

DIAGENESIS OF CLAY MINERALS FROM LOWER CRETACEOUS SHALES OF NORTH EASTERN BRITISH COLUMBIA*

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Abstract—Clay minerals from shale outcrops of the Lower Cretaceous Buckingham Formation (4250 ft thick) were investigated in order to assess their degree of diagenesis and their oil-generating potential. Crystallinity index, sharpness ratio, per cent of illite which is the 2M polymorph and presence of discrete minerals have been studied in the whole clay fraction, while the very fine clay fraction has been subjected to X-ray diffraction, differential thermal, thermogravimetric, differential thermogravimetric, i.r. spectroscopy, surface area and chemical analyses. With information derived from these studies and from published data, a classification scheme was devised which relates variation of clay mineralogy to diagenetic stages and burial depth.

Data on the $<2\ \mu\text{m}$ size fraction show that the crystallinity index decreases while the sharpness ratio and per cent of illite which is the 2M polymorph increase with burial depth. Results on the $<0.08\ \mu\text{m}$ fraction reveal that a three-component interstratified clay mineral exists. In addition, Fourier transform calculations and chemical and physicochemical analyses indicate that both the ratio of the amounts of non-hydrated clays (illite) to hydrated clays and the K₂O content of clays increase with burial depth; cation exchange capacity and surface area decrease with burial depth.

Based upon a classification scheme, which was devised by combining criteria and data derived from the studies of Weaver (1961a), Kubler (1966), Burst (1969) and Dunoyer de Seconzac (1970), the upper and middle parts of the formation (upper 3250 ft) fall within the middle stage of diagenesis whereas the lower part (1000 ft) is allocated to the beginning of late diagenesis. In terms of Burst's (1969) work, the upper 3250 ft are transitional between the stability and dehydration zones indicating that, prior to uplift, hydrocarbons may have been in the process of migration. The lower 10000 ft of the formation are in the restricted dehydration zone, indicating that hydrocarbon migration should have been completed.

INTRODUCTION

The mineralogy of clays has been used to estimate the degree of sediment diagenesis, to reconstruct the geochemical history of sedimentary basins and, very recently, to assess the potential of rocks as sources of petroleum (Weaver, 1960, 1961b, 1967; Powers, 1959, 1967; Khitarov and Pugin, 1966; Long and Neglia, 1968; Muffler and White, 1969; Perry and Hower, 1970, 1972; Weaver and Wampler, 1970; Merchink *et al.*, 1971; Teodorovich and Konyukhov, 1970; von Moort, 1971; Weaver *et al.*, 1971; Cordell, 1972 and Sarkissyan, 1972).

Burst (1969) has shown that, during the transformation of smectite to illite, a dehydration process takes

place which is strongly correlated with the production of hydrocarbons. The transformation occurs at temperatures not greater than 150°C and is accompanied by production of large amounts of water (Perry and Hower, 1972). Price (1973) concluded that deeply buried hot water at 150°C could dissolve large amounts of hydrocarbons. As the transformation of smectite to illite results in the production of large amounts of water at temperatures considered by many workers to be adequate for hydrocarbon generation (Colombo, 1967; Andreev *et al.*, 1968; Kartsev *et al.*, 1971; Yerofeev, 1972), it seems likely that the process has often been important in primary oil migration.

In this work, we have studied diagenesis in shale samples from the Buckingham Formation as a function of: (1) the presence of discrete clay minerals; (2) the sharpness ratio and crystallinity index of illite; (3) the

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per cent of illite which is the 2M polymorph; (4) the ratio of hydrated to non-hydrated layers in the interstratified clay minerals as determined by Fourier transform and chemical methods; (5) the K₂O content, cation exchange capacity and surface area measurements of mixed layer clay minerals. In addition, differential thermal analysis, thermogravimetric analysis, differential thermogravimetric analysis and i.r. spectroscopic measurements were done in order to aid the interpretation of the chemical analysis and elucidate some results obtained from the X-ray work.

All these data have been related to the maximum depth of burial of the shale samples and have been combined with that of others to devise a scheme for the classification of the degree of diagenesis. Using Burst's (1969) data, an effort is made to assess the oil generating potential of the formation during its earlier history.

EXPERIMENTAL

Materials

Shale samples were collected from an outcrop of the Lower Cretaceous Buckinghorse Formation, which is 4,250 ft thick. This rock unit crops out in the vicinity of the Muskwa and Tetsa Rivers, 58°40' N and 124°07' W some 75 miles south of Scatter River in northeastern British Columbia. Samples from this location were collected and diagenetic studies were carried out on this formation because detailed lithologic, stratigraphic and paleontologic data are available (Stott, 1969, 1968a, b, 1972).

Initially, 420 samples, not affected by weathering, were collected at 10-ft intervals and the mineral components were identified in all of them by X-rays. Clay mineralogy was studied in some detail in 50 samples. Finally, the clay mineralogy was studied in great detail in the less-than-2 μm fraction of seven selected samples in order to document changes that take place during diagenesis. The sampling interval was about 650ft and the probable burial depth of the 7 samples

prior to uplift and erosion is obtained by extrapolation from Stott's (1972) restored stratigraphic cross-section and is presented in Table 1.

Methods

Sample preparation. All research work was carried out on the <2 μm fraction. To obtain this fraction, the rock samples initially were ground in a Bleuler mill for 20 sec. The powdered samples were mixed with distilled water at a ratio of 1:10 and then sonified for 30 min at 22 ± 3 kHz frequency. If the clay particles flocculated quickly, 1 ml of a solution containing 1 N (NaPO₃)₆ and 0.12 N Na₂CO₃ was added to stabilize the suspension. The <2 μm fraction of all samples was separated by sedimentation and then, Javex, which contains NaOCl, was added to the clay suspension to destroy organic matter and to homoionically saturate the <2 μm fraction with sodium (Cassidy and Mankin, 1960). The latter was achieved by leaving the mixture overnight on a steam bath. The excess NaCl and NaOCl were then removed by rinsing with distilled water and centrifuging. Finally, the <2 μm fraction was further divided into two size fractions by centrifugation. The presence of discrete clay minerals, the crystallinity index and the sharpness ratio of illite were studied in the <2 μm fraction in which clay minerals are most concentrated. Illite polymorph ratios were determined using the 1-0.2 μm fraction in accordance with Velde and Hower's (1963) method; since interstratified clays are most abundant in the extremely fine sizes they were studied in great detail in the <0.08 μm fraction.

Analytical methods

1. *Identification of discrete layer silicates.* Discrete layer silicates were identified using an oriented, sodium-saturated <2 μm specimen following standard techniques of identification (Brown, 1961). CuK α radiation and a nickel filter were used; the scanning speed was $1^\circ(2\theta)\text{min}^{-1}\text{cm}^{-1}$, the time constant was 2 sec and scanning was done in the range 2-40°(2 θ).

2. *Sharpness ratio and crystallinity index measurements of illites.* Sharpness ratio and crystallinity index of illites were measured on an oriented, potassium-saturated <2 μm specimen at 50 per cent r.h. with CoK α radiation, iron filter scanning speed of $1^\circ(2\theta)\text{min}^{-1}\text{cm}^{-1}$, time constant of 2 sec and range of 2-15°(2 θ). The sharpness ratio (S.R.), which is the ratio of the height of the illite 001 peak at 10.0 Å to the height of the base line at 10.5 Å, was determined using Weaver's (1961a) method. The crystallinity index (C.I.), which is the width of the illite 001 peak at half height expressed in mm, was obtained following Kubler's (1966) method.

Table 1. Maximum burial depth of samples 1063, 999, 823, 278, 201, 109 and 437

Sample No.	Maximum burial depth of analysed samples	
	(ft)	(m)
1063	5900	1967
999	6650	2217
823	7300	2433
278	8000	2667
201	8650	2883
109	9350	3117
437	10,100	3367

3. *Illite polymorphism.* The per cent of illite which was the 2M polymorph was determined using the potassium-saturated 1–0.2 μm size fraction following Velde and Hower's (1963) method.

4. *Identification of interstratified layer silicates.* Studies of interstratified layer silicates were done using the <0.08 μm fraction. This fraction was saturated with Ca by treatment with 1 N CaCl_2 . After the removal of the excess salt by washing and centrifuging, the samples were recovered by freeze-drying (Brydon, Rice and Scott, 1963). These samples were used for: (a) X-ray diffraction analysis; (b) thermal analysis; (c) i.r. spectroscopy; (d) chemical analysis; (e) surface area and cation exchange capacity (CEC) measurements.

(a) *X-ray analysis.* X-ray analysis was done using oriented specimens made by drying 1 ml of a clay suspension containing 40 mg of sample on a 37.5 \times 25.0 mm glass slide. After drying, the sample was X-rayed using Fe filtered $\text{CoK}\alpha$ radiation ($\lambda = 1.7902 \text{ \AA}$), with settings of 45 kV–20 mA, scanning speed $1^\circ(2\theta)\text{min}^{-1} 2\text{cm}^{-1}$ and time constant of 2 sec. Clay minerals were identified on the basis of the criteria used by Kodama and Brydon (1968).

To obtain additional information on the nature of interstratified clays, a portion of the <0.08 μm size fraction was saturated with potassium in a 1 N KCl solution and X-rayed again.

(b) *Thermal analysis.* Differential thermal analysis (DTA) was carried out on calcium-saturated samples. Curves were recorded on an R.L. Stone DTA apparatus using the open cup system with 10 mg of sample. The cups were seated on a ring-type thermocouple with Pt–Pt 10 per cent r.h. junction below the sample holder. The apparatus was run with a constant heating rate of $10^\circ\text{C min}^{-1}$, from ambient temperature to 1000°C .

Thermogravimetric analysis (TGA) was done using calcium-saturated samples, R. L. Stone TGA-5b apparatus and 40 mg samples were used. The range was set to give 50 per cent full scale deflection and each sample was run in a narrow hang-down tube from ambient temperature to 1000°C at a rate of $10^\circ\text{C min}^{-1}$.

Differential thermogravimetric analysis (DTGA) curves were plotted manually using TGA data taken at intervals of 5°C .

TGA and DTGA were used as aids in the chemical analysis to determine the amount of kaolinite, hence the portion of SiO_2 , Al_2O_3 and H_2O due to kaolinite in the <0.08 μm fraction, Table 7.

(c) *Infrared absorption analysis.* Infrared absorption spectra of the layer silicates were obtained with a Beckman IR 12 i.r. spectrophotometer, in order to evaluate its contribution to the study of diagenesis. For

this, pellets containing 0.75 mg of calcium-saturated clays and 400 mg of KBr were used.

(d) *Chemical analysis.* Elemental analysis of air-dried samples was done by fusing with Li_2CO_3 and H_3BO_3 . Elemental concentrations were determined using the methods of Foscolos and Barefoot (1970). Free silica and aluminum were removed by digestion with 0.5 N NaOH (Jackson, 1965); free iron oxides were removed by treatment with a sodium dithionate–sodium citrate solution (Aguilera and Jackson, 1953).

(e) *Surface area and cation exchange capacity (CEC) measurements.* Surface area and CEC measurements were carried out on sodium-saturated clays, using the methylene blue absorption method as described by Thi Hang Pham and Brindley (1970).

RESULTS AND DISCUSSION

Discrete layer silicates, sharpness ratio, crystallinity index and illite (2M/2M + 1Md) ratio

The mineralogy of discrete layer silicates, the sharpness ratio and the crystallinity index of the less than 2 μm fraction were used to evaluate the extent of sediment diagenesis. In the 1–0.2 μm fraction, the per cent of illite which was the 2M polymorph type was used as an indicator of the diagenetic stage. The results are shown on Table 2.

Discrete illite is present throughout the formation whereas chlorite and kaolinite are absent in the lowermost part of the Buckinghorse Formation. Although the absence of chlorite is difficult to interpret, the absence of kaolinite may indicate a certain degree of diagenesis (Dunoyer de Seconzac, 1970).

Crystallinity index decreases and sharpness ratio increases with depth of burial. The same trend has been reported by Kubler (1966), Dunoyer de Seconzac (1970), Frey (1971) and van Moort (1971). The per cent of illite which is the 2M polymorph, also increases with burial depth. The latter is in accordance with the findings of Maxwell and Hower (1967) that increasing temperature and pressure conditions accompanying deep burial caused transformation of the 1Md illites to 2M illites in the Precambrian Belt.

Interstratified layer silicates

X-ray diffraction analysis. X-ray diffractograms were made using the <0.08 μm fraction of all seven samples in order to identify the type of interstratification. 001/001 basal reflections were used. A set of diffraction patterns for sample 278, a typical sample, is shown in Fig. 1. The patterns show that the first order basal reflection peak shifts from 10.2 \AA , at 0 per cent r.h. to 12 \AA , at 50 per cent r.h. and to 14 \AA , at 85 per cent r.h. At 85 per

Table 2. Discrete layer silicates of Na-saturated $<2 \mu\text{m}$ oriented clays, crystallinity index and sharpness ratio of K-saturated $<2 \mu\text{m}$ oriented specimen of illites and per cent 2M illite polymorphs of K-saturated clays between $1-0.2 \mu\text{m}$ size fraction of unoriented specimen in relation to maximum burial depth of analysed samples

Sample No.	Maximum burial depth of analysed samples (ft)	Maximum burial depth of analysed samples (m)	Discrete layer silicates	Crystallinity index	Sharpness ratio	Illite $[(2M)/(2M + 1Md)] \times 100$
1063	5900	1967	Illite-chlorite-kaolinite	26	1.2	20
999	6650	2217	Illite-chlorite-kaolinite	24	1.2	30
823	7300	2433	Illite-chlorite-kaolinite	26	1.2	30
278	8000	2667	Illite-chlorite	19	1.4	30
201	8650	2883	Illite-chlorite-kaolinite	20	1.5	40
109	9350	3117	Illite	15	1.8	50
437	10,100	3367	Illite	13	2.2	45

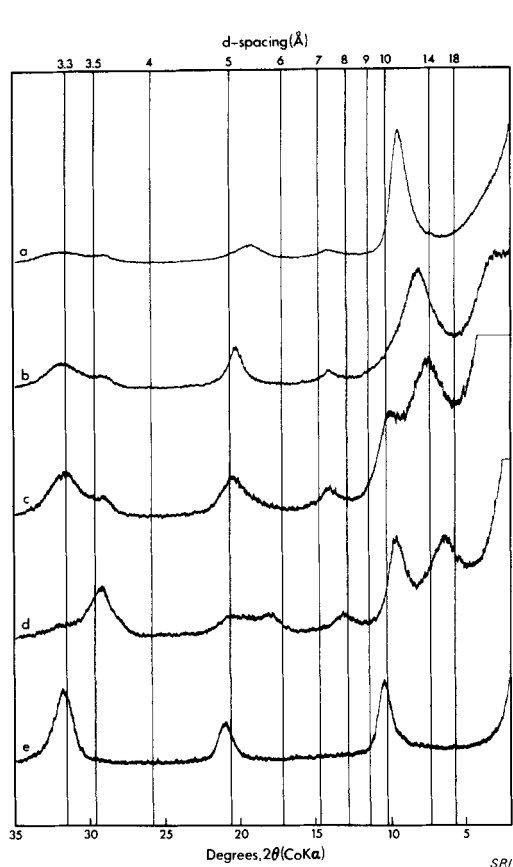


Fig. 1. X-ray diffraction patterns of oriented specimen of the $<0.08 \mu\text{m}$ fraction from sample 278 (a) Ca-saturated dry specimen under N_2 gas atmosphere; (b) Ca-saturated specimen at 50 per cent r.h.; (c) Ca^{2+} -saturated specimen at 85 per cent r.h.; (d) Ca-saturated glycerol treated specimen; (e) Ca-saturated specimen after heating at 550°C .

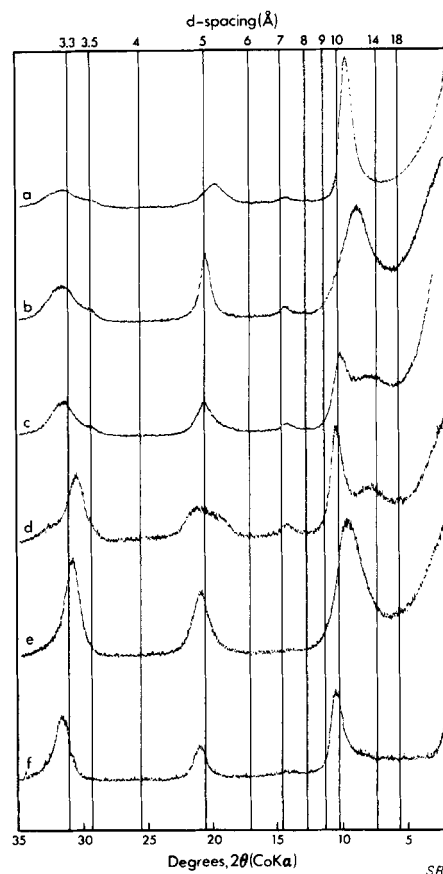


Fig. 2. X-ray diffraction pattern of oriented specimen of the $<0.08 \mu\text{m}$ fraction from sample 999 (a) Ca-saturated dry specimen under N_2 gas; (b) Ca-saturated specimen at 50 per cent r.h.; (c) Ca-saturated specimen at 85 per cent r.h.; (d) Ca-saturated glycerol treated specimen; (e) K-saturated glycerol treated specimen; (f) Ca-saturated specimen after heating at 550°C .

cent r.h., the peak splits into two parts. This splitting was more pronounced after saturation with glycerol in which case the basal spacing of 14 Å increased to 16 Å while the basal spacing of another peak remained unchanged at 10.2 Å. This indicated the presence of smectite. After heating at 550°C, the layers collapsed to 10 Å, indicating the absence of chlorite or chlorite-like compounds. Similar behavior was exhibited by all clay samples.

Upon K-saturation and glycerol saturation of sample 999 (Fig. 2), only limited expansion of basal spacing was observed. This indicates the presence of an expansible clay other than montmorillonite. This clay is considered to be a vermiculite-like layer silicate.

Since neither a 10 nor a 16 Å peak was observed at 50 per cent r.h. and since no 14 Å peak was observable after glycerol saturation, we conclude that the mica, montmorillonite and vermiculite of this sample exist as component layers in an interstratification and not as discrete minerals (Kodama and Brydon, 1968).

To study the influence of maximum burial depth of the samples prior to uplift on the relative proportions of the three components, the direct Fourier transform method (MacEwan, 1956) was used to interpret the X-ray data. This was done because basal peak positions could not be interpreted from the two-component interstratification (MacEwan, Ruiz and Brown, 1961; Cesari, Morelli and Favretto, 1965; Reynolds, 1967), nor from the three-component mica-montmorillonite-chlorite interstratification (Jonas and Brown, 1959; Cesari and Allegra, 1967). Fourier transform analysis was done for all seven samples using basal reflection data obtained from oriented specimens. The area under a peak was obtained by counting pulses. After correcting for the Lorentz-polarization factor, the integrated intensities were converted to Fourier coefficients using the conventional layer structure factors of Cole and Lanchucki (1966). The Fourier transforms were calculated by use of a computer and all calculations indicated three fundamental components with spacings at 10 (A), 13.7 (B) and 15.8 (C) Å. The findings support the argument that three components exist in the interstratified clays. Fourier transforms of samples 437, 278 and 999 are presented in Fig. 3. As an example, lists of assigned peaks with comparison between observed peak heights and calculated ones, for samples 278 and 999, are given in Table 3. For the same two samples the probability coefficients deduced from the Fourier transforms are presented on Table 4. The relative proportions of the three components of all samples (mica-montmorillonite-vermiculite) were obtained from the probability coefficients, and their relation to estimated maximum depth of burial is given in Table 5. Sample 437, which has the greatest esti-

mated maximum depth of burial also has the highest proportion of illite, 82 per cent, while the upper part of the outcrop, sample 1063, contains only 47 per cent illite. The same general trend is seen in Fig. 4, which shows the X-ray patterns of all oriented Ca-saturated specimens of the <0.08 µm fraction at 50 per cent r.h. The d_{001} basal reflection shifts gradually with depth from 13.5 Å, for the uppermost sample 1063, to 10.2 Å, for the lower-most sample 437.

Thermal analyses and infrared absorption spectra. Thermal analyses were made and infrared absorption spectra were recorded in order to identify discrete expandable clay minerals in the presence of interstratified clays. Differential thermal analysis (DTA) curves (Fig. 5) showed that smectite and/or vermiculite did not exist as discrete expandable clay minerals since the characteristic endothermic peak of smectites around

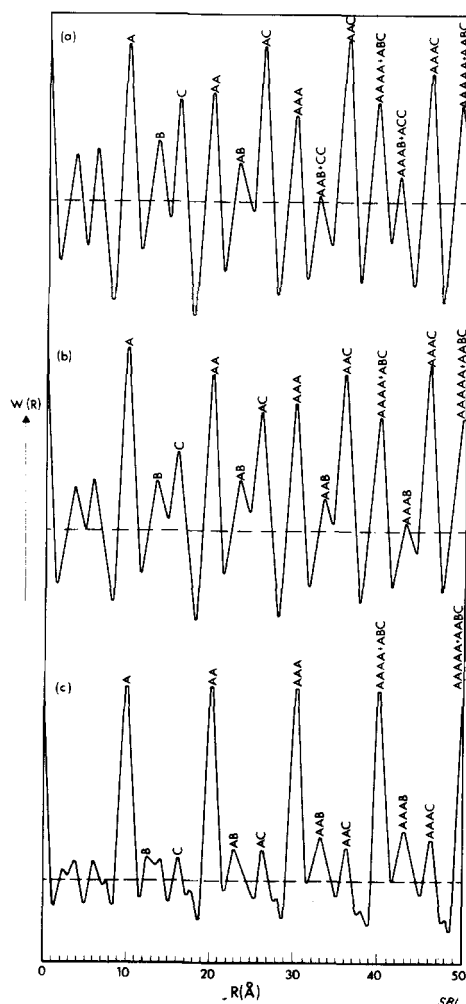


Fig. 3. Fourier transforms of basal reflections of Ca^{2+} -saturated <0.08 µm fraction at 50 per cent r.h. from (a) sample 999; (b) sample 278; (c) sample 437.

Table 3. Comparison of observed peak heights of Fourier transform with calculated peak heights on Ca-saturated samples 278 and 999

278				999			
Spacing (Å)	Peak	Observed height ratios	Calculated* height ratios	Spacing (Å)	Peak	Observed height ratios	Calculated* height ratios
10.0	A	0.59	0.59	9.9	A	0.49	0.49
13.5	B	0.16	0.16	13.4	B	0.19	0.19
15.9	C	0.25	0.25	16.0	C	0.32	0.32
19.9	AA	0.50	0.50	19.8	AA	0.34	0.34
23.3	AB	0.16	0.16	23.0	AB	0.12	0.12
25.8	AC	0.38	0.38	25.9	AC	0.49	0.49
29.9	AAA	0.41	0.42	29.7	AAA	0.27	0.23
33.1	AAB	0.11	0.14	32.4	CC	0.02	0.05
35.8	AAC	0.49	0.38	35.8	AAC	0.51	0.46
39.8	{ ABC AAAA	0.37	{ 0.02 0.35	39.5	{ ABC AAAA	0.31	{ 0.06 0.16
42.8	BBC	0.02	0.00	42.0	ACC	0.08	0.16
45.7	AAAC	0.54	0.39	45.7	AAAC	0.40	0.66
49.7	{ AABC AAAAA	0.36	{ 0.04 0.30	49.3	{ AABC AAAAA	0.29	{ 0.10 0.11

* From probability coefficients.

Table 4. Probability coefficients deduced from Fourier transforms of Ca-saturated samples 278 and 999

278	999
$P_A = 0.59$	$P_A = 0.49$
$P_B = 0.16$	$P_B = 0.19$
$P_C = 0.25$	$P_C = 0.32$
$P_{AA} = 0.84$	$P_{AA} = 0.68$
$P_{AB} = 0.005$	$P_{AB} = 0.00$
$P_{AC} = 0.150$	$P_{AC} = 0.32$
$P_{BA} = 1.00$	$P_{BA} = 1.00$
$P_{BB} = 0.00$	$P_{BB} = 0.00$
$P_{BC} = 0.00$	$P_{BC} = 0.00$
$P_{CA} = 1.00$	$P_{CA} = 0.95$
$P_{CB} = 0.00$	$P_{CB} = 0.00$
$P_{CC} = 0.00$	$P_{CC} = 0.05$

Table 5. Relation between probability coefficients of the three components, P_A = mica, P_B = vermiculite, P_C = montmorillonite, and maximum burial depth of Buckingham Formation

Sample	Maximum burial depth (ft)	Probability coefficients of the three components		
		P_A	P_B	P_C
1063	5900	0.47	0.19	0.34
999	6650	0.49	0.19	0.32
823	7300	0.58	0.13	0.29
278	8000	0.59	0.16	0.25
201	8650	0.55	0.18	0.27
109	9350	0.71	0.16	0.13
437	10,100	0.82	0.09	0.09

700°C was not found (Greene-Kelly, 1957). In addition, the characteristic endothermic peaks of vermiculite around 250 and 800°C as well as the exothermic peak around 825°C (Barshad, 1948) were not observed. The endothermic peak around 100°C is attributed to the loss of adsorbed H₂O whereas the endothermic peak at 540°C is in the range in which a peak may be found for 'normal' illite, 'abnormal' smectite or interstratified illite-smectite (Cole, 1955). The exothermic peak around 340°C is attributed to the combustion of the determined organic matter which remained after the removal of hydrocarbons from the clay surface. The endothermic peak around 910°C reflects the dehydroxylation of 2:1 layer silicates, mainly illite, and the exothermic peak around 975°C results from reactions involved in the breakdown of kaolinite. The DTA data confirmed the conclusion derived from X-ray diffractograms, that kaolinite is absent from the lower part of the formation, (samples 437 and 109) and present in the upper part (samples 201, 278, 823, 999 and 1063).

Thermogravimetric and differential thermogravimetric analyses (Fig. 6) were conducted to determine the amount of kaolinite and, hence, the portion of the SiO₂, Al₂O and hydroxyl H₂O of the <0.08 μm fraction attributable to kaolinite. This had to be subtracted from the chemical composition of the <0.08 μm fraction in order to obtain the chemical composition of the interstratified layer silicates. Thus, the 400°C point or the exact breaking point, Table 7, for the crystal lattice calculations was obtained from

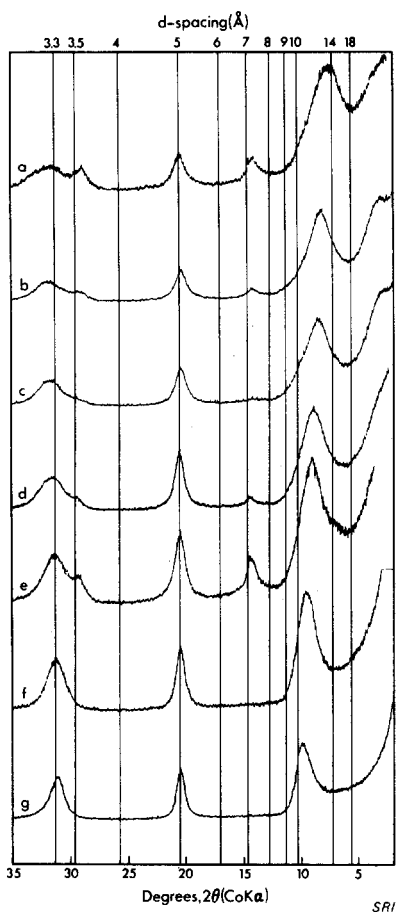


Fig. 4. X-ray diffraction patterns of Ca-saturated specimen of the $<0.08 \mu\text{m}$ fraction at 50 per cent r.h. (a) Sample 1063; (b) sample 999; (c) sample 823; (d) sample 278; (e) sample 201; (f) sample 109; (g) sample 437.

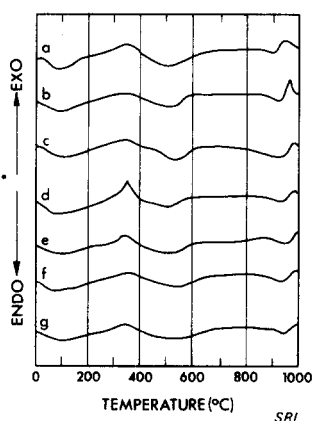


Fig. 5. Differential thermal analysis of Ca-saturated specimen of the $<0.08 \mu\text{m}$ fraction (a) sample 1063; (b) sample 999; (c) sample 823; (d) sample 278; (e) sample 201; (f) sample 109; (g) sample 437.

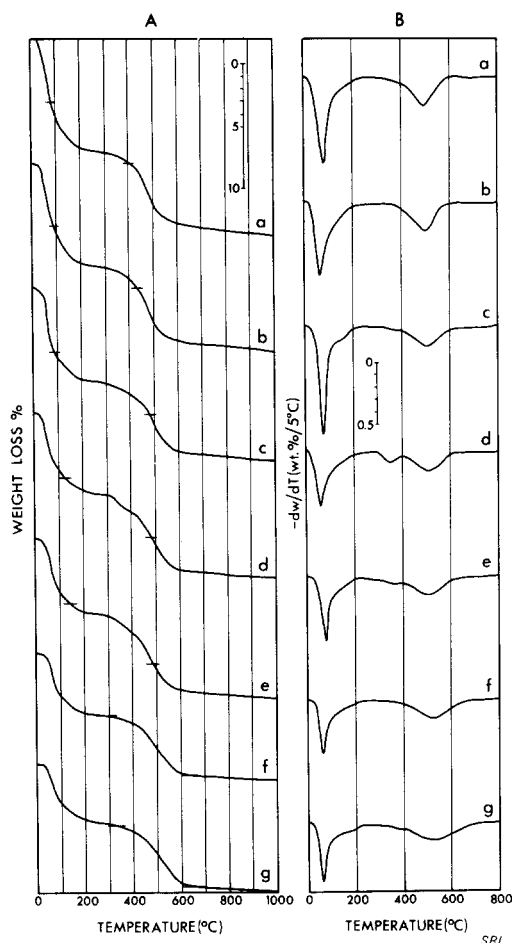


Fig. 6. Thermogravimetric (A) and differential thermogravimetric (B) curves of Ca-saturated specimen of the $<0.08 \mu\text{m}$ fraction (a) sample 1063; (b) sample 999; (c) sample 823; (d) sample 278; (e) sample 201; (f) sample 109; (g) sample 437.

DTGA. The concentration of kaolinite was obtained from TGA. Such calculations are based on the fact that kaolinite contains 16.2 per cent hydroxyl H_2O , while mixed layers contained 5.40 per cent hydroxyl H_2O as determined by ignition. The value of 5.40 per cent was obtained from the two bottom samples, 437 and 109, which were devoid of kaolinite and is in excess of the value of 4.52 per cent for muscovite and of 4.93 per cent for montmorillonite. We attribute this difference to the presence of vermiculite which contains cavity water at temperatures as high as 700°C (Barshad, 1965).

The identification of discrete clay minerals in the presence of interstratified clays using i.r. spectroscopy was difficult. However, the presence of kaolinite was ascertained in samples 201, 278, 823, 999 and 1063, since a shoulder-like peak around 3697 cm^{-1} was

Table 6. Elemental analysis of samples 437, 109, 201, 278, 823, 999 and 1063 after the removal of amorphous SiO₂, Al₂O₃ and free Fe₂O₃

Sample No.	Elemental composition											
	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	MnO	Na ₂ O	CaO	MgO	K ₂ O	H ₂ O	L.O.I.*	Total
1063	49.54	23.45	0.34	3.77	0.00	0.51	1.45	1.69	3.13	9.40	6.30	99.58
999	49.54	24.95	0.00	3.12	0.00	0.13	1.28	1.40	3.57	8.55	6.50	99.04
823	50.38	24.95	0.00	2.13	0.00	0.28	1.15	1.76	4.63	7.75	6.00	99.03
278	49.54	26.84	0.00	1.80	0.00	0.42	1.04	1.40	4.93	6.48	6.70	99.15
201	49.54	26.84	0.00	1.80	0.00	1.08	1.28	1.26	4.87	6.25	6.53	99.45
109	50.38	27.81	0.00	1.80	0.00	0.16	0.74	1.42	6.23	5.05	5.50	99.09
437	50.38	26.84	0.17	0.98	0.00	0.38	0.79	1.64	6.70	5.00	5.45	98.33

* Loss on ignition is considered from 300 to 1000°C.

observed (Kodama and Oinuma, 1963). This confirmed the results obtained by X-ray diffraction and thermal analyses.

Chemical analysis and calculation of structural formula. Chemical analyses were done in order to determine the chemical compositions of the mixed layer clay minerals. Changes in the mixed-layer clay compositions were used to quantify the changes in the relative proportions of swelling and non-swelling portion of the clays as a function of diagenetic alteration.

The chemical composition of the <0.08 µm fraction after the removal of amorphous SiO₂, Al₂O₃ and free Fe₂O₃ is given in Table 6. To obtain the chemical composition of the interstratified clay minerals, the contribution of SiO₂, Al₂O₃ and hydroxyl H₂O derived from the kaolinite content (Table 7) had to be subtracted from the overall chemical composition. The final chemical compositions (Table 8), were converted to a molar basis in order to obtain the overall structural formulae for the mixed layer minerals (Table 9), following Kodama and Brydon's approach. This table shows that aluminum content in the tetrahedral position increases with burial depth. Therefore, as diagenesis proceeds, increasing amounts of aluminum substitute for silicon in the tetrahedral layers. This is in accordance with the findings of Weaver, Beck and Pollard (1971).

In the octahedral position also, aluminum content increases with increasing depth of burial. Iron, and to a lesser extent magnesium, decrease with increasing burial.

Table 9 shows also that potassium content in the overall structural formulae increases with the depth of burial, which is in accordance with the results of Perry and Hower (1970) for Gulf Coast pelitic sediments. On the other hand, calcium decreases with burial depth.

The illite content of the mixed-layer silicates was obtained by the following method:

(1) It was assumed that illite does not have the ideal formula of muscovite (Weaver, 1965) and, therefore,

the number of Si and K ions in the formula were >3.00 and <1.00, respectively.

(2) Inequality equations derived from the Si:Al relation in the overall layer formula were solved. The results yield an Si:Al ratio of 3.35:0.65 for the illite formula, which was also similar to the ratio found by Weaver (1965).

Thus, to calculate the proportion of illite in the expandables the following equation was employed: $(3.35x) + 4.00(1 - x) = \text{No. of Si ion from the overall layer formula}$, where $x = \text{proportion of illite}$, $1 - x = \text{proportion of expandables}$, $3.35 = \text{Si ions in the illite formula}$ and $4.00 = \text{Si ions in the expandable formula}$. The calculations (Table 9) show that the proportion of illite in the interstratified clays follows the same trend as that obtained from X-ray work.

By averaging the illite composition throughout the formation, the formula obtained for illite is $\text{K}_{0.70}\text{Na}_{0.02}(\text{Al}_{2.00})[\text{Si}_{3.35}\text{Al}_{0.65}\text{O}_{10}](\text{OH})_2$. On the basis of this formula, which is very close to the one proposed by Weaver (1965), the calculated percentage of illite in the mixed layer is very close to the value obtained by X-rays using the Fourier coefficients (Table 10). The table shows also that, as the burial depth increases, the illitic composition of the interstratified clays increases.

Table 9 shows that the calculated (OH) content of the overall structure varied from 2.21 to 2.50 atoms per half unit cell. This was undoubtedly caused by errors in evaluating the structural water content from the thermogravimetric analysis on the basis of 400°C (Table 7). The (OH), in excess of 2.00 atoms per half-unit cell is considered to be cavity water associated with vermiculite. Vermiculite loses all its cavity water at a higher temperature (Barshad, 1965).

Surface area measurements and cation exchange capacity (CEC) values. Surface area measurements and CEC values were carried out in order to correlate these values with K₂O content and to evaluate their changes as a function of burial depth of the sediments. The data

Table 7. Determination of per cent kaolinite and per cent SiO_2 , Al_2O_3 and H_2O^+ of mixed layers after subtracting the contribution of kaolinite $[\text{Al}_4(\text{Si}_4\text{O}_{10})\text{OH}_8]$

Sample	Temperature at breaking point (°C)	A	B	B-A	C	C-5·40	Kaolinite (%)		Per cent SiO_2		Per cent Al_2O_3		Per cent H_2O^+			
							Total	Diff.	Total	Diff.	Total	Diff.	Total	Diff.		
1063	405	9·95	15·70	5·75	6·83	6·83	8·83	49·54	4·11	45·43	23·45	3·49	19·96	5·75	1·23	4·52
999	375	8·87	15·05	6·18	7·28	1·88	11·61	49·54	5·40	44·14	24·95	4·59	20·36	6·18	1·62	4·56
823	410	8·38	13·75	5·37	6·23	0·83	5·13	50·38	2·39	47·99	24·95	2·03	22·92	5·37	0·71	4·66
278	410	7·64	12·78	5·14	5·90	0·50	3·09	49·54	1·44	48·10	26·84	1·22	25·62	5·14	0·43	4·71
201	390	7·63	13·18	5·55	6·51	1·11	6·85	49·54	3·19	46·35	26·84	2·71	24·13	5·55	0·95	4·60
109	395	5·66	10·45	4·79	4·84 → 5·40	0·00	0·00	50·38	0·00	50·38	27·81	0·00	27·81	4·79	0·00	4·79
437	400	5·57	10·45	4·88	4·88	0·00	0·00	50·38	0·00	50·38	26·84	0·00	26·84	4·88	0·00	4·88

A = Weight loss up to breaking point on air dry basis.

B = Weight loss from 20–1000°C on air dry basis.

C = Crystal lattice water (H_2O^+) on ignited basis.

Per cent kaolinite = $(C-5·40) \times 100/16·2$.

Table 8. Elemental composition of mixed-layered clays of samples 437, 109, 201, 278, 823, 999 and 1063 at 400°C

Sample No.	Elemental composition g 100 g ⁻¹										
	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	MnO	Na ₂ O	CaO	MgO	K ₂ O	H ₂ O ⁺	Total
1063	56.24	24.70	0.42	4.67	0.00	0.63	1.79	2.09	3.87	5.59	100
999	56.19	25.92	0.00	3.97	0.00	0.17	1.63	1.78	4.54	5.80	100
823	56.12	26.80	0.00	2.49	0.00	0.33	1.34	2.06	5.41	5.45	100
278	54.74	28.50	0.00	2.13	0.00	0.50	1.23	1.65	5.82	5.43	100
201	54.21	28.88	0.00	2.03	0.00	1.22	1.44	1.42	5.49	5.31	100
109	53.98	29.77	0.00	1.94	0.00	0.17	0.79	1.53	6.68	5.14	100
437	54.31	28.94	0.18	1.06	0.00	0.41	0.85	1.77	7.22	5.26	100

Table 9. Summary of the chemical formulae for the seven interstratified clays

Sample	Overall layer structure	Nonswelling layer structure	Swelling layer structure
1063	K _{0.32} Na _{0.07} Ca _{0.12} (Al _{1.52} Mg _{0.20} Fe _{0.23} Ti _{0.02}) (Si _{3.64} Al _{0.36} O _{9.80})(OH) _{2.50}	0.55 K _{0.59} Na _{0.13} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.45 Ca _{0.27} {Mg _{0.45} Fe _{0.50} Al _{0.95} } [Si _{4.00} O _{9.56}](OH) _{2.91}
999	K _{0.37} Na _{0.02} Ca _{0.11} (Al _{1.60} Mg _{0.17} Fe _{0.19}) (Si _{3.63} Al _{0.37} O _{9.75})(OH) _{2.50}	0.57 K _{0.65} Na _{0.03} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.43 Ca _{0.26} {Mg _{0.41} Fe _{0.45} Al _{1.08} } [Si _{4.00} O _{9.42}](OH) _{3.16}
823	K _{0.44} Na _{0.04} Ca _{0.09} (Al _{1.65} Mg _{0.19} Fe _{0.12}) (Si _{3.62} Al _{0.38} O _{9.83})(OH) _{2.34}	0.58 K _{0.77} Na _{0.06} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.42 Ca _{0.22} {Mg _{0.47} Fe _{0.29} Al _{1.33} } [Si _{4.00} O _{9.58}](OH) _{2.82}
278	K _{0.48} Na _{0.06} Ca _{0.08} (Al _{1.71} Mg _{0.16} Fe _{0.10}) (Si _{3.54} Al _{0.46} O _{9.83})(OH) _{2.35}	0.71 K _{0.68} Na _{0.08} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.29 Ca _{0.29} {Mg _{0.55} Fe _{0.35} Al _{1.00} } [Si _{4.00} O _{9.41}](OH) _{3.16}
201	K _{0.45} Na _{0.14} Ca _{0.10} (Al _{1.71} Mg _{0.14} Fe _{0.10}) (Si _{3.51} Al _{0.49} O _{9.85})(OH) _{2.29}	0.75 K _{0.60} Na _{0.19} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.25 Ca _{0.37} {Mg _{0.55} Fe _{0.40} Al _{0.86} } [Si _{4.00} O _{9.40}](OH) _{3.16}
109	K _{0.55} Na _{0.02} Ca _{0.05} (Al _{1.76} Mg _{0.15} Fe _{0.09}) (Si _{3.49} Al _{0.51} O _{9.89})(OH) _{2.21}	0.78 K _{0.70} Na _{0.02} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.22 Ca _{0.25} {Mg _{0.67} Fe _{0.43} Al _{0.90} } [Si _{4.00} O _{9.55}](OH) _{2.95}
437	K _{0.60} Na _{0.05} Ca _{0.06} (Al _{1.73} Mg _{0.17} Fe _{0.05} Ti _{0.01}) (Si _{3.52} Al _{0.48} O _{9.86})(OH) _{2.27}	0.74 K _{0.80} Na _{0.06} {Al _{2.00} } [Si _{3.35} Al _{0.65} O ₁₀](OH) ₂	0.26 Ca _{0.23} {Mg _{0.66} Fe _{0.20} Al _{0.95} } [Si _{4.00} O _{9.24}](OH) _{2.50}

Table 10. Per cent illite determined by chemical analysis and Fourier transform method

Sample	Maximum burial depth (ft)	Chemical analysis	Fourier coefficient
1063	5900	46	47
999	6650	53	49
823	7300	63	58
278	8000	68	59
201	8650	64	55
109	9350	78	71
437	10,100	85	82

Table 11. Surface area measurements, CEC values and K₂O content of mixed layers as related to maximum burial depth of the Buckingham Formation

Sample	Maximum burial depth (ft)	Surface area (m ² /g)	CEC in m-equiv/110 g	Per cent K ₂ O
1063	5900	261	36.8	3.13
999	6650	233	29.8	3.57
823	7300	204	27.7	4.63
278	8000	183	45.4	4.93
201	8650	183	24.7	4.87
109	9350	133	19.1	6.23
437	10,000	120	17.4	6.70

listed in Table 11 and shown graphically in Fig. 7, show that surface area of CEC decreased, while K_2O content increased with burial depth.

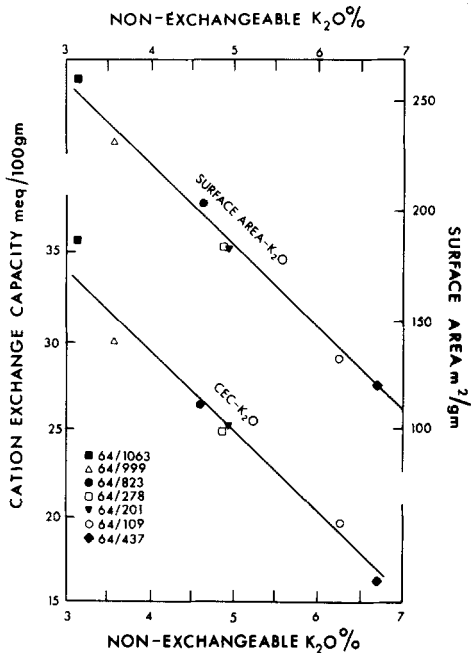


Fig. 7. Relationship between surface area, cation exchange capacity and non-exchangeable K_2O .

Diagenesis of the Buckingham Formation and evaluation of its potential for hydrocarbon production prior to uplifting

To evaluate the degree of diagenesis of the Buckingham outcrop formation and its potential as a hydrocarbon source at the time of maximum burial, parameters which have been derived from the study of clays in this work were combined with the data and information of Kubler, Dunoyer de Seconzac, Burst, Weaver, Perry and Hower, Muffler and White, Long and Neglia and van Moort. Diagenetic stages were defined to best fit the data of the previously-mentioned authors. Results are presented in Table 12 and are summarized as follows:

1. Discrete expandable clays do not occur below 4,500 ft unless a special geochemical condition or hydrothermal phenomenon occurs. Therefore, as a rule, their presence is confined to those rocks which belong to the early diagenetic stage.

2. In general, kaolinite disappears below 9000 ft.

3. Illite—2:1 expandable clays generally occur between 3000 and 12,000 ft. The maximum depth at which they are found is around 15,000 ft (Burst, 1969).

4. In general, 2M illite polymorphs predominate over the 1M + 1Md illite polymorphs in late diagenesis.

The boundary between late and middle diagenesis are defined when:

- (a) the illite component in the interstratified clays is 80 per cent,
- (b) 50 per cent of the illite is the 2M polymorph;
- (c) crystallinity index is ≤ 15 mm;
- (d) sharpness ratio is ≥ 2.0 ;
- (e) kaolinite just disappears from the clay mineral facies.

The division between early and middle diagenesis cannot be sharply drawn but has been defined as the point at which:

- (a) discrete expandable layer silicates and kaolinite are present;
- (b) 1M + 1Md illite polymorphs comprise 75 per cent of the illite or more;
- (c) illite contribution in the interstratified clays equals 25 per cent; or less;
- (d) crystallinity index is ≥ 20 mm;
- (e) sharpness ratio is ≤ 1.5 .

On the basis of this scheme (Table 12), the upper 3250 ft of the Buckingham Formation are classified as having undergone middle diagenesis and the lower 1000 ft as having undergone late diagenesis.

In order to assess the hydrocarbon potential of the Buckingham Formation prior to uplift, the results from the mixed layers also were related to Burst's (1969) work in which it is shown that the maximum dehydration zone of a shaly sediment takes place between 202 and 275°F (Fig. 8). Within this temperature window of 73°F, there is a gradual increase in the amount of illite in the mixed layers from 30 to 70 per cent. Below 30 or above 70 per cent of illite concentrations in the mixed layer phase, there is no correlation between temperature, dehydration of mixed layers and transformation of illite to montmorillonite in the interstratified clays. In the Buckingham Formation, the break-over point, that is the concentration of 70 per cent illite in the mixed layer which corresponds to 275°F, occurs around a burial depth of 9300 ft. Since an average geothermal gradient can be defined as the temperature at a given point in the stratigraphic column, minus the surface temperature, divided by the thickness of the formation and, since from paleoecological and paleobotanical studies the surface temperature at the point of maximum depth of burial was around 75°F (Chamney, 1973), then the paleogeothermal gradient for the formation is: paleoearth. grad. = $(275-75^\circ\text{F})/(9300 \text{ ft}) \approx 2.1^\circ\text{F}/100 \text{ ft}$ or $1.2^\circ\text{C}/33.33 \text{ m}$.

Table 12. Correlation between burial depth and crystallinity index, sharpness ratio, per cent 2M illite polymorphs, illite in the illite-2:1 expandable clay minerals, presence or absence of discrete layer silicates, diagenetic stage, geothermal gradient and occurrence of hydrocarbons for assessing the degree of diagenesis and hydrocarbon generating potential of Buckingham Formation prior to uplift

Diagenetic stage	Occurrence of hydrocarbons	Crystallinity index (mm)	Sharpness ratio	Per cent 2M illite polymorphs	Layer lattice silicates			Burial depth in (ft) prior to uplift	Temp. grad.* of 2.1°F/100' (1.2°C/33.33 m)
					Per cent illite in illite-2:1 expandables	Expandables	Discrete layers Kaolinite		
Early		>20	<1.5	<25	<25	Present	Present	<4.5 × 10 ³	170°F (77°C)
Middle	Hydrocarbons on clay surface	19		35	40-50	Usually present	Present	6.0 × 10 ³	201°F (94°C)
	Flushing of liquid hydrocarbons by clay dehydration	17	1.9	50	70-80	Usually absent	Usually present	10 × 10 ³	285°F (141°C)
Late		16				Absent	Usually absent or transformed to dickite		
		15	2.3	65	80-90	Randomly interstratified	are absent	12 × 10 ³	327°F? (164°C)?
	Non-liquid hydrocarbons	14				Regular interstratified	are present		
		13							
		12.8							

* Surface temperature at 75°F. Example $(4.5 \times 10^3 \text{ ft} \times 2.1^\circ\text{F}) / (100 \text{ ft}) + 75 = 170^\circ\text{F}$.

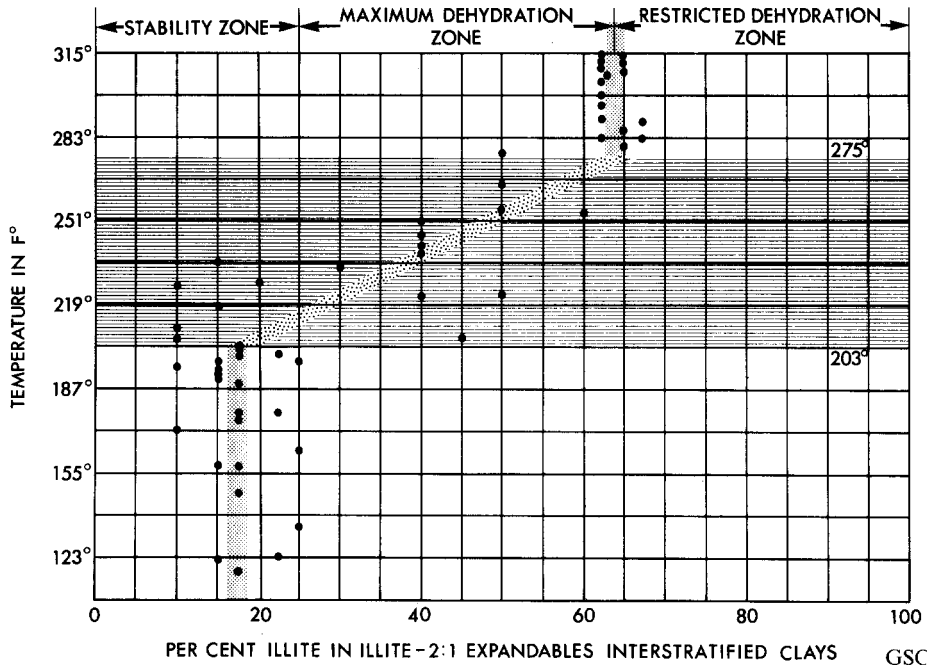


Fig. 8. Correlation between illite in the mixed layers, temperatures in F° and dehydration zones (modified after Burst, 1969).

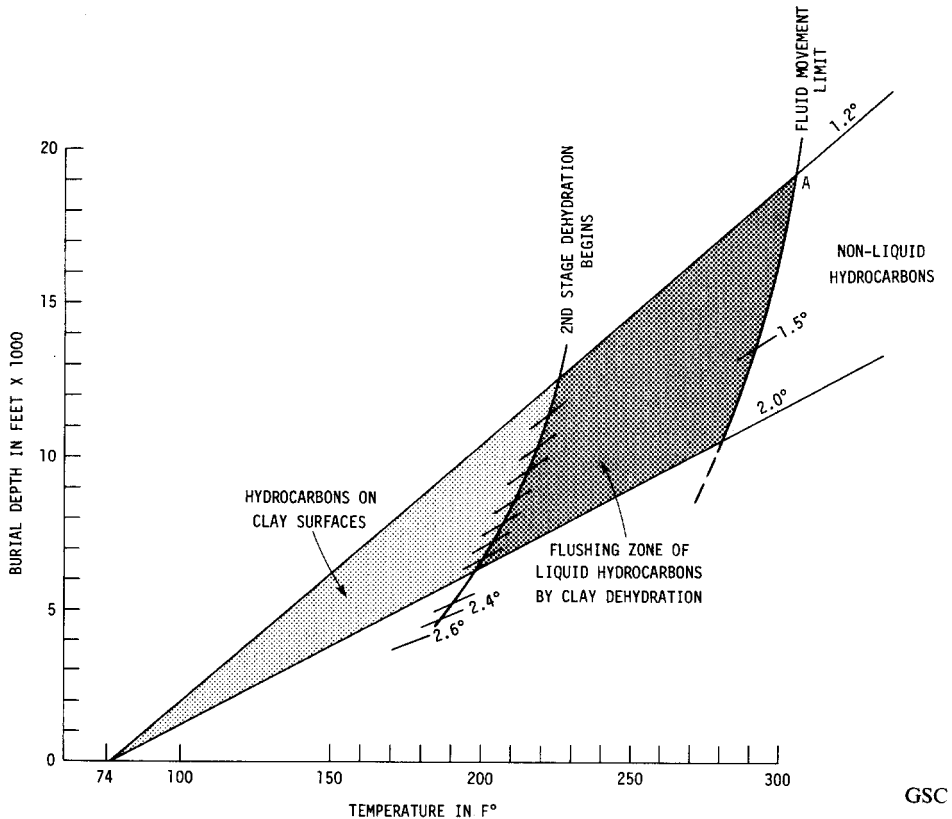


Fig. 9. Correlation between burial depth geothermal gradients in F°/100 ft and occurrence of hydrocarbons (modified after Burst, 1969).

The data presented above permit a rough assessment of the hydrocarbon generating potential of the formation prior to its uplift. Using Burst's (1969) fluid distribution model (Fig. 9), the obtained paleogeothermal gradient, the maximum burial depth of samples, the per cent illite component in the interstratified clays and the dehydration zones, it is concluded that prior to uplift, primary hydrocarbon migration should have been occurring in the upper 3250 ft of the Buckinghorse shales, and accomplished in the lower 1000 ft of the formation.

CONCLUSIONS

Detailed research on clay minerals was carried out on the $<2\ \mu\text{m}$ fraction of seven selected shale samples from the Buckinghorse Formation in order to assess its degree of diagenesis and oil-generating potential prior to uplifting. For this:

1. A classification scheme is proposed to evaluate the diagenetic stage of the source rock based on the combined criteria which have been derived from the study of layer silicates. These are: (a) presence or absence of certain discrete clay minerals. Montmorillonite is usually absent from middle diagenetic stages whereas kaolinite is usually absent from the late stage of diagenesis; (b) crystallinity index of illite. Crystallinity index of illite increases with burial depth; (c) sharpness ratio of illite. Sharpness ratio of illite increases with burial depth; (d) per cent 2M illite polymorph. As the degree of diagenesis increases, the per cent 2M illite polymorph increases; (e) per cent illite in the illite—2:1 expandables mixed layers. Illitization of expandable layer silicates in the interstratified clays increases up to a total of 80 per cent as diagenesis proceeds; (f) K_2O content of interstratified clays. Potassium content of mixed layers increases as the burial depth increases; (g) surface area and cation exchange capacity of mixed layers decrease as the burial depth increase.

2. Using Burst's (1969) data, a correlation between temperature and proportion of illite in the illite—2:1 expandables mixed layers is obtained. On the basis of this correlation and the maximum burial depth of the sample which contains 70 per cent illite in the mixed layers, a paleogeothermal gradient is calculated. The latter, in combination with the maximum burial depth and the proposed fluid distribution model by Burst (1969), is used to speculate the hydrocarbon generating potential of the formation prior to uplift.

Based upon the scheme of classifying the degree of diagenesis and the proposed fluid distribution model, the upper 3250 ft of the formation are classified in the late stage of middle diagenesis and liquid hydrocarbon should have been flushed out from the shales, while the

lower 1000 ft are classified at the beginning of late diagenesis and hydrocarbon migration should have been accomplished prior to uplift.

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Résumé—Des minéraux argileux provenant d'affleurements de schistes de la Formation Buckinghorse du Crétacé inférieur (4250 pieds d'épaisseur) ont été étudiés en vue de déterminer leur degré de diagenèse et leur potentiel de réservoir de pétrole. L'indice de cristallinité, le rapport de finesse des pics, le pourcentage d'illite qui est le polymorphe 2M et la présence de minéraux discrets ont été étudiés sur la fraction argileuse totale, tandis que la fraction argileuse très fine a été étudiée par la diffraction X, les analyses thermique différentielle, thermogravimétrique, thermogravimétrique dérivée, la spectroscopie infrarouge, la mesure de surface spécifique et les analyses chimiques. A l'aide des informations tirées de ces études et de résultats déjà publiés, un schéma de classification a été établi qui relie les variations de la minéralogie d l'argile aux étapes de la diagenèse et à la profondeur d'enfouissement.

Les résultats obtenus avec la fraction $< 2 \mu\text{m}$ montrent que l'indice de cristallinité diminue tandis que le rapport de finesse des pics et le pourcentage d'illite qui est le polymorphe 2M augmentent avec la profondeur d'enfouissement. Les résultats obtenus avec la fraction $< 0,08 \mu\text{m}$ relèvent l'existence d'un minéral argileux interstratifié à trois composants. En plus, les calculs de transformées de Fourier et les analyses chimiques et physicochimiques indiquent que le rapport des teneurs en argiles non hydratées (illite) à celles des argiles hydratées, et que la teneur en K_2O des argiles augmentent simultanément avec la profondeur; la capacité d'échange de cations et la surface spécifique diminuent avec la profondeur.

Si l'on se fonde sur un schéma de classification qui a été établi en combinant les critères et les résultats tirés des travaux de Weaver (1961a), Kubler (1966), Burst (1969) et Dunoyer de Segonzac (1970), les zones supérieures et moyennes de la formation (3250 pieds d'épaisseur) coïncident avec l'étape moyenne de la diagenèse, tandis que la zone inférieure (1000 pieds) est attribuée au début de la dernière diagenèse. Selon les termes du travail de Burst (1969), les 3250 pieds supérieurs sont une transition entre les zones de stabilité et de déshydratation, indiquant que, avant la remontée, les hydrocarbures peuvent avoir participé au processus de migration. Les 1000 pieds inférieurs de la formation sont dans une zone de déshydratation restreinte, indiquant que la migration des hydrocarbures doit avoir été achevé.

Kurzreferat—Tonminerale aus Schichtköpfen von Schiefern der Buckinghorse Formation der Unterkreide (4250 Fuß mächtig) wurden untersucht, um das Ausmaß der Diagenese und ihre Fähigkeit zur Ölbildung abzuschätzen. Der Kristallinitätsindex, das Schärfeverhältnis, der Prozentgehalt des 2M Polymorphs am Illitanteil und das Vorkommen besonderer Minerale wurden in der gesamten Tonfraktion bestimmt, während die feinste Tonfraktion durch Röntgenbeugung, differentialthermoanalytisch, thermogravimetrisch, differentialthermogravimetrisch, infrarotspektroskopisch und durch Bestimmung der spezifischen Oberfläche und der chemischen Zusammensetzung untersucht wurden. Mit Hilfe der aus diesen Untersuchungen erhaltenen Informationen und veröffentlichter Ergebnisse wurde ein Klassifikationsschema entwickelt, das tonmineralogische Veränderungen zum diagenetischen Umwandlungsgrad und der Lagerungstiefe in Beziehung setzt.

Die Werte der Korngrößenfraktion $< 2 \mu\text{m}$ zeigen, daß der Kristallinitätsindex mit zunehmender Lagerungstiefe abnimmt, während das Schärfeverhältnis und der als 2M Polymorph vorliegende Illitanteil ansteigen. Die mit der Fraktion $< 0,08 \mu\text{m}$ erhaltenen Ergebnisse lassen erkennen, daß ein aus 3 Komponenten bestehendes Wechsellagerungstonmineral vorliegt. Darüberhinaus ergeben Fourier-Analysen sowie chemische und physikochemische Analysen, daß sowohl das Verhältnis des Anteils nichthydratisierter Tone (Illite) zu dem hydratisierter Tone als auch der K_2O -Gehalt der Tone mit zunehmender Lagerungstiefe ansteigen. Kationenaustauschkapazität und spezifische Oberfläche nehmen mit der Lagerungstiefe ab.

Auf der Grundlage eines Klassifikationsschemas, das durch Kombination von Kriterien und Werten aus den Untersuchungen von Weaver (1961a), Kubler (1966), Burst (1969) und Dunoyer de Segonzac (1970) aufgestellt wurde, fallen die oberen und mittleren Teile der Formation (die oberen 3250 Fuß) in den mittleren Bereich der Diagenese, während der untere Teil (1000 Fuß) dem Beginn einer späten Phase der Diagenese zuzuordnen ist. Nach den Begriffen der Arbeit von Burst (1969) stellen die oberen 3250 Fuß einen Übergang zwischen den Stabilitäts- und Dehydratationszonen dar. Dies deutet an, daß vor Eintreten der Hebung die Kohlenwasserstoffe im Wanderungsprozeß begriffen waren. Die unteren 1000 Fuß der Formation sind der Zone beschränkter Dehydratation zuzuordnen, was zeigt, daß die Kohlenwasserstoffwanderung abgeschlossen sein sollte.

Резюме — Исследовали глинистые минералы обнаженных пород глинистого сланца нижнего мелового периода Букингхорса (толщиной 4250 фут), чтобы определить степень их диагенеза и их потенциал генерации нефти. На целой фракции глины изучали показатель степени кристаллизации, остроугольность, процентное содержание иллита, являющимся полиморфом 2М и присутствие разрозненных частиц минералов, а очень малую фракцию глины подвергли рентгенографическому структурному, дифференциально-термическому, термогравиметрическому, дифференциально-термогравиметрическому, ИК-спектроскопическому анализам и также анализу поверхностной площади. Посредством информации полученной этими исследованиями и из опубликованных данных, разработали схему классификации, относящую разнобразия минералогии глины к диагенетическим периодам и к глубине залегания.

По данным о фракции 2 $\mu\text{м}$ видно, что в зависимости от глубины залегания степень кристаллизации уменьшается, в то время как остроугольность и процентное содержание иллита, являющимся полиморфом 2М, повышаются. Результаты исследования фракции $<0,08 \mu\text{м}$ показали, что существует переслаивающийся трехсоставной глинистый минерал. Кроме того, преобразование Фурье и химический и физико-химический анализы указали, что как соотношение негидратировавшейся глины (иллит) к гидратировавшейся глине, так и содержание K_2O в глине повышаются чем глубже залегает минерал, а катионообменная способность и площадь поверхности при этом понижаются.

На основании схемы классификации, выработанной комбинацией критерий и данных взятых из исследований Вивера (1961а); Кублера (1966); Бурста (1969) и Дунойра де Секонзака (1970), верхняя и средняя части формации (верхняя 3250 фут) приходятся на среднюю стадию преобразования осадков в горные породы, в то время как нижняя часть (1000 фут) относится к началу позднего диагенеза. По работе Бурста (1969), верхние 3250 фут находятся в переходной стадии между зонами устойчивости и дегидратации указывающими, что до взброса, углеводороды, вероятно, подвергались перемещению.