

DISTRIBUTION, GENESIS AND THERMAL BEHAVIOUR OF CLAY MINERALS IN THE MIOCENE ARGILLACEOUS ROCKS IN QATAR, ARABIAN GULF

By

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ABSTRACT

XRD and DTA were utilized to investigate quantitatively the clay-mineral composition of the argillaceous rocks in four Miocene stratigraphic sequences located in western and southwestern Qatar. These rocks belong to the Dam Formation and are more concentrated in its lower Member "A" than in the overlying Member "B" which is dominated by a carbonate-evaporite facies. The reported clay-mineral suite consists of (in order of decreasing abundance): illite, kaolinite, attapulgite and Ca, Mg-montmorillonite. Illite and kaolinite are intimately associated and their crystallinity characters at a given stratigraphic horizon are closely similar. Well-crystallized attapulgite is present mainly in the marlstones especially those associated with evaporites. The occurrence of montmorillonite is restricted to the siltstones occupying the base of Member "A". Generally, Member "B" sequence consists of three thick illite-attapulgite-kaolinite zones alternating with two thin illite-kaolinite zones.

Evidently, illite and kaolinite were formed by detrital inheritance from weathering horizons and/or soils on the Precambrian rocks in western Saudi Arabia. Montmorillonite seems to have been neformed in the basin of deposition under conditions of alkalinity and high Al^{3+}/Mg^{2+} ratios. It is also probable that the mineral was formed diagenetically through hydration of illite by pore solutions having high H^+/K^+ ratios. Attapulgite had also originated in the basin of deposition mostly by alteration of montmorillonite and/or magnesium-rich debris derived from weathered basic igneous rocks.

INTRODUCTION

The Miocene argillaceous rocks in Qatar constitute a major part of the Dam Formation (Middle to Late Miocene) the exposures of which are restricted to the

western and southwestern parts of the country. The formation overlies with a slight unconformity the Early to Middle Eocene Dammam Formation and acquires a maximum thickness of about 80 m in areas where the overlying Hofuf Formation (Late Miocene to Early Pliocene) is preserved. The rock unit consists of calcareous claystones, marlstones and limestones with occasional evaporites.

Abu-Zeid and Khalifa (1983) investigated the lithologic and biogenic characters of the Dam Formation in the Al-Nakhsh section (southwestern Qatar) and subdivided it into two informal members designated "A" (older) and "B" (younger). Member "A" (about 28 m thick) is made up of calcareous claystone beds alternating with limestones whereas Member "B" (about 45 m thick) is dominated by a carbonate-evaporite facies with a relatively restricted occurrence of the argillaceous sediments. The work of Abu-Zeid and Khalifa, which was mainly devoted to the investigation of the carbonates, showed that Member "A" was laid down in a shallow marine environment of normal salinity and weakly oxidizing conditions. Member "B" was deposited under relatively arid conditions in a tidal flat environment having a wider range of depositional energy, a lower rate of terrigenous deposition and a higher net evaporation.

The only published work on the Miocene argillaceous rocks was carried out by the present authors (Hilmy *et al.*, 1987) and comprised the investigation of the areal and vertical variations in their petrographic characters. The results obtained indicated that the detrital fraction of the argillaceous rocks was inherited from the Precambrian rocks of the Arabian Shield in western Saudi Arabia. Moreover, Hilmy *et al.* (op. cit.) concluded that the Miocene sea was advancing from north to south and suggested a depositional model which is, to a large extent, consistent with that proposed by Abu-Zeid and Khalifa (1983).

The prime objective of this paper is to study the spatial and temporal clay-mineral distribution patterns in the Miocene argillaceous rocks. The possible genesis and provenance of the reported clay minerals are discussed. Besides, their thermal behaviour is investigated for probable future applied aspects.

MATERIAL AND METHODS

Fifty seven argillaceous samples were collected from four stratigraphic sequences located in western and southwestern Qatar (Fig. 1). The Al-Nakhsh sequence is the most complete section as it includes the whole succession of Member "B" and the upper and middle parts of Member "A". The lower part of the latter rock unit constitutes the southern Nafkha section. The Kharzat ad Darb and the Clay Quarry sequences are located to the east and north of the Al-Nakhsh section respectively and represent the lower and middle parts of Member "A". A *Fibularia damensis* bed was used as a marker horizon for the lithostratigraphic correlation between these sequences (Fig. 1).

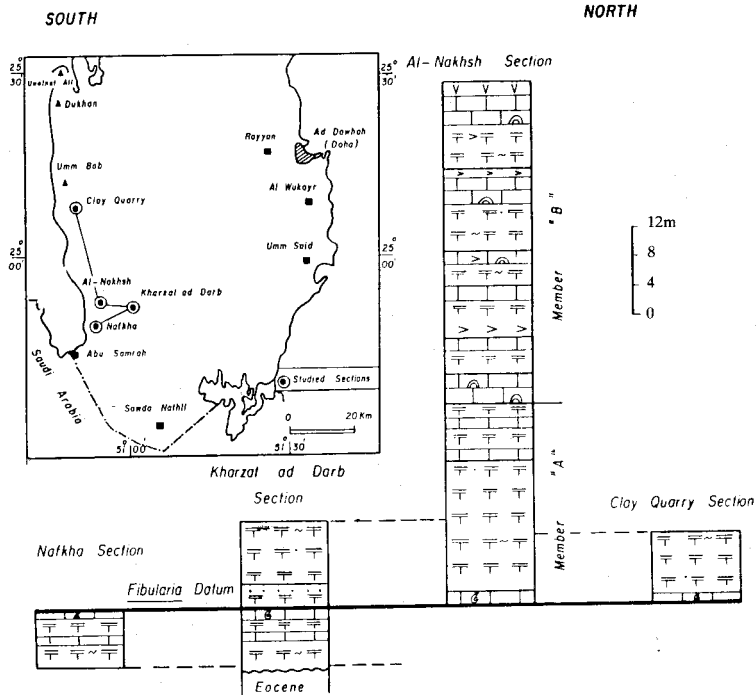


Fig. 1: Location and lithostratigraphic correlation of the studied sections (after Hilmy *et al.*, 1987).

X-ray diffraction analysis was utilized to investigate the clay and nonclay mineralogic composition of 36 samples representative of the various lithologic types. For each sample, one randomly-oriented particle mount was prepared from the whole-rock specimen using the powder-press technique. Four oriented-particle mounts were prepared from the purified clay fraction (2 μ m) applying the smear-on glass slide technique (Gibbs, 1965). The latter mounts were X-rayed in the natural state, after glycol treatment and after one hour of heat treatment at 180°C and 550°C.

The X-ray diffraction data were supplemented by DTA which was carried out on 17 samples representing the various clay-mineral assemblages. The analysis was undertaken in a flowing nitrogen atmosphere using a platinum sample holder and calcined alumina as a reference.

RESULTS AND DISCUSSION

Mineralogy

The obtained X-ray data were interpreted using the A.S.T.M. cards together with data published by Brown (1961), Brindley (1961), Mac Ewan (1961), Deer *et al.* (1962) and Carroll (1970). Quantitative determination of the reported clay minerals was carried out following the procedure adopted by Shaw *et al.* (in Griffin, 1971). This procedure was slightly modified to include the estimation of the relative proportions of attapulgite by direct comparison of its peak areas with those of illite.

The nonclay-mineral assemblage in the Miocene argillaceous rocks consists of (in order of decreasing abundance in samples): quartz, dolomite, halite, calcite, gypsum, plagioclase, orthoclase, muscovite and glauconite. Quartz is present in almost all the investigated rocks except for the highly calcareous claystones at the base of the Al-Nakhsh section and the middle part of its Member "B". Dolomite and halite are also very common but do not appear in the Clay Quarry sequence. In the Al-Nakhsh section, halite was found to be more abundant in the rocks of Member "B" than in those of Member "A". Albite is reported in the sandy claystones at the top of the latter rock unit and in the middle part of the Nafkha sequence which contains also orthoclase and muscovite. The occurrence of glauconite is restricted to the top of the Clay Quarry section and the equivalent succession of Member "A" at Al-Nakhsh.

The clay-mineral suite in the Miocene argillaceous rocks is composed of (in decreasing order of abundance): illite, kaolinite, attapulgite and Ca, Mg-montmorillonite. The distribution of these minerals permits the subdivision of each of the investigated sequences into a number of clay-mineral zones (Figs. 2 to 6).

Illite and kaolinite are present in almost all the investigated rocks except for the sandy claystones near the top of the Nafkha section. The crystallinity characters of the two minerals and their variation throughout time are closely similar. Both minerals are poorly to moderately crystallized in the lower parts of the successions of Member "A" while better crystallized in their upper parts. Well-crystallized attapulgite is reported only in the marly claystones and marlstones occupying the top of Member "A" and several horizons in Member "B" in the Al-Nakhsh section, and also the lowermost and upper parts of the Kharzat ad Darb sequence. Montmorillonite appears in the middle and upper parts of the Nafkha section either as a sole constituent or accompanying illite and kaolinite.

Spatial and Temporal Distribution of Clay Minerals

Member "A": the lowermost part of Member "A" exposed at Kharzat ad Darb has a clay-mineral suite composed of illite (~37%), kaolinite (~7%) and attapulgite (~56%) at the base of the sequence (zone I), while it consists of illite (average

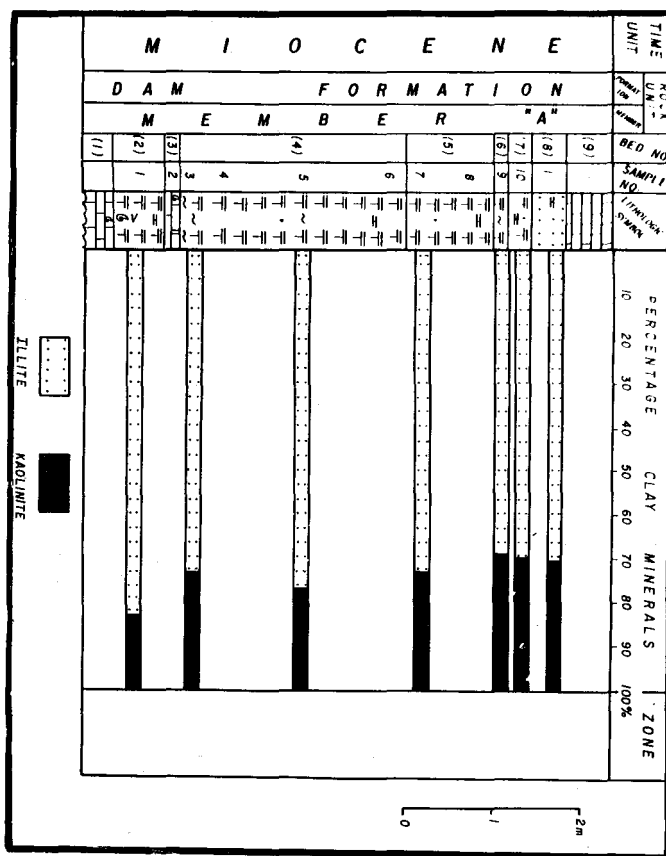


Fig. 2: Percentage-constituent of the clay fraction of the rocks in the Clay Quarry section.

~86%) and kaolinite (average ~14%) only in its middle and upper parts (zone II). The stratigraphically equivalent succession exposed at Nafkha has a remarkably different clay-mineral composition as attapulgite disappears and its place is taken by montmorillonite. The association at the base of the sequence consists of illite (average ~70%) and kaolinite (average ~30%) (zone I), while it is made up of illite (56 and 60%), kaolinite (25 and 24%) and montmorillonite (19 and 16%) in the middle and upper parts (zones II and IV). The latter two zones are separated by a layer composed solely of montmorillonite (zone III).

The middle part of Member "A" exposed in the Al-Nakhsh section has a clay-mineral suite consisting of illite (average ~78%) and kaolinite (average ~22%). A quantitatively identical composition is reported in the equivalent Clay Quarry succession. The vertical fluctuations in the relative proportions of the two

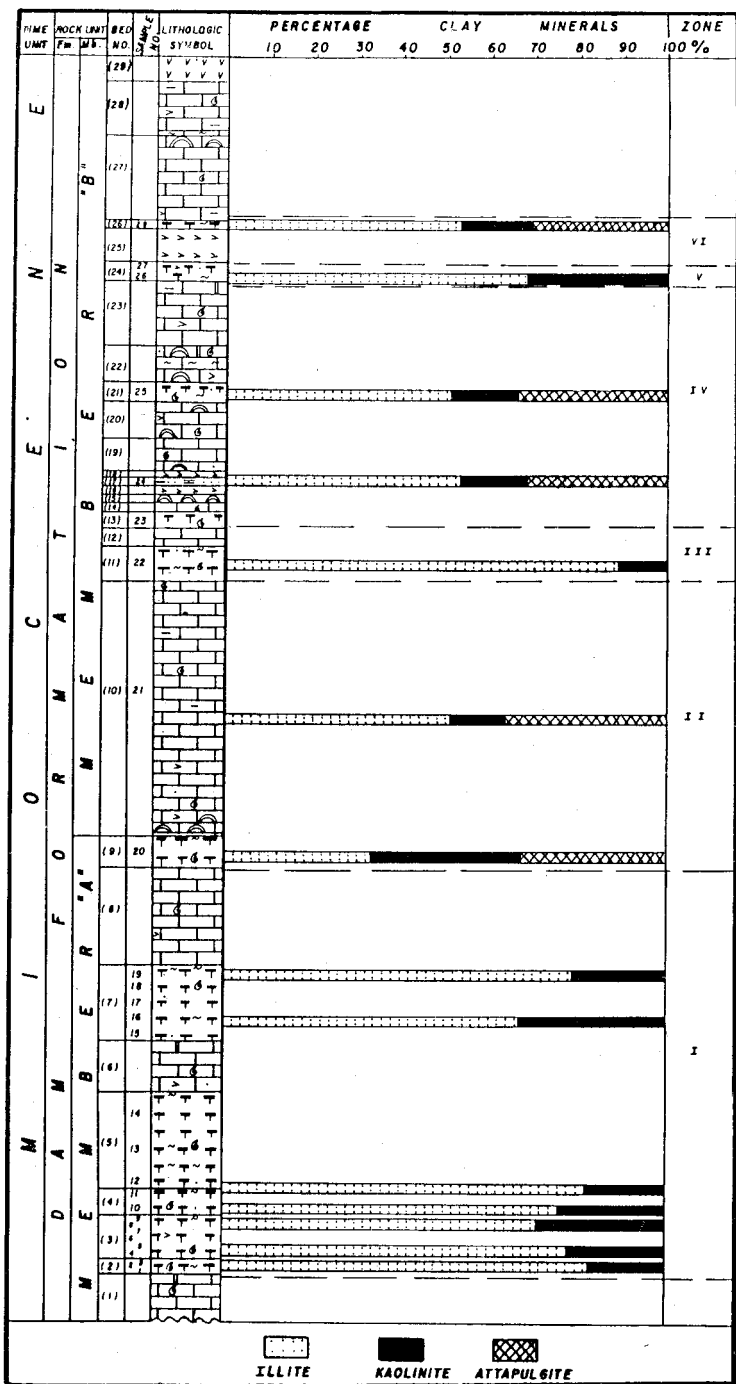


Fig. 3 : Percentage-constituent of the clay fraction of the rocks in the Al-Nakhsh section.

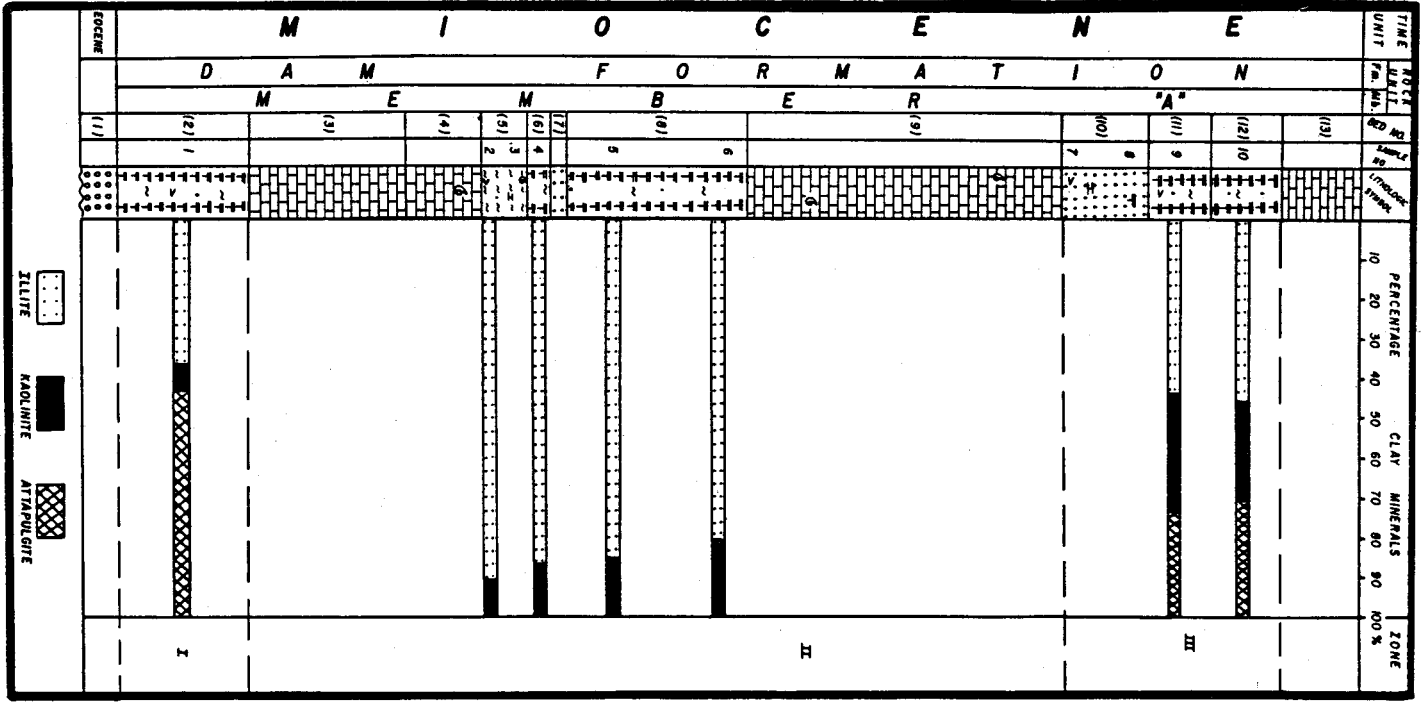


Fig. 4 : Percentage-constituent of the clay fraction of the rocks in the Kharzat and Darb section.

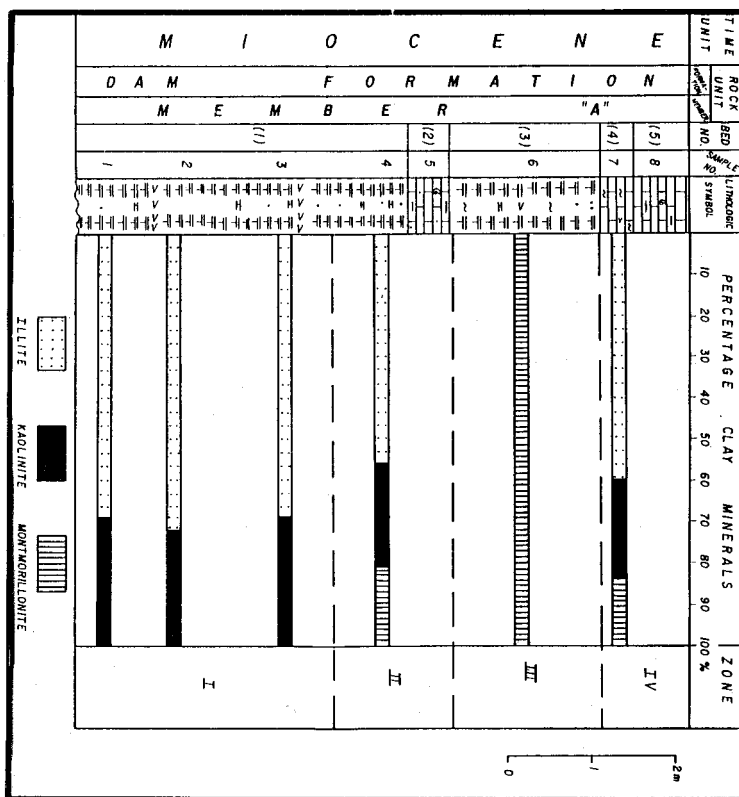


Fig. 5 : Percentage-constituent of the clay fraction of the rocks in the Nafkha section.

minerals in both sequences are also very similar. These are represented by a remarkable upward increase in kaolinite percentages accompanied by a decrease in illite content. A similar situation is noticed in the equivalent succession at Kharzat ad Darb. However, attapulgite (average ~28%) appears at the top of the sequence at the expense of illite (average ~45%) while the concentration of kaolinite remains unchanged (average ~26%) (zone III).

The upper part of Member "A", which is exposed only at Al-Nakhsh, has a clay-mineral composition consisting of illite (average ~ 73%) and kaolinite (average ~ 27%) (zone I). Attapulgite (~33%) appears at the contact with the overlying Member "B" (zone II) where the relative proportion of illite shows a marked decrease (~33%).

The spatial variation in the average clay-mineral composition of Member "A" rocks is illustrated in Fig. 7. The figure shows a slight decrease in the relative

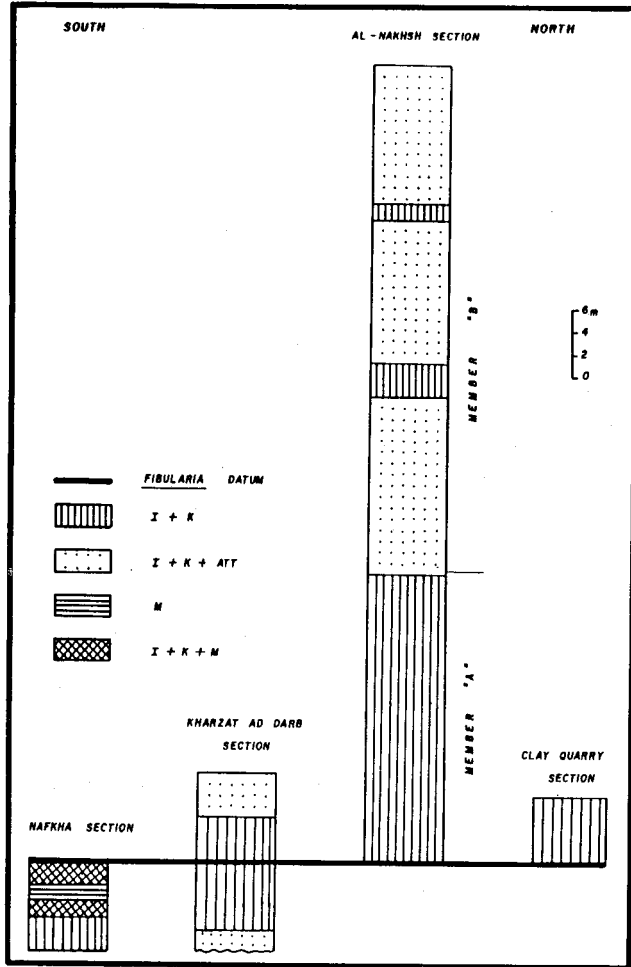


Fig. 6 : Distribution of clay-mineral zones in the Miocene sequences.

proportions of illite and kaolinite from north to south accompanied by the appearance of attapulgite and montmorillonite in the eastern and southern sequences respectively.

Member "B": the clay-mineral suite in this rock unit lacks montmorillonite. It consists of illite (average ~ 61%), attapulgite (average ~ 22%) and kaolinite (average ~ 17%). There is a remarkable upward increase in the relative proportions of attapulgite and kaolinite at the expense of illite content. Generally, Member "B" sequence consists of three thick illite-attapulgite-kaolinite zones alternating in a cyclic pattern with two thin illite-kaolinite zones (Figs. 3 and 6).

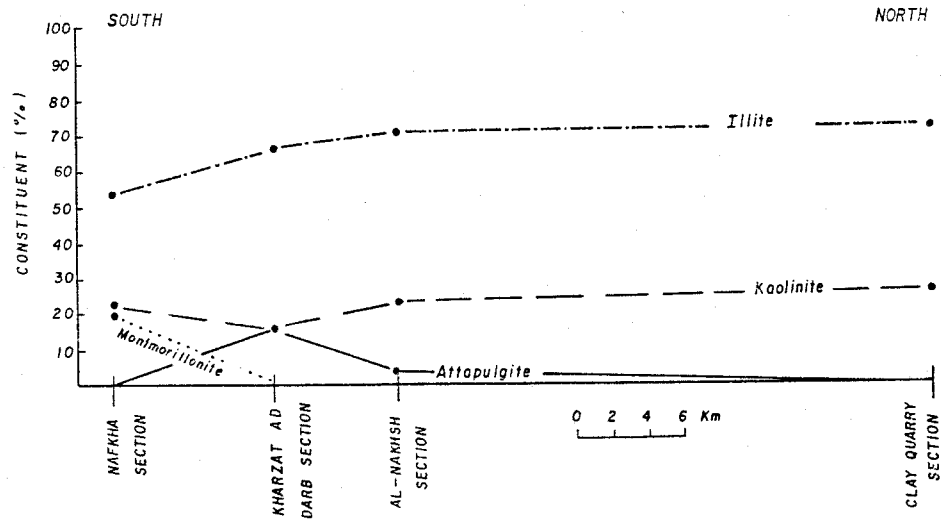


Fig. 7 : Spatial distribution of clay minerals in the argillaceous rocks of Member "A".

Genesis

The nature of the clay-mineral assemblage reported in the Miocene argillaceous rocks suggests that various processes of clay-mineral formation were operative. However, it seems that detrital inheritance played the most important role in the genesis of the great bulk of these minerals.

Illite and kaolinite, which constitute the major part of the clay-mineral suite, were evidently inherited from weathering horizons and/or soils on the same source rock and transported by freshwaters and/or wind to the basin of deposition. Criteria for such origin include: (i) the intimate association and widespread occurrence of the two minerals in the investigated rocks; (ii) the remarkably narrow range of variation in their relative proportions throughout the stratigraphic sequences (iii) their low to moderate crystallinity which suggests prolonged transportation and precludes in situ formation; (iv) the close similarity in their crystallinity at a given stratigraphic level as well as throughout the successions; and (v) the well-known high stability of the two minerals during weathering and transportation (Perrin, 1971). In a previous study carried out by the present authors (Hilmy *et al.*, 1987), it was indicated that the Precambrian rocks in western Saudi Arabia represented the only possible source for the detrital fraction of the Miocene argillaceous rocks. Consequently, it is most probable that the reported illite and kaolinite were originated from feldspars and aluminosilicates in these rocks under mild to strongly acidic conditions and favourable potassium equilibrium. These conditions involved considerable leaching with the subsequent removal of Na^+ and the divalent cations as Ca^{2+} , Mg^{2+} and Fe^{2+} .

The possibility of formation of illite by diagenetic transformation from montmorillonite can be precluded since the latter mineral is reported in the oldest rocks of Member "A" (at Nafkha) while it disappears in the overlying younger sediments. The conversion of montmorillonite into illite requires deep burial involving high temperatures and pressures (Lahann, 1980; McHargue and Price, 1982 and Dypvic, 1983) and is controlled by geologic time (Hower *et al.*, 1976).

The fact that the conditions of formation of montmorillonite in weathering horizons and soils are not favourable for the genesis of illite and kaolinite precludes its formation by detrital inheritance. This is further confirmed by the remarkably restricted occurrence of the mineral in the investigated rocks. It is believed that montmorillonite was neoformed in the basin of deposition during the sedimentation cycle of carbonates and calcareous claystones. The predominance of these lithologies indicates the prevalence of alkaline chemical characters which greatly favour montmorillonite neoformation (Milot, 1970). The process requires conditions of high $\text{Al}^{3+}/\text{Mg}^{2+}$ ratios unfavourable for the formation of attapulgite which satisfactorily explains the absence of the latter mineral in the rocks containing montmorillonite.

It is also possible, if not probable, that montmorillonite was diagenetically transformed from illite. During deep burial, montmorillonite could have been formed through hydration of illite by pore solutions having high H^+/K^+ ratios (Velde and Nicot, 1985). Such an origin for montmorillonite may explain the fact that the mineral is reported only in the oldest rocks of Member "A" (at Nafkha) where it shows some sort of mixed-layer characters indicative of the presence of illitic layers.

The remarkably high crystallinity acquired by the reported attapulgites precludes inheritance from older attapulgite-bearing weathering horizons, soils and/or sediments (e.g. the Permo-Triassic Khuff limestones in Saudi Arabia). This mineral is stable only in its genetic environment and is vulnerable during weathering and transportation. Hence, it is most likely that attapulgite was originated in the basin of deposition by neoformation or, more effectively, transformation processes which were greatly favoured by the prevailed alkalinity. The latter process involved the alteration of montmorillonite and/or magnesium-rich debris derived from weathered basic igneous rocks. Periods of highly evaporitic conditions resulted in the frequent deposition of gypsum with the consequent increase in magnesium in solutions which became favourable for the alteration process. This satisfactorily explains: (i) the attapulgite-montmorillonite compensatory relationship observed in the Miocene argillaceous rocks; and (ii) the widespread occurrence of attapulgite in Member "B" sequence which comprises considerable thicknesses of evaporitic limestones and evaporites and contains stromatolites.

The spatial variation in the clay-mineral composition of Member "A" rocks could be attributed to the situation of the studied sequences in the basin of deposition. In the previous study undertaken by the writers (Hilmy *et al.*, 1987), it was indicated that the southern Nafkha section was located near the margin of that basin while the northern Clay Quarry sequence was situated in a slightly deeper zone and the other two successions were lying in between. Such distribution is in accordance with: (i) the predominant presence of the typical marine clay mineral illite in the Clay Quarry and the Al-Nakhsh sections and the slight decrease in its relative proportions from north to south; (ii) the remarkable increase in grain size and crystallinity of the detrital minerals illite and kaolinite in the southern sequence; and (iii) the appearance of attapulgite in the Kharzat ad Darb succession. On the other hand, the vertical variation in the clay-mineral composition of the Miocene argillaceous rocks is consistent with the depositional model suggested by Abu-Zeid and Khalifa (1983). These workers showed that the deposition of the Dam Formation was accompanied by a progressive regression of the sea which was intermittently interrupted by several transgressions during the deposition of Member "B" sequence. This explains the upward increase in the relative proportions of kaolinite and attapulgite at the expense of illite and the cyclic pattern displayed by the clay-mineral zones in Member "B"

Thermal Behaviour of Clays

The data obtained from the DTA curves of the clay fractions of the Miocene argillaceous rocks (Figs. 8 to 11) confirm the presence of various associations of illite, kaolinite, attapulgite and montmorillonite. These curves represent an overlap between the reaction peaks characteristic of the minerals in the association. Consequently, the reported peak intensities, sizes and temperatures are greatly affected not only by the particle size and crystallinity of each of the contributing minerals but also by their relative proportions in the mixture.

The dehydration of the reported illites is represented by an abrupt and considerable water loss which produced a sharp to very sharp, moderate to intense endotherm at 94 to 120° C. In a few instances, the process was only completed at a slightly higher

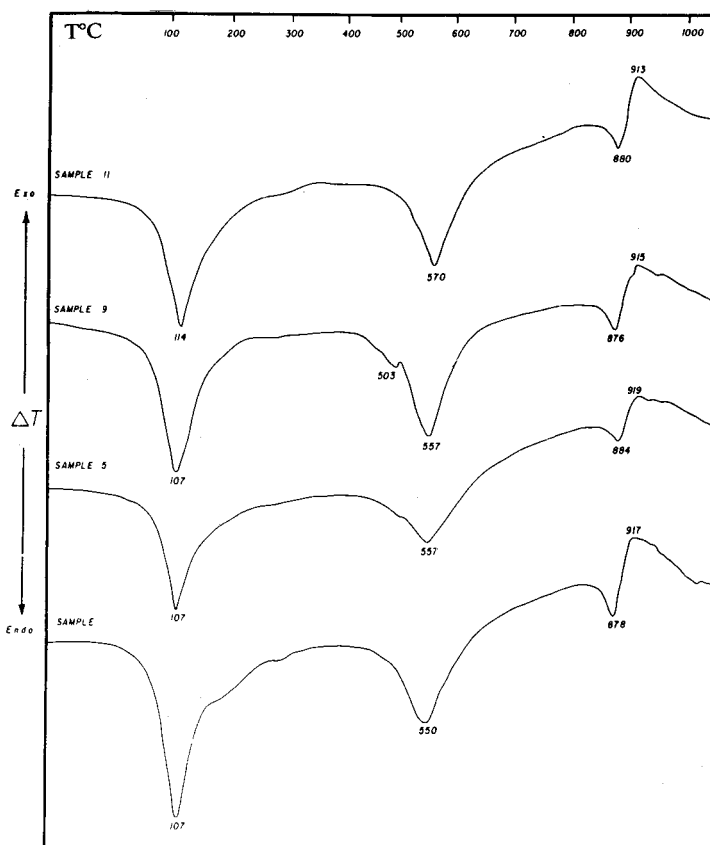


Fig. 8 : DTA curves of the clay fractions of the Clay Quarry samples.

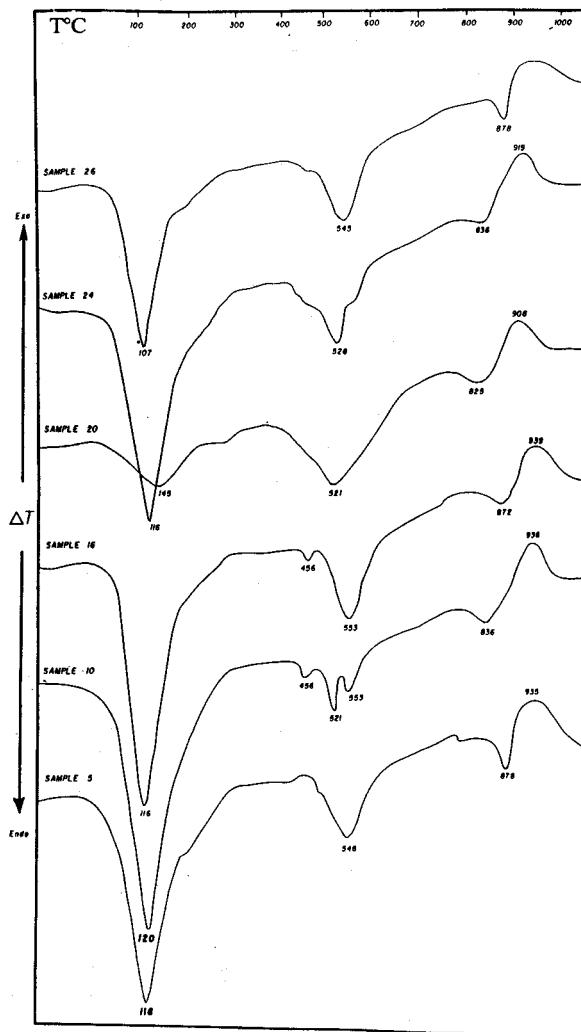


Fig. 9 : DTA curves of the clay fractions of the Al-Nakhsh samples.

temperature (145°C). The dehydration peaks of the illites in the eastern and southern sequences have relatively smaller sizes which suggest that they acquire relatively larger particle sizes and/or better crystallinity (Wilson, 1971). On the other hand, the dehydroxylation of illites is rather gradual and less pronounced as indicated by the broad, weak endotherm at 484 to 560°C. In the well-crystallized species, however, the final stage of dehydroxylation, accompanied by a structural loss, was reached only at 825 to 884°C and is represented by the endothermic part of an S-shaped peak. The exothermic part of the latter peak (868 to 939°C)

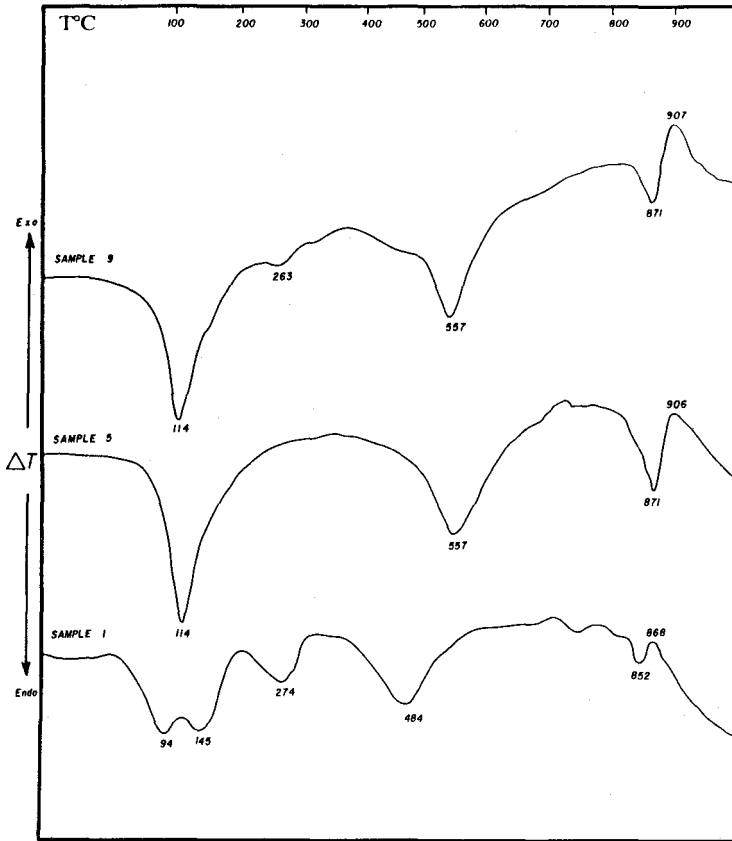


Fig. 10: DTA curves of the clay fractions of the Kharzat ad Darb samples.

corresponds mostly to the formation of spinel and amorphous glass (Grim and Bradley, 1940).

Although the absorbed water content of the reported kaolinites is low, their dehydroxylation is much more pronounced than for the other clay minerals. In the relatively poorly-crystallized kaolinites, the process started with an initial abrupt loss of a part of the hydroxyl water which produced a sharp, very weak to weak endotherm at 456 to 503°C. On the other hand, the dehydroxylation of the moderately and well-crystallized species was completed only at a higher temperature (484 to 560°C) and a meta-kaolin was formed. The final stage of dehydroxylation and the complete loss of the meta-kaolin structure (Grim and Bradley, 1948) are represented by the endothermic part (825 to 884°C) of an S-shaped peak. The exothermic part of the latter peak occurs at 868 to 939°C and

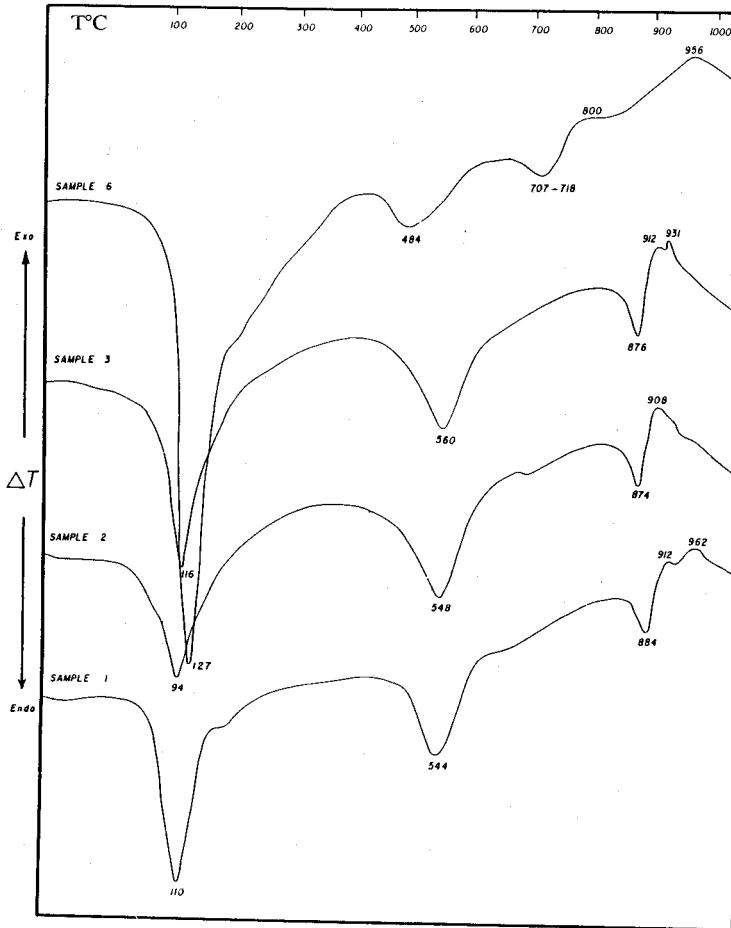


Fig. 11: DTA curves of the clay fractions of the Nafkha samples.

corresponds mostly to an initial stage in the transformation of the meta-kaolin into mullite and/or γ - Al_2O_3 . Such a relatively wide range of dehydroxylation temperatures, which are generally lower than those characteristic of typical well-crystallized kaolinites, could be attributed to the effect of dilution with other clay minerals and the considerable variation in the mineral crystallinity. The well-crystallized species are encountered in the Nafkha sequence only and their thermograms exhibit a sharp exothermic peak at 962°C.

The dehydration of montmorillonites is markedly pronounced and seems to be a quite complex process as indicated by the structure of the corresponding very intense endotherm at 127°C. Such complexity of the peak was attributed by

Neumann (1977) to the presence of three components representing the removal of water molecules held in different ways. On the contrary, the dehydroxylation of the mineral is much less pronounced and produced a broad, weak endotherm at 484°C. The broadness of this peak and its relatively low temperature confirm the results of X-ray diffractometry which showed that the smectite is dioctahedral (montmorillonite) rather than trioctahedral and that the exchangeable cations are represented by calcium rather than sodium (Neumann, 1977). The final stage in the dehydroxylation of montmorillonites occurs at 707 to 718°C and is accompanied by their structural breakdown (Grim and Bradley, 1940). Evidently, the reported montmorillonites have a low iron content as may be pointed out by the presence of an exothermic shoulder at 800°C which is related mostly to the formation of *B*-quartz (Bradley and Grim, 1951). The thermogram of a sample composed entirely of montmorillonite exhibits a broad, weak endotherm at 956°C. This peak may correspond to the formation of enstatite, mullite or anorthite in addition to *B*-quartz depending on the amount of magnesium and the exchangeable Ca²⁺ present (Bradley and Grim, 1951).

The dehydration of the reported attapulgite is relatively abrupt and moderately pronounced as indicated by the sharp, medium-sized endothermic peak at 94 to 114°C. In some cases, the process was completed only at 145°C suggesting the presence of better-crystallized species. A broad, weak to medium endotherm was reported in a few thermograms at 263 to 274°C. This peak may be attributed to final stages in the dehydration of the mineral or, probably, an initial stage in its dehydroxylation comprising the removal of a small portion of its hydroxyl water accompanied by no structural changes (Long Chambon, 1936). The main dehydroxylation stage of attapulgite, which is accompanied by the contraction of its structure, occurs at 484 to 557°C. In the well-crystallized species, however, the process was completed only at 825 to 870°C and produced a broad to sharp, weak endotherm.

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التوزيع والنشأة والسلوك الحراري لمعادن الطين في الصخور الطينية التابعة لعصر الميوسين في قطر - الخليج العربي

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تشتمل هذه الدراسة على فحص التوزيع الكمي لمعادن الطين في الصخور الطينية التابعة لعصر الميوسين في أربعة قطاعات إستراتيجية تقع في غرب وجنوبي غرب قطر . وتتبع هذه الصخور تكوين الدام حيث تتركز في جزئه السفلي (العضو أ) بينما يتميز جزؤه العلوي (العضوب) بسيادة واضحة للصخور الجيرية والمتبخرات . وقد أوضحت نتائج الدراسة أن طاقم معادن الطين يتكون من : الإيليت والكاولينيت والأتابولجيت والمونتموريللونيت (مرتبة حسب التناقص في درجة الإنتشار) . وقد لوحظ أن هناك علاقة قوية تربط بين معدني الإيليت والكاولينيت ، كما أن هناك تشابهاً واضحاً في خصائص تبلورهما في المستوى الإستراتيجرافي المعين . ويتميز معدن الأتابولجيت بدرجة تبلور مرتفعة ويرتبط وجوده بصخور المارل المصاحبة لرواسب المتبخرات . وينحصر وجود المونتموريللونيت في الصخور الغرينية التي تحتل الجزء السفلي من العضو (أ) .

وتشير الدلائل إلى أن كلا من الإيليت والكاولينيت عبارة عن مواد فتاتية استمدت من نطاقات تجوية صخور ما قبل الكامبري الموجودة في غرب المملكة العربية السعودية ، بينما يرجع إستحداث معدن المونتموريللونيت في حوض الترسيب تحت ظروف قلووية وفي وجود تركيزات مرتفعة من Al^{+3} / Mg^{+2} ومن المحتمل أيضاً نشأة المعدن أثناء العمليات اللاحقة للترسيب عن طريق نموه الإيليت بواسطة المحاليل التي تميزت بنسب مرتفعة من H^{+} / K^{+} أما بخصوص معدن الأتابولجيت فتشير الدلائل إلى إستحداثه أيضاً في حوض الترسيب عن طريق تحول المونتموريللونيت أو الفتات الغني بالماغنسيوم والذي إستمد من نواتج تجوية الصخور النارية القاعدية ، وقد أدت الفترات التي تميزت بمعدلات تبخير مرتفعة إلى ترسيب الجبس بكثافة مما ترتب عليه زيادة تركيز الماغنسيوم في المحاليل التي أصبحت ملائمة لعملية التحول .