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by

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THE LIGNIN FRACTION OF MARINE SEDIMENTS

by

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The lignin fraction of marine sediments

RICHARD G. BADER

Abstract—The lignin in 31 surface samples of marine sediments from the Gulf of Maine and 26 samples from two cores collected in Puget Sound is an extremely variable fraction of their organic matter. In the two sediment cores, the loss of nonligninous carbon is about six times that of lignin carbon. Apparently lignin is extremely stable in the marine environment.

Lignin is important from a geological standpoint. Knowledge of its concentration in sediments may permit palaeoecologic interpretations which are not possible by use of total organic carbon alone. It is potentially useful as a tool in studies of rates of sedimentation; but it must be determined whether it is dependent or independent of the deposition of inorganic particles.

The possible effects of lignin on carbon-nitrogen relationships and the base-exchange capacity of the organic matter in marine sediments are also discussed.

INTRODUCTION

LITTLE is known of the distribution of lignin in the marine environment. Its presence in marine sediments was first reported by TRASK (1932) and WAKSMAN (1933). BADER (1954) studied some surface concentrations of lignin in sediments from the Gulf of Maine.

Lignin is a very resistant organic complex, formed only by living plants (BERGENSTEIN, in BRAUNS, 1952) never by enzymatic or bacterial decay of other plant products. The decomposition of lignin is extremely slow (WAKSMAN and TENNY, 1926, 1927, 1929; BORUFF and BUSWELL, 1934; LEVINE *et al.*, 1935; BRAUNS, 1952, and others) and relatively constant under both aerobic and anaerobic conditions (ACHARYA, 1935, 1935a). In aerobic soils lignin is decomposed in traces or not at all (WAKSMAN and STEVENS, 1929, 1929a). It is the most resistant of all common plant materials. It is possible that in the microbiological decomposition of plant materials, lignin may act as an inhibitory factor (REGE, 1927; WAKSMAN and IYRE, 1932; FULLER and NORMAN, 1943) by the formation of stable lignoprotein complexes.

GOTTLIEB and HENDRICKS (1945) and others have attempted to isolate lignin from soils but were unable to secure definitely characterizable products. Methyl groups are apparently split off the lignin molecule (WAKSMAN and SMITH, 1934) and its cation exchange capacity increases (MILLER *et al.*, 1936). Despite the evidence for marked alteration, SCHOFFER and WELTE (1950) observed a striking similarity between the ultra-violet spectra of soil-lignin complexes and isolated lignin.

In the present investigation three general aspects were examined: (1) its distribution and variation in some marine sediments, (2) its stability in subsurface samples, and (3) its relationship to the remaining organic material.

METHOD OF ANALYSIS

Other organic compounds interfere with lignin determinations (WAKSMAN, 1933; NORMAN, 1937; BRAUNS, 1952) and must be removed. Lignin must then be determined indirectly because of its complexity and resistance to solution.

To remove the nonligninous organic matter, an extraction and hydrolysis series was used. From 50 to 100 grams of dried sediments were crushed, powdered, and then treated with acetone in a

Soxhlet extraction apparatus for 48 hours. The sediment was then removed from the Soxhlet and refluxed in 95 per cent ethyl alcohol for 24 hours. The solvent was changed three times during this period in order to increase the efficiency of extraction. This was followed by a transfer of the sample to a fritted disk filter. The sediment was then vacuum filtered and thoroughly washed with fresh, hot, ethyl alcohol. The sample was then dried, removed from the filter and the filter was washed with 4 per cent HCl. The first hydrolysis was then accomplished by refluxing the sediment in 4 per cent HCl. After 24 hours, the sample was vacuum filtered and washed thoroughly with distilled water, transferred to 72 per cent H_2SO_4 and kept at 8°C for three hours. This solution was then diluted to 4 per cent H_2SO_4 and refluxed for four hours. Following vacuum filteration and washing with distilled water the samples were dried, powdered, and thus made ready for the determination of lignin carbon. The residual organic material after the H_2SO_4 treatment is termed lignin; this includes its transformation products as well as nitrogenous compounds such as synthesized microbial lignoprotein complexes. The carbon from this source shall thus be called lignin carbon; the remaining carbon is termed nonligninous carbon and represents that carbon removed by previous treatment.

Table 1. Surface sample data from are bounded by 44°05′ *to* 44°28′*N and* 68° *to* 68°30′*W*

Sample No.	*Depth (ft.)*	*Total carbon (%)*	*Lignin carbon (%)*	*Nonligninous carbon (%)*	$\frac{\textit{Lignin carbon}}{\textit{Total carbon}} \times 100$
1	57	0·88	0·38	0·50	43·2
2	27	1·72	0·66	1·06	38·4
3	20	0·52	0·18	0·34	34·6
4	40	1·23	0·85	0·38	69·1
5	52	0·94	0·40	0·50	42·6
6	20	0·42	0·18	0·24	42·8
7	14	0·45	0·20	0·25	44·4
8	35	0·66	0·31	0·35	47·0
9	30	1·02	0·73	0·29	71·6
10	25	0·46	0·21	0·25	45·7
11	20	0·35	0·07	0·28	20·0
12	55	1·74	0·69	1·05	39·7
13	85	1·15	0·38	0·77	33·0
14	75	0·49	0·16	0·33	32·7
15	30	4·12	2·76	1·36	67·0
16	33	4·74	2·48	2·26	52·3
17	22	4·12	2·56	1·56	62·1
24	140	0·34	0·09	0·25	26·5
25	150	0·30	0·07	0·23	23·3
26	75	1·96	1·25	0·71	63·8
27	8	2·13	1·07	1·06	50·2
28	18	1·86	1·00	0·86	53·8
29	30	2·12	1·28	0·84	60·4
30	70	2·34	1·44	0·90	61·5
31	85	1·78	0·32	1·46	18·0
32	87	0·26	0·09	0·17	34·6
33	95	1·38	0·48	0·90	34·8
34	71	0·34	0·05	0·29	14·7
35	61	0·86	0·15	0·72	17·2
36	67	3·17	2·11	1·06	66·6
37	65	0·37	0·08	0·29	21·6
Mean ..			0·73	0·69	43·0
Standard deviation ..			0·25	0·15	16·8
Coefficient of variation			34·2	22·7	39·0

The total organic carbon and the lignin carbon were determined by a semimicro dry combustion method. The samples, dried to constant weight, were combusted in a measured volume of oxygen under controlled pressure. The evolved CO_2 was absorbed on Ascarite, determined gravimetrically and the per cent carbon computed. In determining the total organic carbon the sample was first treated with warm, 4 per cent HCl to remove the carbonates. The use of such dilute HCl causes no loss of carbon from the organic matter. Carbon determinations by this method are reproducible to ± 0·03 per cent.

DISTRIBUTION AND RELATIVE STABILITY

The total organic carbon, lignin carbon, and the nonligninous carbon was determined for the 31 surface samples of marine sediments off the coast of Maine in the vicinity of Mt. Desert Island. These samples were collected by means of a "snapper" sampler and a gravity coring tube (see Table 1). The wide range and relatively large coefficient of variation of lignin carbon in total carbon indicates that lignin does not have a constant ratio to the other organic constituents.

Core No. 169 from Puget Sound (Table 2) is primarily composed of inorganic particles of a relatively uniform size in the silt range (0·062-0·004 mm). Between the depths of 15 and 43 cm in the core, the particle size grades from silt to fine sand (0·125 mm). From 43 to 65 cm the particle size grades back into the silt range. The total organic carbon correspondingly decreases in this sandy layer reaching a minimum value at 43 cm. The remaining variations in organic content are not associated with particle-size changes. The maximum concentration of total carbon and non-ligninous carbon occurs on the surface. With some minor variations, the per cent lignin carbon in the total carbon increases with depth.

Table 2. Subsurface sample data

Sample No. and location	*Depth (cm)*	*Total carbon (%)*	*Lignin carbon (%)*	*Nonligninous carbon (%)*	$\frac{\text{Lignin carbon}}{\text{Total carbon}} \times 100$
169	0	1.02	0·66	0·36	64·7
(250 ft.)	6	0·94	0·64	0·30	68·1
	15	0·92	0·63	0·29	69·2
	30	0·37	–	–	–
	43	0·09	0·06	0·03	66·6
	55	0·36	–	–	–
47°21·5′N	65	0·79	0·64	0·15	81·0
123°08·2′W	85	0·76	0·62	0·14	81·6
	110	0·74	0·47	0·27	63·4
	115	0·74	0·69	0·05	93·2
	125	0·79	–	–	–
	134	0·78	–	–	–
	140	0·77	0·60	0·17	77·9
	148	0·85	0·64	0·21	75·3
221-C	0	1·96	1·16	0·80	59·2
(200 ft.)	7	1·63	1·08	0·55	66·3
	16	1·56	1·18	0·38	75·7
	32	1·93	1·11	0·82	57·6
	43	1·71	0·98	0·73	57·3
47°43·9′N	62	2·09	0·90	1·19	43·0
122°31·7′W	74	2·38	1·50	0·88	63·0
	97	1·97	1·02	0·95	51·8
	115	2·27	1·08	1·19	47·6
	125	1·78	1·49	0·29	83·7
	150	2·00	1·37	0·63	68·5
	155	1·98	1·16	0·82	58·5

Core No. 221-C from Puget Sound (Table 2) consists entirely of relatively uniform silt-sized particles. The fluctuations in organic content are not associated with particle-size variations. The maximum concentration of total organic carbon is at 74 cm. For the immediate surface region, the per cent lignin carbon of the total carbon increases with depth, but below 16 cm this characteristic is not evident.

It appears that in the immediate surface sediments (0 to 6-7 cm) of both cores (Fig. 1) the lignin carbon decreases less rapidly with depth than the nonligninous carbon. Core No. 169 has a lignin carbon loss of 3·0 per cent and a nonligninous carbon loss of 16·6 per cent in the top 6 cm. Core 221-C has a 6·8 per cent lignin carbon loss and nonlignin carbon loss of 52·5 per cent in the top 7 cm. If it be assumed that environmental conditions have been relatively constant in both localities during this period of decomposition and that the organic decrease is due to decomposition, the percentage of nonligninous carbon loss is 5·5 to 7·7 times that of the lignin carbon. This indicates that the ligninous fraction is more resistant to decomposition than the greater part of the other organic compounds. These data (Table 2, Fig. 1) indicate that changes in the lignin carbon in excess of 1 per cent or 2 per cent per cm are due to supply changes, i.e., the lignin carbon loss due to decomposition is less than 1 per cent to 2 per cent of the total amount of lignin carbon.

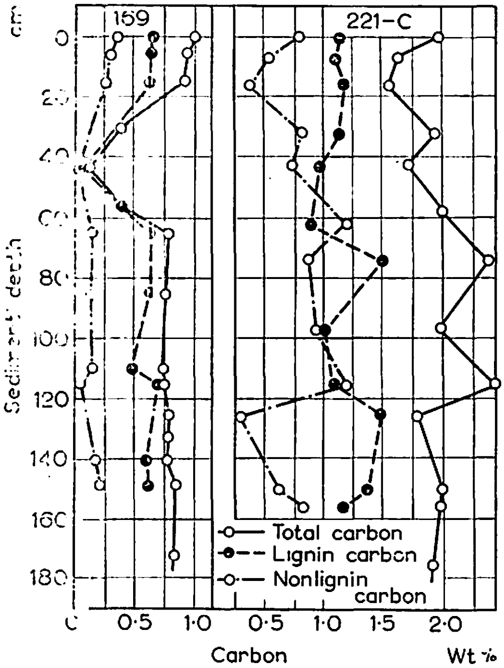

Fig. 1. Vertical distribution of total organic carbon, lignin carbon, and nonligninous carbon in two cores of Puget Sound sediments. The change in total, lignin, and nonlignin carbon in core 169 between 15 and 65 cm is a reflection of an inorganic particle size change, i.e. a sand layer. The remaining variations in core 169 and all of those in core 221-C are not related to particle size changes.

SOURCE OF ORGANIC MATTER

Although the greater part of the organic matter in marine sediments may be derived from the decomposition of planktonic organisms (SVERDRUP *et al.*, 1942) in regions of significant river drainage a considerable fraction may be derived from the land (GRIPENBERG, 1934). Since all analyses of ligninous material have been made on samples obtained nearshore (Tables 1 and 2; TRASK, 1932; WAKSMAN, 1933), much, if not all, of this fraction too may have been derived from the land. Indeed, it has not yet been demonstrated that marine algae have lignin-like complexes similar to those of terrestrial algae (WAKSMAN, 1936). Hence, no definite conclusions can yet be made as to the actual source of the ligninous matter under consideration here.

SEDIMENTATION

The amount of organic matter included in marine sediments is related to the rate of inorganic sedimentation as well as to organic supply and decomposition. Variations in concentrations of organic matter can be explained by the following means:

(1) A high rate of sedimentation of inorganic material would result in diluting the simultaneously deposited organic matter and *vice versa.*

(2) A low rate of sedimentation would increase the time that the organic fraction remains in the upper oxidizing zone of the sediments. It would thus have a longer period of active decomposition.

(3) A decrease in the supply of organic material would cause a corresponding decrease in the sediments, and *vice versa.*

(4) Environmental changes not effecting the supply or rate of sedimentation of organic material may alter the rate of microbiological decomposition.

The data from Core 221-C (Fig. 1) shows a decrease in total organic carbon from 74 to 43 cm. This could be explained on the basis of any of the four mechanisms listed above. One might conclude that this segment existed in an environmental situation that changed continually and gradually with time. However, the lignin carbon concentration decreases appreciably from 74 to 62 cm, as the nonligninous carbon increases. From 62 to 43 cm the lignin carbon increases slightly although the nonlignin carbon decreases markedly. Similar situations occur between 97 and 74 cm and 150 to 155 cm. These observations of the ligninous portion indicate that there was not a constantly and gradually changing environment over these depth ranges; other factors must be involved.

CARBON-NITROGEN RATIO

The wide variation in the lignin content of marine sediments and its resistance to decomposition may explain the wide variations in carbon-nitrogen ratios in marine sediments (GRIPENBERG, 1934; MOHAMED, 1949; ARRHENIUS, 1950; BADER, 1955). Since lignin may inhibit organic decomposition and may be associated with the microbiological formation of stable lignoprotein complexes (REGE, 1927; WAKSMAN and IYER, 1932; FULLER and NORMAN, 1943), sediments with a high lignin content have the potentiality of " trapping " more nitrogen per unit of organic material than sediments with low lignin concentrations, since nitrogen associated with lignin would be less mobile than that in the remaining organic compounds.

BASE-EXCHANGE CAPACITY

Discussions of base exchange in marine sediments have been confined to the clay-water system (KELLY, 1955) and little is known of the effect of organic material in this connection.

The organic fraction of soils has approximately seven to eight times the exchange capacity of the mineral fraction (TURNER, 1932; GEDROIZ, 1933). The base exchange capacity of soil organic matter accounts for as much as 61 per cent of the total capacity (MITCHELL, 1932; WILLIAMS, 1932). In a podsol soil 87 per cent of the exchange capacity was due to the organic constituents (BARSHAD and ROJAS-CRUZ, 1950). In a soil containing 4 per cent organic matter, well within the range found in continental-shelf sediments, 40 per cent of the total exchange capacity has been attributed to this organic fraction. Ligninous complexes are capable of irreversibly adsorbing NH_3 and other bases (WEDEKIND and GARRE, 1928).

Although free lignin has a very low exchange capacity, when it is combined with protein to form a complex, the increase in base exchange capacity far exceeds the sum of the two and closely approximates the exchange capacity of the total humus (WAKSMAN and IYER, 1932). These data indicate that the lignoprotein fraction may be responsible for much of the base-exchange capacity in the organic matter of marine sediments. The contribution to the total exchange capacity by the organic matter in the sediments should vary markedly according to the amount of lignin complexes present. Hence, organic matter in general and lignin in particular may be important factors in the early diagenetic processes in marine sediments.

SUMMARY

(1) The total lignin content and its ratio to the total organic material in marine sediments is extremely variable.

(2) Lignin is an organic complex displaying extreme resistance to microbiological decomposition. The loss of nonligninous carbon is approximately six times greater than the lignin carbon loss. Lignin carbon changes which exceed 1 per cent to 2 per cent per cm of the total may thus be ascribed to changes in the amount originally supplied to the sediments.

(3) Concentration of carbon from ligninous sources in marine sediments should give further information for palaeoecology.

(4) The concentration of lignin in marine sediments may be associated with variations in carbon-nitrogen ratios, due to its variability, stability, and the formation of nitrogen-bearing lignin complexes.

(5) When investigating the physicochemical system in marine sediments the effect of lignin and lignin complexes on this system should be considered, since studies of soils indicate that this fraction accounts for most of the base-exchange capacity attributed to organic materials.

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