Clay mineral investigation of the Rhaetic – Jurassic – Lower Cretaceous sediments of the Børglum 1 and Uglev 1 wells, Denmark

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The clay mineral assemblages of the Rhaetic – Jurassic – Lower Cretaceous sequences from the Børglum 1 and Uglev 1 wells have been investigated to evaluate the diagenesis of the sediments. Besides grain size analyses, the methods include X-ray diffraction analyses and scanning electron microscopy in combination with energy dispersive analyses.

The distribution of clay minerals essentially shows a rapid change from smectite to kaolinite dominance with increasing depth. Kaolinite dominates the older sediments below the lower part of the Haldager Formation, including the Fjerritslev and Gassum Formations, while smectite dominates the younger sediments above the upper part of the Haldager Formation, which includes Bream and Vedsted Formations. This change is primarily attributed to differences in the composition of the mineralogy and geochemical environment rather than to burial diagenesis. While some early diagenesis has taken place to form authigenic kaolinite, thorough depth-dependent diagenesis can hardly be detected.

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Introduction

The clay minerals in recent sediments from marine, fluvial and lacustrine environments broadly reflect the input of detrital clays from the source area, and only to a lesser extent consist of neoformed clay phases (Shaw 1978).

The detrital clay assemblages are formed in soils and weathering profiles via the breakdown of pre-existing silicate minerals generated at high pressures and temperatures and therefore mostly unstable at the Earth's surface. The composition of the clays formed in this way is dependent on parent rock type and climate.

Interpretation of clay mineral assemblages in ancient sediments is, however, complicated by burial diagenesis, which due to changing physical and chemical conditions with increasing depth causes neoformation and transformation of the clay minerals (Weaver 1967, de Segonzag 1970, Perry & Hower 1970, 1972, Hower et al. 1976, Curtis 1977).

The diagenesis of clay minerals has been intensively investigated since Weaver (1967) showed a statistical distribution of the relative proportions of clay minerals in argillaceous sediments over geological time and related to depth of burial. The principal changes with depth are increasing proportions of illite and chlorite with corresponding decreases in smectite and kaolinite. This tendency is attributed to the effect of burial diagenesis on the clay minerals which is a function of the chemistry of the fluids in the sediments, geothermal temperature, pressure, initial mineralogy and geological reaction time. The clay minerals are most sensitive to changes in the physical and chemical parameters and different stability fields exist for the individual clay minerals in the subsurface (de Segonzag 1970).

There are only few publications concerning the clay mineral distribution in the Mesozoic sequence from the Danish Subbasin (e.g. Nielsen & Friis 1985).

The purpose of this investigation is to describe

the clay mineral assemblages in the sediments from the two wells Børglum 1 and Uglev 1 and to determine the extent to which the clay composition is controlled by subsurface diagenesis or provenance.

Geology

Concerning the regional geological and stratigraphical aspects and environments of deposition of the Mesozoic sequence in the Danish area, reference is made to papers by Larsen (1966), Christensen (1974), Michelsen (1975, 1978), Bertelsen (1978, 1980) and Koch (1983).

The locations of the Børglum 1 and Uglev 1 wells in the Danish Subbasin are shown in figure 1.

Well data are published by Sorgenfrei & Buch (1964) and in the Well Data Summary Sheets, vol. 2 (1981).

A brief characterization of the sedimentary sequences in the wells is given in Schmidt (1985).

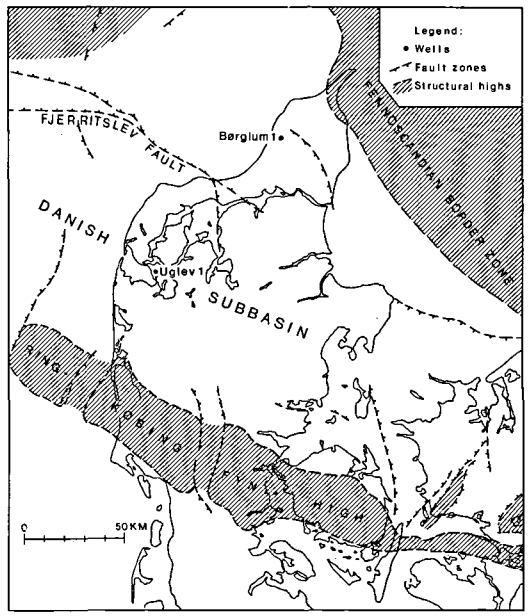


Fig. 1. Location map (modified from Michelsen (1978).

Materials

The investigation is based on core material available from the Rhaetic-Jurassic-Early Cretaceous sequence in the wells Børglum 1 and Uglev 1.

Analytical procedures

X-ray diffraction analyses

For bulk mineral analysis pulverised and randomly orientated specimens were used.

For clay mineral analysis the samples were pretreated with sodium acetate (NaAc) to remove any carbonates present while organic matter was removed by oxidation with sodiumhypochlorite (NaOCl) solution. Iron and aluminium oxide coatings were removed by sodium dithionite ($Na_2S_2O_4$) according to the method of Mehra & Jackson (1960). Small portions of the clay fraction (separated by sedimentation in water) were saturated with magnesium (Mg^{2+}) ions and oriented specimens were prepared for X-ray examination. Four different X-ray diffractograms have been produced from each sample: Mg^{2+} saturated (1), Mg^{2+} saturated and further glycerolated (2), K^+ saturated (3), K^+ saturated and heated to 300°C for 1 hour (4). All specimens were run on a Philips diffractometer, bulk minerals from 5° to 85° 2 θ and clay fractions from 4° to 16° 2 θ using Co K-α-radiation. Mineral identification was carried out by comparing the peak positions with the reflection values reported in the Powder Diffraction File and ASTM charts. Identification of the clay minerals was performed by comparing the (001) reflections in the X-ray diffractograms obtained from the differently treated suspensions.

The 7 Å peak which is not affected by heating to 300°C or glycerolation is ascribed to kaolinite. Illite is identified by 10 Å reflections which are not affected by other treatments. Smectite is characterized by reflections at 12–15 Å, depending on the interlayer cations. It is identified by 14 Å reflections in the Mg^{2+}-saturated samples which on glycerolation move to about 18 Å and on heating collapse to 10 Å. The 14 Å peak which is unaffected by heating is ascribed to chlorite, while that part of the 14 Å reflection which is unaffected by glycerolation but collapses to 10 Å on heating is ascribed to vermiculite. Reflections be-

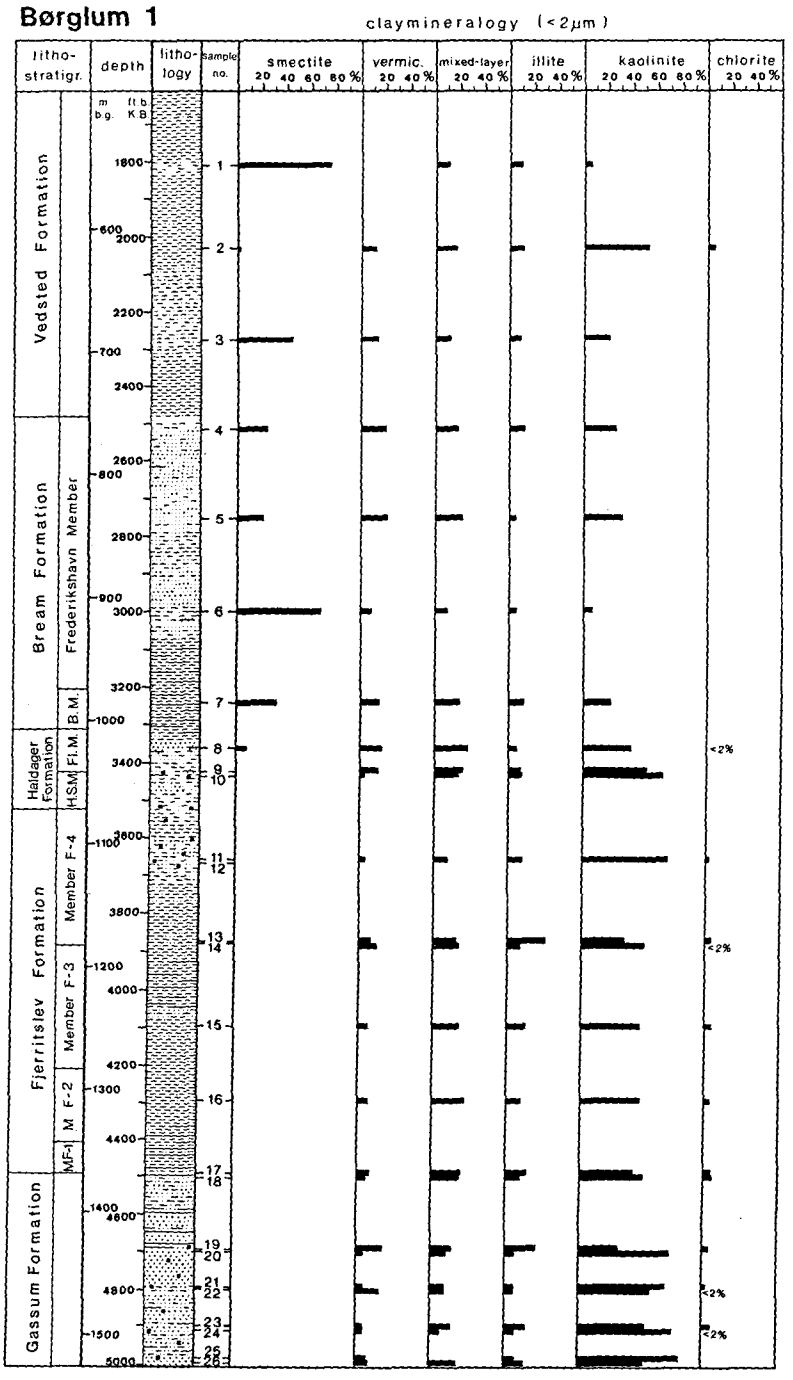
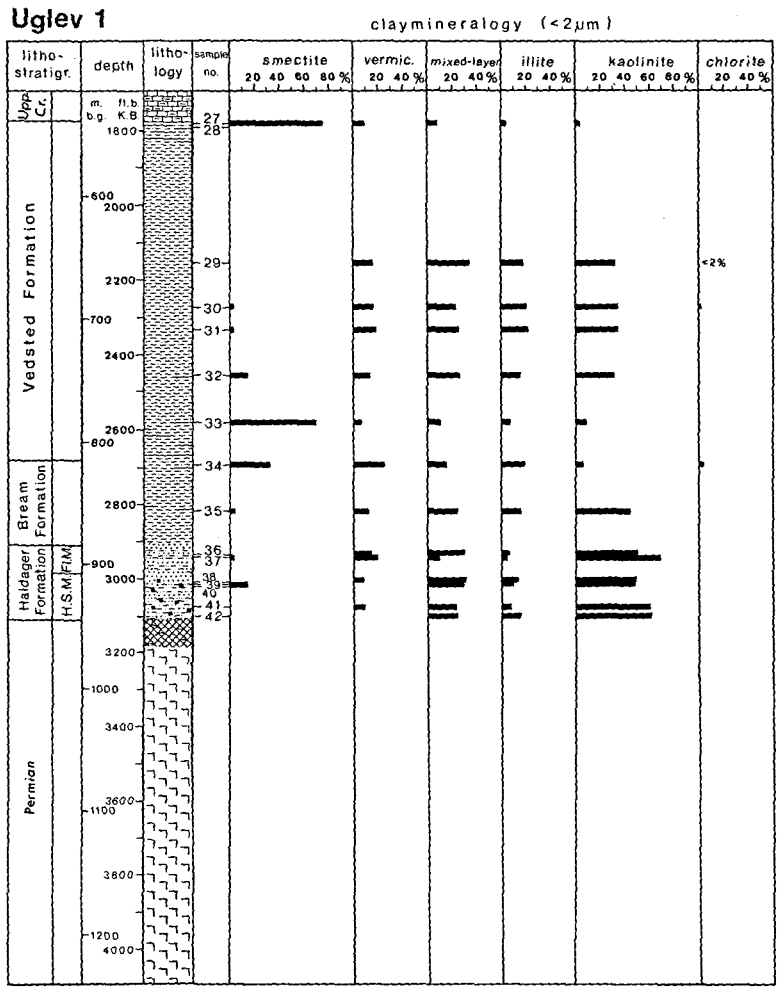
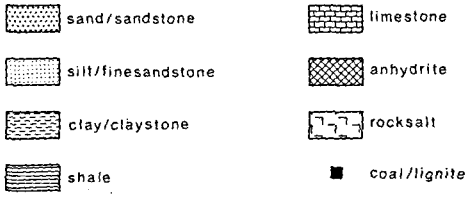


Fig. 2 & 3. Clay mineral distribution.
H. S. M. = Haldager Sand Member,
Fl. M. = Flyvbjerg Member
B. M. = Børglum Member.
(The lithology is modified from Larsen (1966) and Michelsen (1978).





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tween 10 and 14 Å are interpreted as mixed-layer illite-smectite (H. Lindgreen, pers. comm.)

Semiquantitative estimations of the bulk mineral composition were performed using peak intensities according to Schultz (1964).

The clay mineral compositions were calculated as relative integrated reflection intensities, whole peak areas have been used for quantification, with the total clay reflections area being equalled 100% in the Mg^{2+}-diagram (H. Lindgreen, pers. comm.).

SEM & EDX

Small rock fragments of untreated samples were examined using a Cambridge Stereoscan S 180 scanning electron microscope with an attached energy-dispersive X-ray analyser.

Only the more coarse-grained material was suitable for investigation as in claystone and shales single crystals cannot usually be recognized.

Mineralogical identification of the clays was based on comparing the morphology with the micrographs and data from, among others, Bohor & Hughes (1971), Almon & Davies (1979), Wilson & Pittman (1977), Sarkisyan (1972), Tompkins (1981) and Güven et al. (1980). The results were supported by determination of the chemical composition of the characteristic minerals as follows: kaolinite (Si-Al-rich), chlorite (Si-Al-Mg/Fe), smectite (Si-Al-Mg/Fe-Ca/Na), illite (Si-Al-K). Since the scanning was performed on samples with fractured, rough surfaces, only qualitative chemical analyses were possible. The low atomic number elements Na, and partly Mg, can only be detected if they are present in very large amounts (EDAX 1976).

Results

X-ray diffraction analyses

Clay mineral distribution is shown in figures 2 and 3 while bulk mineral distribution is shown in figures 4 and 5. Some characteristic minerals are shown in SEM micrographs (plate 1–2).

The smectite and kaolinite show the greatest variations. Smectite only occurs in the upper part of the investigated sequence. In the Vedsted and Bream Formations it is locally the dominant clay mineral in both wells, showing a minimum in the middle part of the Vedsted Formation. In the Haldager Formation the smectite content decreases rapidly to zero and it has not been identified in the older deposits of Børglum 1. Kaolinite, on the other hand, occurs in all samples and is clearly dominant in the Fjerritslev and Gassum Formations. The kaolinite content does not increase gradually with depth, the highest proportions is found in the coarse grained sediments, especially from the Gassum Formation. No marked, systematic changes with depth are seen in the distribution of the other clay minerals. Only very small amounts of chlorite have been tentatively identified in some samples in the two wells, but from different depths. The illite content seems to be highest in the fine grained sediments, but the differences are not pronounced.

SEM & EDX

Kaolinite has been identified in most investigated samples in varying amounts, often occuring as authigenic crystals in coarse-grained sediments. Authigenic kaolinite is easily recognized as platy crystals with pseudohexagonal morphology, most frequently occurring as long vermicular aggregates which line or fill pores in some sand/siltstones (Plate 1. 1–4). The delicacy of the euhedral clay flakes, and the morphology of the aggregates, are unlikely to be preserved if subjected to extended transport and reworking, thus providing good evidence for their authigenic nature. Crystal size often exceeds "normal" clay size (> 2 μm) and crystals up to 12–15 μm have been observed. Detrital clays are generally smaller than 5 μm (Wilson & Pittman 1977). Authigenic kaolinite is common in samples from the Gassum Formation and some of the Fjerritslev Formation (Børglum 1) and from the Haldager Formation (Uglev 1).

Individual crystals of smectite cannot be seen, as they are very small (normally < 1 μm) and dispersed (Bohor & Hughes 1971). Smectite has only been identified in samples from the Vedsted and Bream Formation (Børglum 1) (Plate 1.5–6) and shows a crincly ridged texture, characteristic for this mineral.

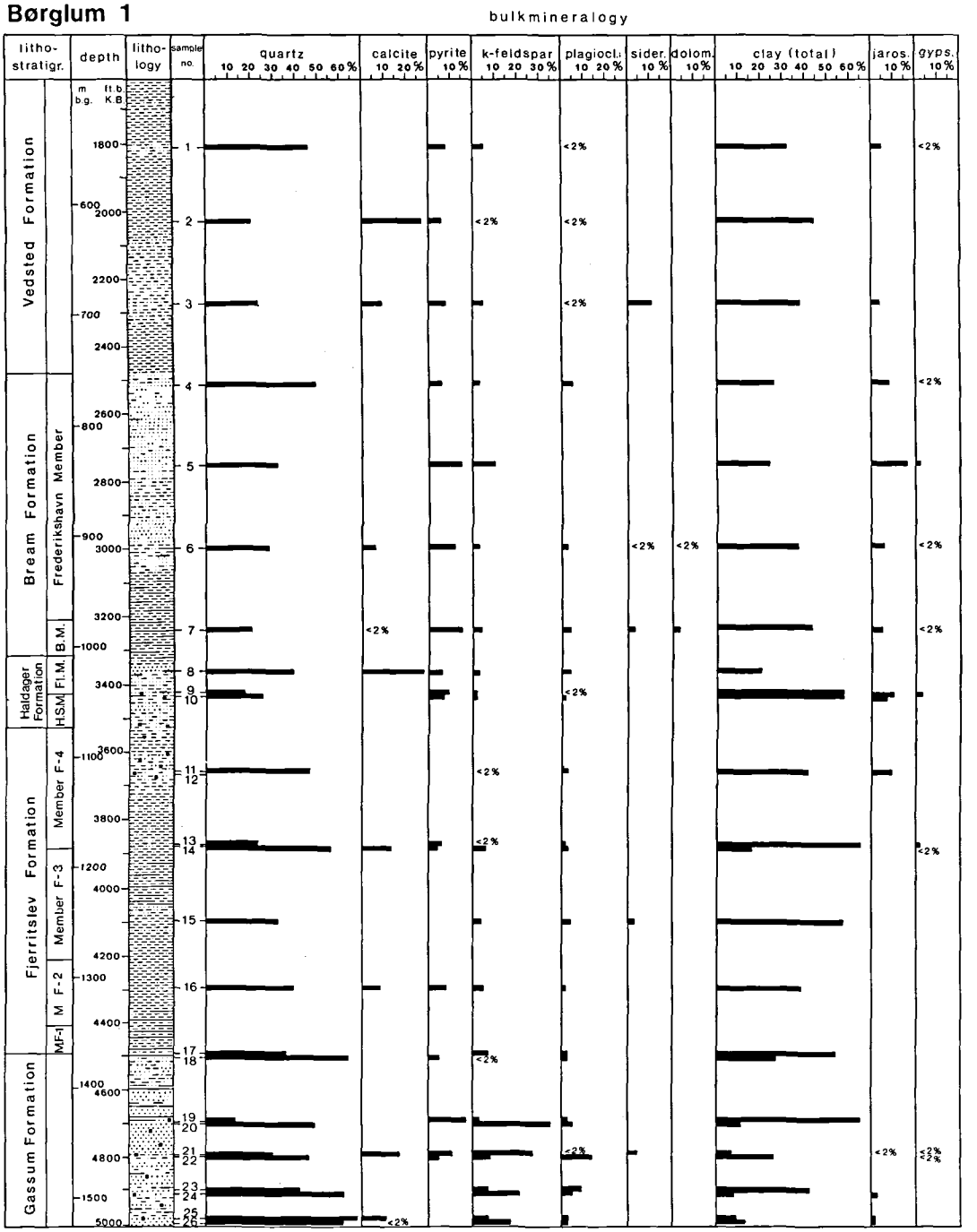
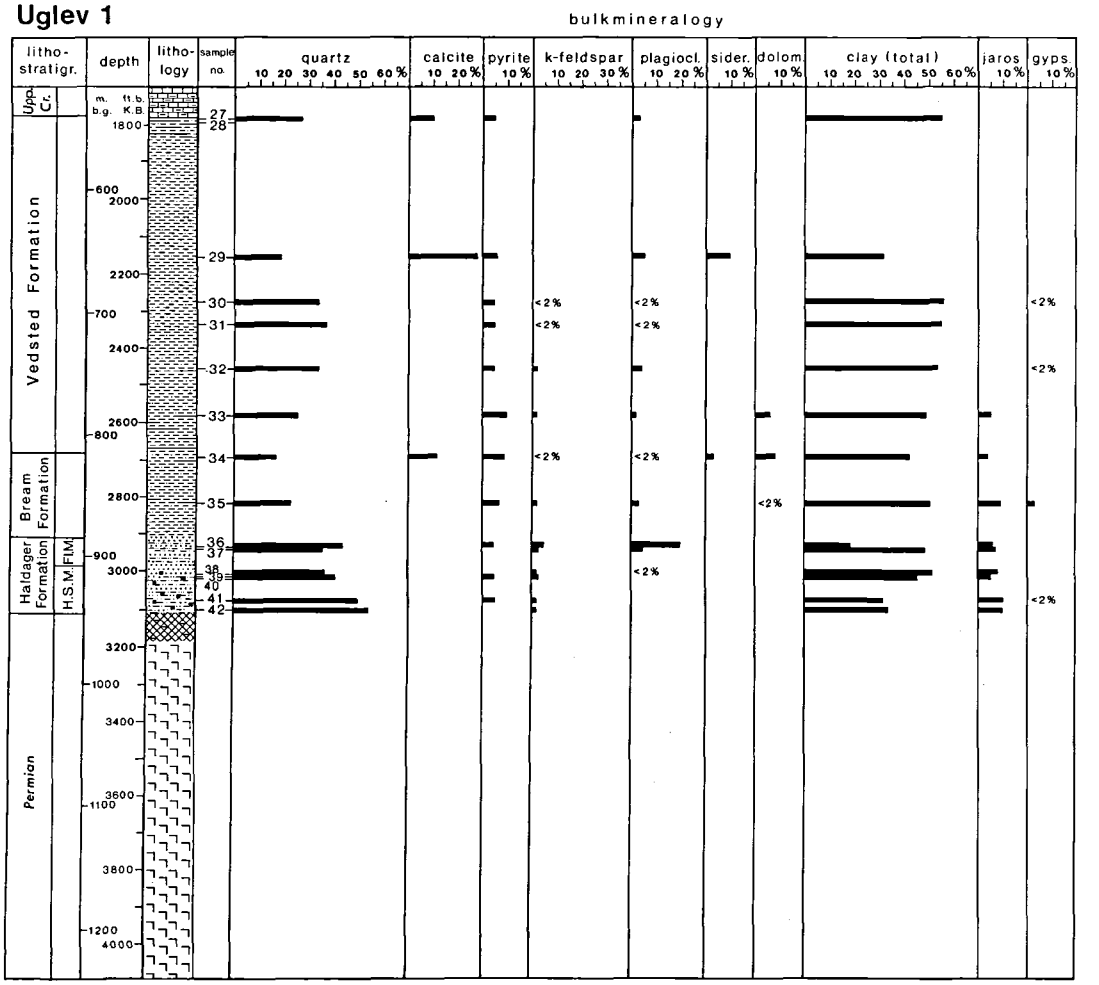


Fig. 4 & 5. Bulk mineral distribution.
H. S. M. = Haldager Sand Member,
Fl. M. = Flyvbjerg Member,
B. M. = Børglum Member
(The lithology is modified from Larsen (1966) and Michelsen (1978).



Authigenic illite with the hairy, fibrous crystal growth form described by Güven et al. (1980) and Wilson & Pittman (1977) have not been found. Because of their inconspicuous morphology, with anhedral plates and aggregates, detrital illites are very difficult to identify. According to EDX-analysis some of the detrital clay matrix present in most samples must contain illite, either as discrete particles or as a component in a mixed-layer structure.

Mixed-layer clays have characteristics similar to the participant clay minerals and their distinction from the discrete clays is unreliable. In some of the investigated samples (Bø 4–6, Bream Formation, Fredrikshavn Member) clay particles can be seen with short lath-like projections (Plate 2.7) very similar to clay minerals interpreted as mixed-layer illite-smectite by Wilson & Pittman (1977). This type is only seen in samples which also contain smectite.

Chlorite has not been identified with certainty. According to Wilson & Pittman (1977) authigenic chlorite occurs with very variable morphologies. Chlorite-like particles have been observed in two samples (Bø 24, 26) from the Gassum Formation (Børglum1) (Plate 2.8), but their identification is doubtful as EDX-analysis only showed Fe-Si-Al and no Mg content.

Quantification of the clays by SEM is unreliable, and the clay mineral distribution obtained

by X-ray diffraction can not be proved directly. In SEM authigenic kaolinites (and other authigenic minerals) were easier to identify than e.g. detrital illites because of their distinctive morphology, better crystallinity and greater size. Their abundance can therefore easily be overestimated. It is not possible to directly compare the SEM and X-ray results as optically recognizable clay minerals, at least for kaolinite, exceed 2 μm while only clay fractions (< 2 μm) were analysed by X-rays, but there seems to be a good agreement. In samples where the X-ray analyses show that smectite dominates the clay fraction (Vedsted and Bream Formations), it is also seen in large amounts by SEM, while other clay minerals can hardly be detected. SEM also confirms that kaolinite is the dominant clay mineral in the coarse grained sediments from Gassum, Fjerritslev (Børglum 1) and Haldager Formations (Uglev 1). Smectite has not been found in these kaolinite-dominated samples and other clay minerals have only rarely been seen. Some chloritelike particles may be present in the Gassum Formation but have not been observed elsewhere.

It is a general feature in all samples that much of the clay content does not posess distinct morphological characteristics, probably due to a detrital origin or mixed layering of the minerals. Detrital clay is often parallel oriented because of compaction, and clay lamina are often smoothed around sand grains. Single crystals cannot normally be distinguished and SEM identification is impossible.

Other mineralogical observations under SEM provide useful supplementary information to the X-ray results. Authigenic kaolinite is seen in association with authigenic quartz overgrowths on detrital silica grains, and sometimes also in paragenesis with feldspar in samples from the Fjerritslev and Gassum Formations (Bø 15, 22, 24 and 26) (Plate 2.12). X-ray analyses show a relatively large content of feldspars in the sediments from the Gassum Formation, especially in the coarse grained samples where they can make up to 40%. Under SEM, large grains of feldspar very similar to those illustrated by Dypvik & Vollset (1980), Bjørlykke et al. (1979), Hancock & Taylor (1978) and Almon & Davies (1979) are seen with corroded appearance indicating decomposition. Extensively leached feldspar grains are seen in the Gassum Formation sediments (Plate 2.11).

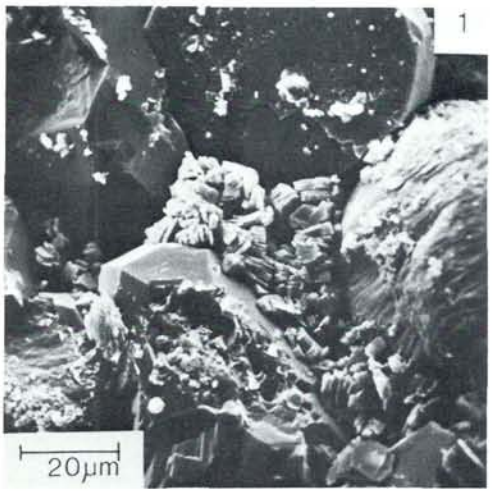
Discussion

In burial diagenesis, gradual changes are usually seen in the clay mineral composition with depth. Smectite is generally unstable with increasing depth, the end product being dependent on pore water chemistry. In alkaline pore water, as is normally the case with increasing depth, smectite is progressively transformed to illite in a K-rich environment, or to chlorite in a Mg-rich environment, via intermediate mixed-layer phases. Illites and chlorites will be preserved, and burial diagenesis will enhance their degree of crystallinity. Kaolinite, on the other hand, is unstable under alkaline conditions and is normally destroyed or transformed during burial (de Segonzag 1970).

In this study, clay mineral compositions obviously do not show this diagenesis with increasing depth. The most remarkable change in the clay mineral distribution of the investigated sediments is the abrupt shift in the Haldager Formation from smectite to kaolinite dominance downwards. Apart from changes in the smectite and kaolinite content no other minerals show significant variations with depth. It seems clearly unreasonable to assume that depth dependant diagenesis has caused this abrupt shift without noteworthy changes in other clay minerals. The initial composition and grain size of the sediments largely effect the course of diagenesis and make it difficult to detect a depth dependant diagenesis. As many different lithologies and facies are concerned in this study only tentative conclusions are possible.

Provenance, as well as diagenesis, can cause

Plate 1: 1–2; Booklets and vermicular aggregates of authigenic pseudohexagonal kaolinite and authigenic quartz filling a pore (Børglum 1, 15, 4096'). 3; Vermicular aggregates of authigenic pore-lining aggregates of kaolinite (Uglev 1, 41, 3073'). 4; Authigenic kaolinite forming vermicular aggregates (Børglum 1, 24, 4909'). 5; Smectite with honeycomb-like crenulated surface, kaolinite near the upper left margin of the micrografh (Børglum 1, 1, 1806'). 6; Smectite (Børglum 1, 6, 3001').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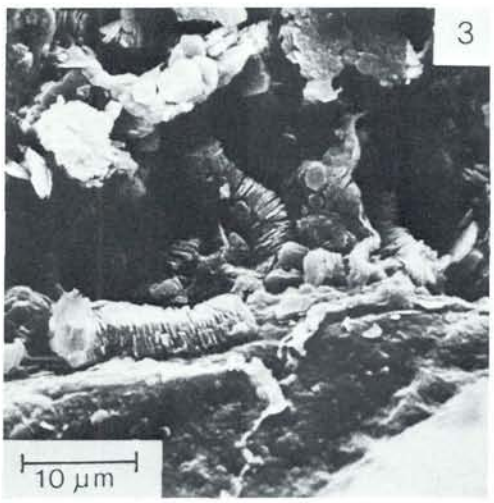
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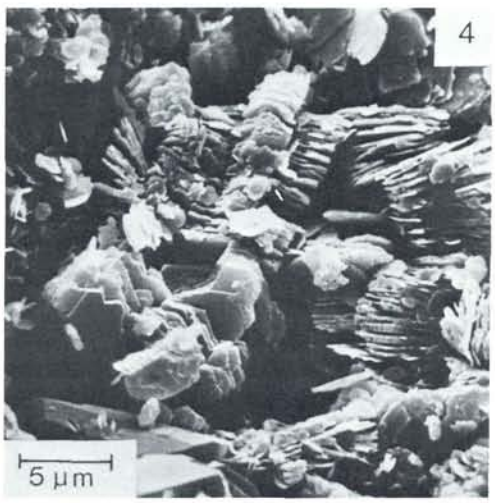
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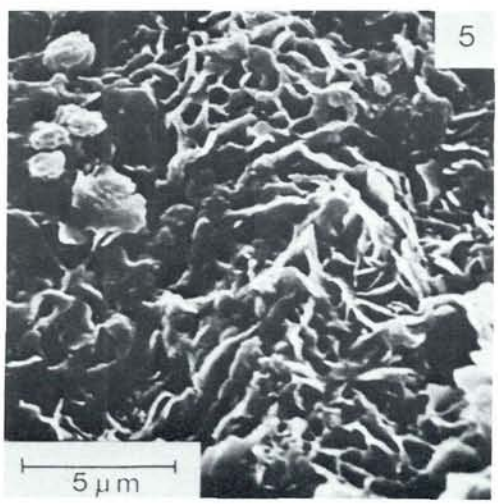
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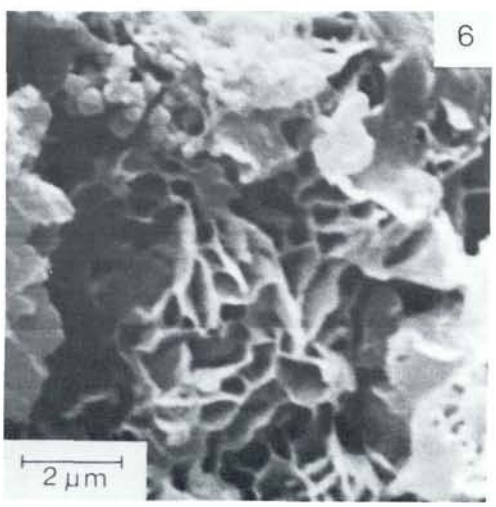
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2 µm

high kaolinite contents in sediments. One aspect of provenance is the climate and weathering conditions in the source area. As the climate in western Europe changed from arid to humid in the Rhaetic, and warm humid conditions continued into the Jurassic (Hallam 1982), chemically stable conditions for kaolinite were created. Thus kaolinite might have entered the sedimentary deposits in considerable amounts.

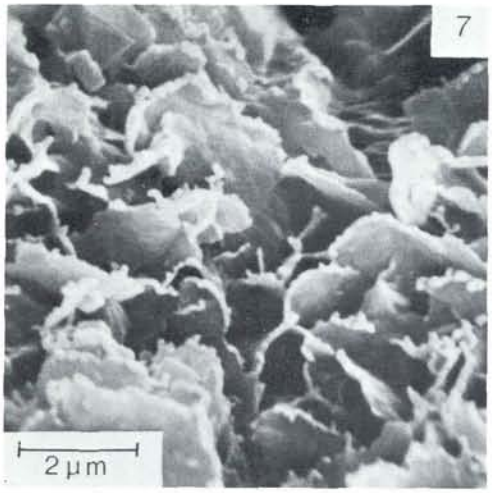
This may partly explain the high kaolinite content in the sediments, but it obviously cannot account for the higher content in the coarse grained Gassum, Fjerritslev and Haldager Formation sediments. A grain size dependance at least in the Gassum Formation sediments can be illustrated by comparing the clay mineral composition in some cores with large lithological variations where samples from fine and coarse grained lithologies were analysed (Fig. 6). The lower the clay content (< 2 μm fraction), the higher the fraction of kaolinite. According to Weaver (1961) kaolinite does not appear to be strongly concentrated in sands rather than muds in recent sediments. So the higher kaolinite content in the sandstone samples compared with the associated finer grained sediments must be attributed to the generally higher sand permeability allowing post-depositional kaolinite formation directly from solution in the porespace.

Authigenic kaolinite was observed by SEM in the coarse grained Gassum, Fjerritslev and Haldager Formation sediments while no other authigenic clay minerals have been identified with certainty. The amount of authigenic kaolinite is difficult to estimate but simple visual inspection suggests that the proportion of authigenic kaolinite is high since much of the kaolinite is often present as large aggregates. Even small quantities of precipitated kaolinite may readily effect the clay mineral composition in a coarse grained sandstone, while in shaly samples the clay content is mainly of detrital origin.

In SEM authigenic kaolinite is sometimes seen in association with authigenic quartz overgrowths and corroded detrital feldspars (Plate 2.12). Especially in Gassum Formation sediments extensively leached feldspars are seen (Plate 2.11). Kaolinite and guartz overgrowths might be formed from the breakdown of the K-feldspar grains. Abundant terrestrial plant debris, especially noteworthy in the deltaic/fluvial sandstone of the Gassum Formation and Haldager Sand sediments, may have caused an acid reaction with the pore fluid with consequent lowering of the pH-value. The exclusive breakdown of feldspar to form kaolinite only occurs at low K^+/H^+-ratios and under slightly acidic conditions (Almon & Davies 1979). This requires that large quantities of low salinity or fresh water pass through the sediment. Al^{3+}, and some Si^{4+}, provided from the leaching of feldspar, participated in the formation of kaolinite, while the surplus of Si^{4+} caused silification as quartz overgrowths. At least some of the authigenic quartz overgrowths must have been generated during the breakdown of K-feldspars, but whether this solely is responsible for all quartz overgrowths is doubtful as large amounts of quartz overgrowths are also seen in the Haldager Formation sediments with only low K-feldspar content. The K^+ released by the breakdown of K-feldspars may have flushed away by a moving pore fluid to maintain a low K^+/H^+-ratio. In SEM no authigenic illite, or increase of illite with depth, is seen; generally the illite content recorded by X-ray analyses must therefore be of detrital origin. In addition, as the illite shows low crystallinity, no "repair work" or "reconstruction diagenesis" (Bucke & Mankin 1971) seems to have happened here and no illite has served as K^+-acceptor.

The diagenesis seen in this investigation seems to be early, as in the lowermost part of the Børglum 1 sediments well crystalline kaolinite and some calcite still exist. No late diagenesis minerals, like ankerite and dolomite, or pronounced amounts of chlorite (Boles & Franks 1979) have been detected and no other newly formed clay minerals or marked changes in the mixed layer contents are observed. Boles & Frank (1979) showed mineral transformations

Plate 2; 7; Mixed-layer illite-smectite (?) (Børglum 1,6, 3001'). 8; Flakes of chlorite (?) with irregular rounded edges oriented perpendicular to the surface of the host grain (Børglum 1, 24, 4909'). 9; Almost perfect prisms of authigenic quartz crystals (Børglum 1, 15, 4096'). 10; Authigenic quartz overgrowth on a detrital quartz grain (Børglum 1, 24, 4909'). 11; Laths of extensively leached feldspar (Børglum 1, 26, 5001'). 12; Association of authigenic quartz, authigenic kaolinite, and feldspar (Børglum 1, 15, 4096').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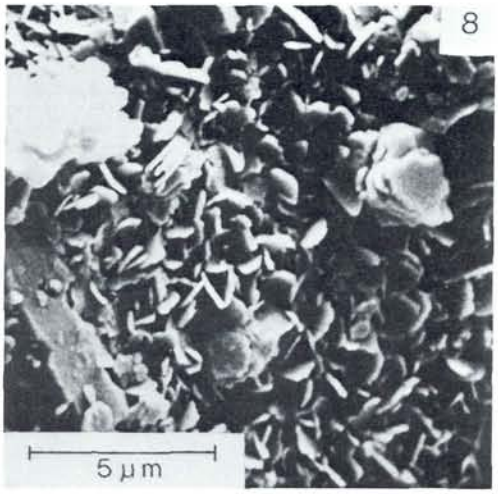
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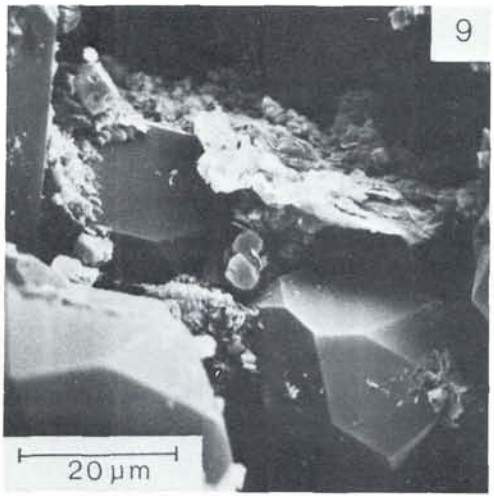
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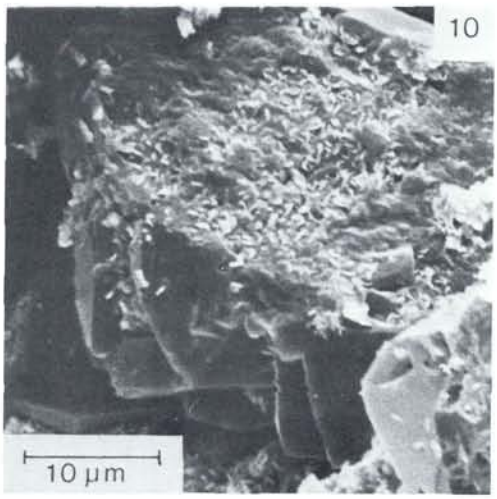
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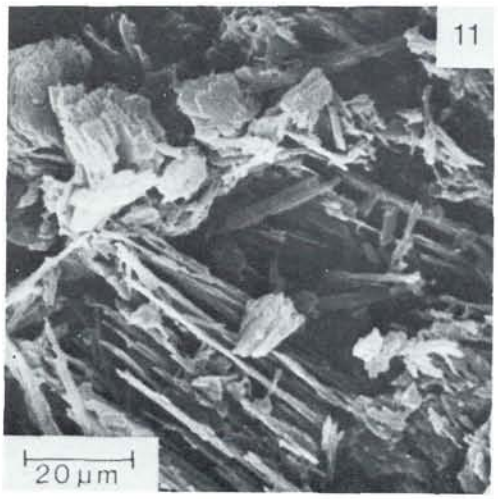
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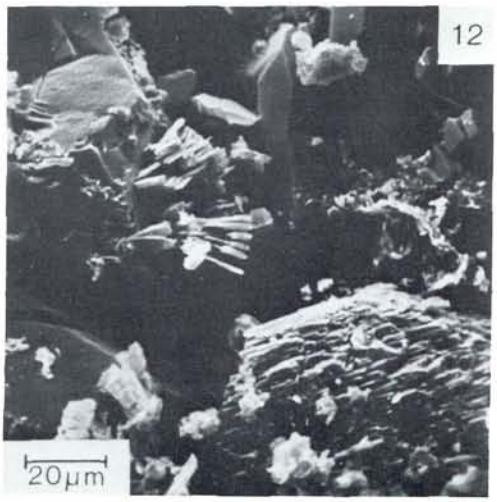
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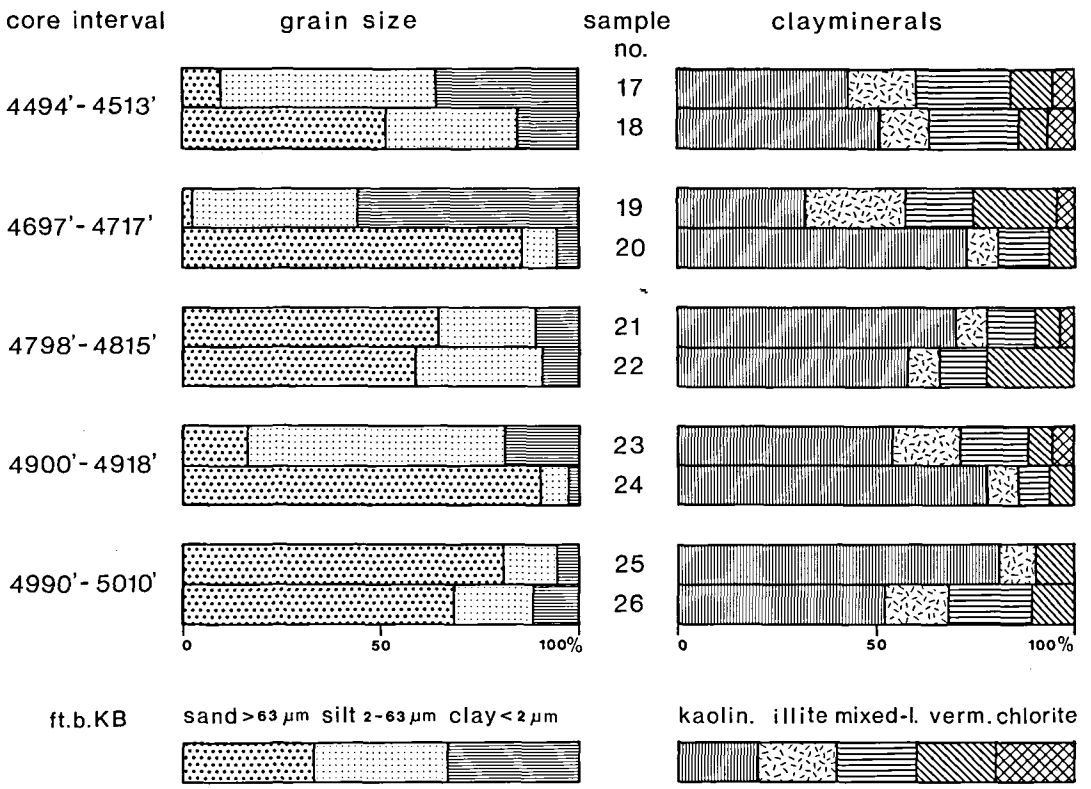

Fig. 6. Comparison of the clay mineral composition of adjacent coarse grained and fine grained samples (Børglum 1).

over a range of temperatures and pressures in sandstones and shales. They proposed a diagenetic model where K-feldspar is unstable during burial, as was also shown by Hower et al. (1976). According to them, K-feldspar disappears over that temperature interval (55–120°) where kaolinite is the most abundant clay mineral, and quartz overgrowths are also present sometimes together with early calcite cement. Ankerite or dolomite first appears at greater depth and temperatures (100–200°C) as replacement of calcite cement by reactions of calcite and the released Fe and Mg from smectite → illite transformations, while the disappearance of kaolinite (150–200°C) is accompanied by an increase in chlorite. This model may be valid for the investigated sediments in the lower temperature region as severe transformations of the minerals have not been detected, taking place at temperatures higher than those reached in the two investigated wells. In Uglev 1 and Børglum 1 uncorrected bottom hole temperatures of 38°C (above the caprock) and 37°C have been measured respectively (possibly 10–15% higher after correction) (Madsen 1978). Even though the deposits probably have been exposed to larger depth with higher temperature before the Cimmerian uplift this had no significant effect on the sediments, as seen by reflectance analysis performed on the more temperature sensitive organic matter (Schmidt 1985). It therefore seems reasonable to discount late, depth dependent, diagenesis as no significant burial effects on clay mineral composition have been detected, and only early diagenetic minerals have been recorded. The mineralogy seems primarily to be dependent on initial composition and early changes in chemistry (Eh and pH) of the circulating pore fluids.

Diagenesis, especially of the Middle Jurassic sandstones, from northern North Sea wells, has been intensively investigated by e. g. Sommer (1978), Dypvik & Vollset (1979), Hancock & Taylor (1978) and Blanche & Whittaker (1978). The authigenic kaolinite generation in these sediments was closely linked with the late Cimmerian tectonic phase (Hancock & Taylor 1978, Sommer

1978) when parts of the North Sea were uplifted and exposed to meteoric water. Circulation of fresh water has probably also happened in the investigated sediments, but the timing of this event is difficult to estimate. Smectite occurs in late Middle Jurassic to Lower Cretaceous sediments (i. e. Flyvbjerg Member of Haldager Formation, Bream and Vedsted Formations) while kaolinite is dominant in the Middle Jurassic and older deposits (i.e. Haldager Sand Member of the Haldager Formation, Fjerritslev and Gassum Formation). It is therefore unreasonable to assume that the kaolinization here can be related to the same tectonic event as mentioned above, since Upper Jurassic sediments should also have been kaolinized then. The kaolinization of the more permeable sandy deposits must therefore have happened earlier, maybe in connection with the mid-Cimmerian movement during the Middle Jurassic or even earlier shortly after deposition of the sediments as also suggested by Nielsen & Friis (1985).

Conclusion

The most remarkable variations in the clay mineral compositions are in kaolinite and smectite contents, with a rapid change from smectite to kaolinite dominance in the Haldager Formation, while most other minerals do not show any depth-related variations.

Kaolinite is dominant in the lower part of the Haldager Formation and in the Fjerritslev and Gassum Formations, with the highest content in the coarse grained samples, while smectite only occurs in the younger sediments.

The high kaolinite content can be related to either provenance or diagenesis. The warm and humid climate from the Rhaetic into Jurassic times, with severe chemical weathering of the land masses, favoured kaolinite formation and might partly be responsible for the generally high kaolinite content in the sediments. The generally higher kaolinite content in the coarse grained sediments of the Gassum Formation and some Fjerritslev and Haldager Formation samples is attributed to authigenic kaolinite generation but the proportion of authigenic and detrital kaolinite is difficult to estimate. Authigenic kaolinite is associated with authigenic quartz overgrowths and probably originates at least partly from the destruction of K-feldspars in a slightly acid pore fluid, possibly fresh water. The excess K^+-ions must have been flushed away as no illitization is seen.

Late diagenesis due to the depth of burial had no significant effect on the mineral composition as only early diagenetic minerals have been detected. The high temperatures required for severe mineral transformations were not reached in the two investigated wells, or temporary submergence to larger depth before the Cimmerian uplift had no significant effect. The clay mineral compositions observed in this investigation seem primarily to have been influenced by the initial mineralogy and geochemical environment rather than depth of burial.

The timing of the kaolinization cannot be estimated. A relationship to late Cimmerian movements as suggested by other authors for the Middle Jurassic sandstones in the North Sea is not likely; the kaolinization must have happened earlier, possibly connected to the mid-Cimmerian movements or even earlier shortly after deposition.

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Dansk sammendrag

Lermineralsammensætningen af Rhæt – Jura – Nedre Kridt sekvensen fra boringerne Børglum 1 og Uglev 1 er blevet undersøgt med henblik på at klarlægge sedimenternes diageneseforhold. Undersøgelserne omfatter udover kornstørrelsesanalyse af sedimenterne røntgendiffraktionsanalyser og scanningelektronmikroskopi i kombination med energidispersive analyser af især lermineralerne.

Lermineralfordelingen viser i det væsentlige et hurtigt skift fra smectit til kaolinitdominans med tiltagende dybde. Mens kaolinit dominerer i de ældre sedimenter fra den nedre del af Haldager Formationen, Fjerritslev og Gassum Formationerne, er smectit dominerende i de yngre sedimenter fra den øvrige del af Haldager Formationen, Bream og Vedsted Formationerne. Denne ændring henføres fortrinsvis til forskelle i den primære mineralsammensætning og det geokemiske miljø frem-for en dybdeafhængig diagenetisk påvirkning. Selv om en tidlig diagenese har ført til en authigen kaolinitdannelse, kan en direkte dybderelateret sen diagenese ikke observeres.

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