15 0.13 0.18 0.08	0.77 1.09 1.00 0.93 1.00	432. 420. 436. 432.
74-3, 83-118 75-1, 29-62 76-1, 0-41 77-1, 120-130 73-3, 135-145	1129.83 1135.79 1145.00 1165.20 1177.85	0.56 0.80 0.52 0.44 0.52	0.41 0.50 0.48 0.56 0.75	18. 27. 24. 32. 28.	205. 236. 206. 120. 93.	0.07 0.14 0.11 0.18 0.21	0.84 1.18 0.99 0.67 0.70	417. 425.
79-3, 131-134 80-2, 70-80 81-4, 20-30 82-3, 0-37 84-5, 60-92	1187.31 1194.70 1206.70 1214.50 1237.10	0.28 0.44 0.56 0.56 0.64	0.59 0.49 1.32 0.53 0.89	27. 13. 38. 11. 13.	75. 186. 148. 208. 146.	0.16 0.06 0.50 0.06 0.12	0.44 0.91 1.95 1.10 1.30	413. 435. 420. 427.
85-3, 13-50 86-5, 0-38 87-5, 120-130 88-6, 120-130 89-4, 130-140	1243.13 1255.50 1266.20 1277.20 1283.80	0.28 0.68 0.88 0.80 0.92	0.76 0.80 1.01 0.86 0.70	17. 12. 25. 7. 12.	141. 178. 111. 141. 143.	0.13 0.10 0.25 0.06 0.09	1.07 1.42 1.12 1.21 1.00	437. 432. 427.
90-3, 125-135 91-2, 130-140 92-6, 120-130 93-1, 0-50 95-6, 110-120	1291.75 1299.80 1315.20 1316.00 1336.10	0.68 0.84 0.92 0.72 0.72	0.86 1.12 0.94 0.93 1.36	13. 24. 12. 11. 28.	117. 77. 104. 138. 86.	0.11 0.27 0.11 0.10 0.38	1.01 0.86 0.98 1.28 1.17	437. 432. 441. 436. 420.
96-5, 115-125 97-1, 130-140 99-3, 112-150 100-3, 10-50 101-4, 131-150	1342.15 1345.80 1367.62 1376.10 1388.31	1.00 0.92 0.68 7.36 0.48	1.57 1.19 0.98 1.00 0.93	20. 17. 17. 17. 15.	103. 85. 98. 83. 102.	0.32 0.20 0.17 0.17 0.14	1.62 1.01 0.96 0.83 0.95	427. 434. 436. 433. 430.
102-4, 120-130 103-3, 80-90 104-2, 106-116 106-3, 66-98 107-5, 10-44	1397.70 1405.30 1413.56 1433.66 1445.60	0.80 0.72 0.64 1.22 1.20	0.66 1.31 0.55 0.59 0.42	10. 16. 18. 19. 15.	155. 87. 120. 215. 264.	0.07 0.21 0.10 0.11 0.05	1.02 1.14 0.66 1.27 1.11	424. 429. 429. 427.
108-2, 13-33 111-5, 110-120 112-3, 120-130 113-4, 140-150 114-4, 130-140	1450.63 1484.60 1491.20 1502.40 1511.80	0.96 0.56 0.44 0.42 0.88	0.54 0.31 0.36 0.62 0.48	15. 14. 20. 16. 13.	302. 265. 211. 181. 152.	0.08 0.04 0.07 0.10 0.06	1.63 0.82 0.76 1.12 0.73	424.
116-3, 10-60 117-1, 10-67 118-4, 113-115 119-5, 24-44 120-5, 60-70	1528.10 1534.60 1549.63 1559.74 1569.60	0.96 1.16 1.08 0.68 0.80	1.03 0.87 0.59 1.10 1.10	21. 40. 18. 31. 27.	118. 114. 220. 55. 89.	0.22 0.35 0.11 0.34 0.29	1.22 0.99 1.30 0.61 0.98	415. 424. 424. 421. 426.
125-5, 122-150 126-4, 10-43 127-5, 80-110	1617.72 1625.10 1636.30	2.40 0.88 1.60	1.40 1.37 1.80	33. 22. 53.	79. 69. 67.	0.46 0.30 0.95	1.11 0.95 1.20	419. 422. 419.

TABLE 1 Location of Hole 398D Samples With Data From Carbon and Pyrolysis Assays



Figure 4. Pyrolysis assays with hydrogen and oxygen indexes (data related to organic carbon).

ynology facies, and our Interval 2. The lower part (Cores 103 to 94) of Interval 3 corresponds to the base of Habib's Unit IV, where phytoplankton is poor or absent. The overlying series, i.e., the upper part of Interval 3 plus the Interval 4, cannot be assigned directly to the other palynologic units. However, they can be related to a progressive increase of phytoplankton content from Unit V to Unit VI, while the palynomorphs and the sporomorphs decrease. The indication of a more marine contribution for organic material of Section 56-3 to 56-1 can be referred to the xenomorphic palynofacies of Unit VII.

The temperature of the top of the pyrolysis peak (Table 1) during pyrolysis assays corresponds to the oil immature zone in the 400° to 440° C range, to the main oil zone or mature zone from 440° to 460° C, and to the cracking or gas zone beyond 460° C. Almost all the observed temperature data are in the 417° to 437° C range; only one sample reached 444° C. The samples can be assigned to the immature stage of evolution as previously seen for nearly all other DSDP material from the Cretaceous of the Atlantic Ocean.

To conclude the pyrolysis assays study, it appears that the organic matter present in Cores 127 to 56 is principally derived from detrital material of continental origin. Some alteration prior to deposition can be invoked for the poorest intervals. Contribution of marine autochthnonous organic matter represents a significant change in Cores 79 to 56 of Albian and Cenomanian ages; that contribution is also present for the Senonian Section 56-1 which rests upon the Cenomanian deposits.

Humic Compounds

The preparation of a humic extract was performed for 10 selected samples. The content of humic compounds, i.e., humic plus fulvic acids, is expressed in Table 2 by the percentage of the total organic carbon incorporated in these compounds.

All the samples contain between 0.12 and 0.50 per cent of humic carbon related to the weight of rock. Except for the Senonian Section 56-1 and Albian Section 59-3, the content of humic carbon relative to total organic carbon is relatively high (16 to 50%). Huc and Durand (1977) have shown that the humic fraction of carbon constitutes a greater part (>15%) of immature Type III than of immature material of Types II and I composition. This is in agreement with the assignment of most Albian-Aptian samples to Type III material and also with some contribution of Type II material to the uppermost Albian and Senonian samples. The relatively low humic content for Sections 125-5 and 127-5 can probably be explained by some effect of thermal evolution, according to the same authors.

Kerogen Fraction

The same samples selected for study of the humic compounds were analyzed, plus one sample from Section 56-1. The preparation of kerogen by HCl and HF acidic treatment under a nitrogen atmosphere was followed by the elemental analysis of the kerogen. The data (Table 3) are plotted on a Van Krevelen diagram (Figure 6) where the three reference evolution paths I, II, and III are drawn.

Most samples analyzed can be referred to the hydrogen poor Type III. However Sections 56-1 and 59-3 stand out from the others due to slightly higher H/C ratios. That distinction is reinforced for the Section 56-1 material when a second sample (B) was prepared and analyzed. The elemental composition suggests a mixture of Type II and Type III material, at least for Section 56-1.

Some Albian-Aptian kerogens like those for Sections 96-5 and 104-2 show a very low hydrogen content, which suggests primary oxidation of the organic material on land, before transportation and deposition.

Chloroform Extracts

The 10 previous samples generally provided a low extract yield (35 to 12 mg) except for the Senonian Section 56-1 (388 mg). The extract to organic carbon ratio is always low (0.008 to 0.035). The extracts were fractionated using thin-layer chromatography. They show large NSO content (73 to 85%) except for the Section 81-4 sample which contains only 55 per cent of



Figure 5. Pyrolysis assays with vertical distribution of mineral and organic carbon and hydrogen indexes related to the weight of rock.

TABLE 2 Humic Compounds and Organic Carbon Content, Hole 398D

		Total	Humic Compounds			
Sample (Interval in cm)	Sub-Bottom Depth (m)	Organic Carbon ^a (wt. %)	Humic Carbon (% of rock)	Total Org. C (%)		
56-1, 120-139(A)	946.70	5.80	0.17	3		
59-3, 135-146	978.35	1.74	0.24	14		
60-2, 0-10	985.00	0.84	0.41	50		
76-1, 0-41	1145.00	0.60	0.12	20		
81-4, 20-30	1206.70	1.39	0.36	26		
96-5, 115-123	1342.15	1.59	0.50	32		
104-2, 106-116	1413.56	0.60	0.28	47		
118-4, 113-115	1549.63	0.56	0.13	24		
125-5, 122-150	1617.72	1.38	0.27	20		
127-5, 80-110	1636.30	1.81	0.30	16		

^aAfter CHCl₃ extraction.

NSO compounds. The saturated fraction varies from 7 to 18 per cent (Figure 7)

For all the samples, gas chromatography of total saturated and unsaturated hydrocarbons in the C15 to C₃₀ interval reveals a normal alkanes distribution with an odd to even carbon number predominance in the C21 or C23 to C29 range. Continental input related to higher plants explains that distribution. However, a C16 to C18 maximum, which was not always present, may be related to alkanes characteristic of algal material. Poilcyclic structures are present in the C_{29} + n-alkanes range. They are related to saturated and unsaturated hydrocarbons (Figures 8a, b) for the Senonian Section 56-1 and the Aptian Sections 125-5 and 127-5. The separation between saturated and unsaturated hydrocarbons was not possible for the other samples because of the low yield of total hydrocarbons (Table 4).

CONCLUSIONS

The Cenomanian-Aptian dark shales and siltstones encountered in the DSDP cores of Leg 47B contain organic matter of predominantly continental and detrital origin. Some non-thermal alteration effects on that organic matter (possibly oxidation prior to deposition) are deduced for the intervals that are late Aptian and the early to middle Albian in age (Cores 118 to 80). Contribution of marine organic matter which is generally altered represents a marked change for the overlying sediments of late Albian to Cenomanian age (Cores 79 to 56). In the samples from Section 56-1 (earliest Senonian in age), a mixed origin is also invoked to account for combined kerogen, pyrolysis, and humic data. Typical of other Atlantic Cretaceous material, an immature stage of evolution can be assigned to all the analyzed samples.

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TABLE 3

Kerogen Data From Elementa	Analysis, With Composition and	Ash Content	, From Hole 398D
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Sample (Interval in cm)	Sub-Bottom Depth (m)	Weight Per Cent, on Ash-Free Basis						Ashes	Atomic	
		С	Н	N	0	S	Fe	(wt. %)	H/C	O/C
56-1, 120-139(A)	946.70	45.6	3.9	1.9	14.6	19.1	13.4	23.7	1.02	0.24
56-1, 120-139(B)	946.70	29.8	2.9	1.8	9.1	30.0	23.9	36.0	1.18	0.23
59-3, 135-146	978.35	34.4	2.6	1.7	10.1	27.6	24.8	33.5	0.90	0.22
60-2, 0-10	985.00	50.9	3.1	1.8	14.9	15.7	11.9	21.2	0.74	0.22
76-1, 0-41	1145.00	46.2	2.2	1.4	13.9	16.1	11.7	23.5	0.58	0.23
81-4, 20-30	1206.70	58.7	3.4	1.9	16.7	9.7	6.1	9.8	0.70	0.21
96-5, 115-123	1342.15	61.5	3.1	1.8	19.5	6.9	4.9	8.8	0.61	0.24
104-2, 106-116	1413.56	53.8	2.6	1.5	19.4	6.1	3.6	16.5	0.58	0.27
118-4, 113-115	1549.63	51.4	2.6	1.4	15.9	9.8	7.0	21.4	0.62	0.23
125-5, 122-150	1617.72	39.2	2.4	1.1	14.5	20.2	16.1	28.6	0.73	0.28
127-5, 80-110	1636.30	39.7	2.6	1.2	14.8	20.4	16.0	27.6	0.80	0.28



Figure 6. Kerogens plotted in an H/C and O/C diagram.



Figure 7. Gross composition of the chloroform extracts.



Figure 8. Gas chromatography of saturated (top chromatogram) and unsaturated (bottom chromatogram) hydrocarbons. (a) Section 398D-56-1 (post Cenomanian in age). (b) Sections 398D-125-5 and 398-127-5 (early Aptian in age).

Sample (Interval in cm)	Sub-Bottom Depth (m)	HCCl ₃ Extract (wt. mg)	Extract Related to Rock (wt. %)	Extract Related to Org. Carbon (wt.)	Thin Layer Chromatography Fractionation			
					NSO	Hydrocarbon Fractions		
					Compounds (%)	Aro (%)	Sat + Unsat (%)	
56-1, 120-139(A)	946.70	387.8	0.21	0.035	80	8	12	
59-3, 135-146	978.35	35.4	0.05	0.028	79	11	10	
60-2, 0-10	985.00	16.6	0.02	0.022	85	7	8	
76-1, 0-41	1145.00	19.9	< 0.01	0.013	85	6	9	
81-4, 20-30	1206.70	19.9	0.02	0.017	55	38a	7	
96-5, 115-123	1342.15	18.0	0.02	0.013	85	5	10	
104-2, 106-116	1413.56	11.8	0.01	0.019	83	8	9	
118-4, 113-115	1549.63	16.6	< 0.01	0.010	77	9	14	
125-5, 122-150	1617.72	34.0	0.01	0.008	73	9	18	
127-5, 80-110	1636.30	26.1	0.02	0.011	78	8	14	

TABLE 4 Composition of Chloroformic Extracts, Hole 398D

^aMostly non-aromatic waxes.