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Thermal maturation of organic matter by a thick basaltic sill in Upper Cretaceous shales, Svartenhuk Halvø, central West Greenland

by

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Thermal maturation of organic matter by a thick basaltic sill in Upper Cretaceous shales, Svartenhuk Halvø, central West Greenland

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Abstract

Thermal effects of igneous intrusions on sedimentary organic matter indicate that organic compounds are more stable under geologically short but intense exposure to high temperature compared to those affected by slow burial.

In the immediate contact zone a drastic reduction occurs in kerogen content, accompanied by high reflectance values. The presence in this zone of *n*-alkanes in the C_{15} - C_{20} range suggests hydrogenation of kerogen. Heat effects are still recognisable at a distance of three times the thickness of the intrusive body.

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Fig. 1. Map of south-eastern Svartenhuk Halvø showing sampling positions at Itsako and Firefjeld.

Introduction

Thermal maturation of organic matter in sediments is now generally recognised as the essential process in the formation of the main constituents of hydrocarbons. Investigation of potential source lithologies (dark shales) of the Nûgssuaq embayment has provided regional data on the content, composition and maturation of the sedimentary organic matter (Schiener & Leythaeuser, 1978; Perregaard, 1979). Basaltic sills & dykes of varying dimensions have locally intruded the organic rich shales and have provided an excellent opportunity for studying in detail the thermal effects on the organic matter. Similar investigations have been performed earlier (Bostick, 1974; Sauvan *et al.*, 1975; Simoneit *et al.*, 1978, Peters *et al.*, 1979), but were either restricted to a microscope approach, followed by pyrolysis experiments, or when applying organic geochemical methods produced slightly different results from the ones presented here.

The present study is a continuation of earlier work on West Greenland sediments, aiming at a comparison of the effects of geologically short lived maturation by igneous intrusions with diagenetically induced changes by subsurface burial over considerable geological periods. Both organic geochemical and microscopic methods were applied in order to achieve improved control between the various analytical parameters.

Sample material

Samples were collected from outcrops in lithologically uniform carbonaceous silty shales below a 30 m thick basaltic sill on Itsako, Svartenhuk Halvø, West Greenland (fig. 1). Wherever possible samples were obtained from between 30 and 60 cm below the outcrop surface to avoid weathering effects (Clayton & Swetland, 1978). According to palynostratigraphic determinations (Croxton, 1978) the sampled sequence is of Upper Cretaceous – Lower Tertiary age. Marine macrofossils of Turonian to Campanian age are reported from the Svartenhuk Halvø – Itsako region (Rosenkrantz, 1970) suggesting a possible contribution by marine organisms to the organic matter.

Experimental procedures

Organic geochemistry

Analytical arrangements follow largely the procedures described in Perregaard (1978). Samples are dried before grinding to particle size of $<160 \mu$ for more than 90 percent of the particles. Before determination of the content of organic carbon (% C_{org}) by combustion and

measurement of the quantity of liberated CO_2 by infrared absorption in a Leco IR 12 analyser, the carbonate carbon is removed by HCl treatment.

Soxhlett extraction, with dichloromethane (CH₂Cl₂) as solvent, is used for the quantitative determination of extractable organic matter (EOM). Extracts are subsequently separated by column chromatography (Al₂O₃ + silicagel) into aliphatic hydrocarbons (eluted with *n*-hexane), aromatic hydrocarbons (eluted with dichloromethane), hetero components (eluted with methanol) and asphaltenes (retained on the column). The cut between different fractions is decided with UV and ¹H NMR analysis. All solvents are predestilled. Saturated hydrocarbons in the C₁₅₊ boiling range are analysed by GLC separation on a Hewlett-Packard 5840A gas chromatograph equipped with an OV-1 (20 m) capillary column. Quantitative measurements were performed by integration of peak areas followed by an internal standard (using 1,2,3,4-tetrahydronaphthalene) calibration programme. A computer programme was used to provide plots of relative *n*-alkane distributions, tables of *n*-alkane concentrations in the analysed sediments and calculations of component ratios by treating selected analytical data from each sample.

¹H NMR spectra were recorded at 60 MHz on a Varian T60 spectrometer using TMS as an internal reference standard.

Microscopy

Crushed material (1 mm < diam. < 2 mm) was embedded in epoxy blocks as whole rock preparations in which the morphological characteristics and relationships between organic matter and mineral matrix remain preserved and thus aid correct identification. The blocks were subsequently polished to coal petrographic standards (Stach *et al.* 1975). A Zeiss Photomicroscope 03 equipped with photomultiplier and oil immersion objectives was used for measurement of vitrinite reflectance at 546 nm and for observation of the kerogen in both reflected white light and under UV-radiation at 365 nm.

Reflectance measurements were carried out in polarised light in order to record maximum reflectance (R_{max}) of the partly anisotropic vitrinite. Where possible between 40 and 60 vitrinite particles were measured per sample; readings were recorded digitally on punch tape and treated offline in a PDB 11 mini-computer to provide histogram plots and calculations of mean and standard deviation.

Results

Organic geochemical parameters reflecting the thermally induced changes in content and composition of both extractable and non-extractable organic matter (bitumen and kerogen) of the samples are reported in Table 1. Vitrinite reflectance values are presented as reference data to indicate the extent of thermal alteration.

Organic carbon (Corg)

The high organic carbon content in the range of 3-4% C_{org} characteristic of the Upper Cretaceous and Paleocene of Svartenhuk Halvø (Perregaard, 1979), is found in the unaffected samples investigated. A marked decrease in the content of C_{org} is found close to the

GGU No.	Dist. from basaltic sill margin (m)	C _{org} w/wt%	Vitrinite reflectance R _{max} (R _m) *	Extractability mg EOM/g C _{org}	Hydrocarbons (% of EOM)	Sat. hydrocarbons (% of EOM)	CPI (24→32)	Pristane/n-C 1	7 Ratio ¹ aliph. to aromat hydrogen
177835	contact	0.11	6.00	- CAC	2	12	223	(24)	123
177831	1.5	0.29	5.80	-	÷.		1.2.1	100	(25.1
177832	2.0	0.63	4.60	-	÷.	-	1.00		141
177833	2.5	1.49	2.61	2.5	÷.	-	140	240	12
177834	3.0	2.29	3.37	1.9	86.4	65.9	-	-	1
177836	40.0	3.42	1.00	17.3	70.8	46.6	1.16	0.3	171
177838	55.0	3.27	0.72	17.7	65.6	34.7	1.34	0.6	2.50
177839	65.0	3.66	0.65*	16.7	58 8	23.0	1.46	0.9	2.81
177840	85.0	3.00	0.68 *	12.6	55.2	17.5	1.48	3.5	2.92
177868	reference sample	4.00	0.45*	8.7	37.4	6.3	1.47	4.1	

Table 1. Geochemical data and vitrinite reflectance values on organic matter in Cretaceous shales matured by thermal alteration

Average reflectance values
¹ H NMR analyses of aromatic fractions
Aliphatic hydrogen: integrated area in the range 40-200 Hz
Aromatic hydrogen: integrated area in the range 400-550 Hz

igneous body (Table 1). In the immediate contact zone only 0.11% C_{org} is preserved. This decrease is equally well observed under the microscope where high reflecting organic matter $(R_{\text{max}} > 5\%)$ is condensed to wispy stringers parallel to the bedding and to irregular microveins at roughly right angles to the bedding (pyrobitumen?) (fig. 2c).





Fig. 2. Photomicrographs of polished whole rock preparations under reflected light showing bands of vitrinite particles. Magnification \times 200.

a: Collected 85 m below igneous contact, slightly affected by igneous heat, $C_{org} = 3.00\%$, $R_m =$ 0.68% (GGU 177840). b: Collected 2.5 m below igneous contact, strongly affected by igneous heat, $C_{org} = 1.49\%, R_{max} = 2.61\%$ (GGU 177833). c: Collected 1.5 m below igneous contact, severely affected by igneous heat, contact zone, Corg = 0.29%, $R_{\text{max}} = 5.8\%$ (GGU 177831).

Extractable organic matter (EOM)

From the immature sample to the mature samples (GGU 177868 ($R_m = 0.45\%$) \rightarrow GGU 177836 and GGU 177838 ($R_{max} > 0.7\%$)) extract yields are seen to double, followed by a sharp drop in the metamorphosed zone (Table 1).

From liquid chromatography of the extractable organic matter it was found that both absolute concentration (mg HC/g C_{org}) and the relative proportion of hydrocarbons of EOM reach a maximum at a maturation level of between 0.7 and 1.0% R_{oil} .

This agrees with earlier observations that progressive thermal alteration of sedimentary organic matter during the burial process results in increased production of hydrocarbons (Albrecht *et al.*, 1976). In particular the proportion of saturated hydrocarbons in EOM increases with corresponding decrease of the thermally unstable NSO (nitrogen-sulphur-oxygen) compounds (fig. 3). Only a moderate increase in the absolute concentration (mg/g C_{org}) of aromatics has been observed.

Fig. 3. Relative distribution of saturated hydrocarbons, aromatic hydrocarbons and hetero components in extracts. Asphaltenes and evaporative losses during preparation are excluded. Increasing thermal alteration is indicated on the diagram. a: 177868, b: 177840, c: 177839, d: 177838, e: 177836, f: 177834.



Composition of hydrocarbons

The changes in distribution of *n*-alkanes (fig. 4) confirm the progressive thermal alteration of the organic matter as the sill is approached. Compared to the thermally unaffected sample GGU 177868 sample GGU 177840 shows only slight effects of thermal influence (Table 1) indicated by the dominant pristane peak and the predominance of higher boiling *n*-alkanes (heavy end) with odd numbers of carbon atoms. The carbon preference index (CPI)* is the numerical expression of this odd/even dominance (Table 1). The range of n-alkanes for the CPI calculations was chosen to include as many of the heavy end n-alkanes as possible within a reasonable performance range of the gas chromatographic system. Above $n-C_{31}$ integration of peak areas becomes uncertain and values below $n-C_{25}$ are generally insignificant.

* Carbon preference index (CPI) used in this report is expressed by

$$\frac{1}{2} \cdot \left(\frac{C_{25} + C_{27} + C_{29} + C_{31}}{C_{24} + C_{26} + C_{28} + C_{30}} \right) + \left(\frac{C_{25} + C_{27} + C_{29} + C_{31}}{C_{26} + C_{28} + C_{30} + C_{32}} \right)$$



Fig. 4. GLC chromatograms of saturated hydrocarbon fractions. Distances given are in metres from the heat source. Sample 177865 is equivalent to the reference sample 177868 on Firefjeld. Figures indicate chain lengths of *n*-alkanes. a: C_{16} -isoprenoid, b: nor-pristane, c: pristane, d: phytane.



Fig. 5. Concentrations of n-alkanes pristane and phytane as function of distance from intrusion.

Similar distributions are found in recent immature sediments and biological systems where humic kerogen contributes to the organic matter (Tissot *et al.*, 1977).

The unaffected reference sample (GGU 177868) contains saturated hydrocarbons with an analogous distribution. Nearer the igneous intrusive the predominant contribution of heavy n-alkanes is replaced by a light end predominance and the CPI values approach unity. Crude oils are usually characterised by CPI values close to 1.

By quantitative GLC analysis it is found that *n*-alkanes increase in yield while the concentration of the isoprenoids pristane and phytane remain almost constant throughout the oil generating zone $(0.5 < R_m < 1.35, Dow, 1977)$ (fig. 5). These features appear in Table 1 as declining pristane/*n*-C₁₇ ratios concurrent with increasing maturation. Only lower boiling alkanes below C₂₀ are found in the severely heated sample GGU 177834 (figs 4 & 5).

Compositional changes among the aromatic hydrocarbons were also observed by GLC analyses. With increasing maturation lower boiling aromatic compounds are generated. Heating results in dealkylation of aliphatic side chains and aromatisation of partly saturated rings.

These changes are reflected in ¹H NMR analyses. Hydrogen atoms bound to aromatic rings resonate in the range 400–550 Hz, while hydrogen bound to aliphatic parts of the hydrocarbon molecule resonates in the range 40–200 Hz (fig. 6). With increasing thermal influence the ratio of aliphatic hydrogens to aromatic hydrogens decreases (Table 1).



Fig. 6. ¹H NMR spectrum of total aromatic hydrocarbon fraction from sample GGU 177839.

Microscope investigations

In the samples where vitrinite reflectance ranges from 0.45% to 1.00% the organic matter is characterised by a predominance of humic constituents (vitrinite + fusinitic inertinite 95–98%). Two types of vitrinites may be distinguished by their morphology. The equigranular, particulate vitrinites show signs of strong oxidation (higher reflecting rims of fissures and grain margins). In vitrinite seamlets and wisps parallel to the bedding the degree of oxidation is less well developed.

The sample suite from Itsako (fig. 1) is characterised by a predominance of particulate organic matter of uniformly small grain size $(10-20 \ \mu m)$.

Low reflecting material occurring as fillings of irregular microfissures, frequently orientated oblique to the bedding, is interpreted as non-fluorescing asphaltic bitumen (? pyrobitumen) (fig. 2a).

The reference sample from Firefjeld (GGU 177868) differs in the humic components only in considerably larger grain size $(50-100 \ \mu m)$ of the particulate vitrinites and more continuous and thicker vitrinite bands.

Only a few microspores are found in the figured exinitic constituents, but in sample GGU 177868 some yellow fluorescing amorphous material occurs which might represent microbial degradation products of exinitic components. In the same sample faintly yellow fluorescing interstices between calcite crystals are indicative of generated bitumen (bituminite: Teichmüller, 1974).

Discussion

Thermal maturation of sedimentary organic matter is now generally recognised as the essential process in the formation of hydrocarbons found in petroleum. Depending upon geothermal conditions and on the effective time of exposure to elevated temperatures of the sediments, burial to adequate depths results in the formation of hydrocarbons from kerogen (Philippi, 1965; Tissot *et al.*, 1971). The main generation phase is taken to lie in the temperature range of 60–150°C. Laboratory pyrolysis of sediments and kerogens has shown that hydrocarbons are released in the temperature range 300–500°C, depending on experimental conditions and on the maturity and type of the organic matter used in the experiments (Douglas *et al.*, 1975; Espitalié *et al.*, 1977).

The drastic drop in organic carbon content close to igneous intrusions where the sedimentary organic matter suffered exposure to the most intense heating is now well established from detailed studies in the Nûgssuaq Embayment, West Greenland (Perregaard, 1979) and from the study of a series of Jurassic outcrops in Milne Land, East Greenland (Perregaard & Schiener, 1979). Similar findings are reported by other authors (Degens, 1968; Sauvan *et al.*, 1975; Akiyama *et al.*, 1979; Peters *et al.*, 1979). Oxidation and gasification of the organic carbon to CO₂ or CO through reduction with pore or absorbed water of the mineral matrix is thought to be responsible for the reduction in kerogen content (Sauvan *et al.*, 1975; Perregaard & Schiener, 1979). Laboratory pyrolysis of sediments containing immature kerogen at 500–700°C in an inert atmosphere in glass ampoules corroborate these findings (Perregaard, unpublished results). According to Bostick (1974) vitrinite reflectance values of $R_{max} > 4.0\%$ correspond to pyrolysis temperatures in excess of 600°C for shales containing humic organic matter.

The quantity of extractable hydrocarbons (EHC) (3.3 mg EHC/g C_{org}) found in the immature reference sample (GGU 177868) is in accordance with concentrations of C_{15+} hydrocarbons reported by Philippi (1968) for recent shallow marine sediments (4.3 mg EHC/g C_{org}) and recent sediments from deep marine basins (3.7 mg EHC/g C_{org}). Also in the matured Itsako samples the hydrocarbon concentrations are comparatively low, evidently owing to the unfavourable humic origin of the organic matter. Weathering effects may be responsible for some additional reduction in hydrocarbon content (Clayton & Swetland, 1978). Good petroleum source rocks are reported to contain 30–150 mg EHC/g C_{org} (Philippi, 1968). In the Itsako samples the highest concentration of hydrocarbons was found in sample GGU 177836 (12.2 mg EHC/g C_{org}) which, according to the reflectance value of $R_{max} = 1.0\%$, falls well within the zone of peak hydrocarbon generation for the humic type kerogen. The abundant contribution of *n*-alkanes to the saturated hydrocarbon fractions similarly is an expression of the higher land plant origin of the organic matter (Brooks, 1973; Lijmbach, 1975).

Generation of hydrocarbons by exposure to increasing subsurface temperatures creates a relative increase in *n*-alkanes compared to cyclic and branched alkanes with increasing maturation (Tissot, *et al.*, 1971; Milner *et al.*, 1977). A similar temperature related evolution emerges for the Itsako samples. From the slightly matured sample GGU 177840 where *n*-alkanes constitute approximately 40%, the *n*-alkane contribution in the well matured sample GGU 177836 increases to approximately 70% of the total saturated hydrocarbon fraction.

The relatively high proportion of isoprenoid alkanes (figs 4 & 5) is thought to indicate

high photosynthetic activity prior to deposition. The chlorophyll molecule includes a phytyl side chain which may be hydrolysed during early diagenesis to the acyclic diterpenoid phytol. In an oxygen deficient environment phytol may then be directly reduced to phytane whereas under oxidising conditions it may become oxidised and subsequently decarboxylated to pristane. The ratios of pristane/phytane may thus be used as indicators for the depositional environment (Powell & McKirdy, 1973). In the analysed samples concentrations and ratios of pristane to phytane remain constant throughout the temperature regime of the oil generating zone (samples GGU 177836 to 177840, fig. 5) and would thus seem to indicate a low temperature, diagenetic origin of these isoprenoids. Laboratory pyrolysis af mature sediments and kerogens has shown that only insignificant amounts of these compounds are formed by thermal cracking of mature kerogens (Perregaard, 1979, and unpublished results). Only low energy oxidation and reduction processes seem to control the formation of these isoprenoids.

Deeply buried sediments (well into the metamorphic dry gas zone) generally do not contain liquid hydrocarbons in the C_{15+} range (Hunt, 1977). Nevertheless, even in the severely metamorphosed samples GGU 177834 and 177833 appreciable quantities (1.5-2.0 mg EHC/g C_{org}) of low molecular weight hydrocarbons, particularly saturated hydrocarbons in the C_{15+} range, were present. Migration cannot be ruled out to explain the presence of these hydrocarbons. It should be remembered that the chemical conditions for the cracking process that generates hydrocarbons under relatively high temperature conditions differ from those that exist during the geological burial situation. In the case of geologically short but intense heating by igneous intrusions, the extractable hydrocarbons of low molecular weight in the range C15 to C20 can also be considered as having formed in situ by hydrogenation of kerogen (Akiyama et al., 1979). Their preservation in the high temperature environment is tentatively related to particular kinetic conditions where the carbon-carbon bonds of free hydrocarbons show greater stability than the bonds of aliphatic structures containing heteroatoms. These considerations are just an exemplification of the behaviour of chemical structures in relation to the different activation energies established for the thermal cracking processes of the three different types of kerogens as outlined in Tissot & Welte (1978).

In the literature it is claimed that heat effects of igneous intrusions on sedimentary organic matter are limited to a distance of approximately 100% of the thickness of the intrusion (Degens, 1968; Bostick, 1974; Sauvan *et al.*, 1975; Perregaard & Schiener, 1979). In the references cited, the thickness of the intrusive bodies did not exceed about five metres. In the present study maturation parameters like vitrinite reflectance, extractability and hydrocarbon distribution indicate that the thermal effects may reach up to 300% of the thickness. This is attributed to the considerably greater thickness of the intrusive body, with accordingly greater capacity for heat flow (Jaeger, 1959). During burial to comparable maturation levels the organic matter is exposed to considerably lower temperatures, but over much longer periods of time.

The microscopically observable organic matter is predominantly of humic origin, which commonly is not rated as a source for significant amounts of saturated hydrocarbons in the C_{15+} range. Thermal maturation of humic substances yields proportionally greater quantities of aromatic and asphaltic compounds. The asphalt observed in fissures in the matured samples is thus probably derived from the vitrinitic components. But the relatively high proportion of paraffinic hydrocarbons in the extracts (Table 1) can partly be attributed to thermally effected reduction of polar lipoid components and partly to a derivation from a

minor part of the kerogen (derived from exinitic organic matter – leaf cuticles, spores and pollen) by thermal cracking (Brooks, 1973). Figured exinites constitute less than 5% of the organic matter (microspores and cuticular fragments). The weak fluorescence of the matrix, as well as of intergranular spaces, suggests the presence of additional microscopically irresolvable exinitic material which may have originated from microbial degradation of figured organic matter (Lijmbach, 1975).

Conclusions

The following conclusions can be drawn from the study of the Upper Cretaceous–Tertiary samples from Svartenhuk Halvø.

1. The content of organic matter of samples with no or only moderate exposure to heat from the igneous intrusive is above average compared to oil source rocks.

2. A drastic reduction in organic carbon content is observed in the zone of extreme heating and is tentatively related to oxidation processes leading to the formation of CO or CO_2 .

3. The extracts and hydrocarbon fractions undergo progressive changes dependent on the distance from the intrusion, which are correlatable to temperature zones observed in deep bore holes.

4. The concentration per unit of C_{org} of the isoprenoid alkanes pristane and phytane are practically unaffected by increasing temperatures within the oil generating zone suggesting thermal stability.

5. Results of similar investigations of organic rich sediments intruded by igneous rocks can be used in the evaluation of their hydrocarbon source potential.

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