A STUDY OF THE CLAYS FROM UPPER CRETACEOUS BENTONITES AND SHALES IN ALBERTA

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Abstract

Clay minerals of four bentonites and two shales from Upper Cretaceous continental sedimentary rocks in western Alberta were studied in detail using x-ray diffraction and fluorescence methods. The study of the bentonites was carried out in order to appraise their usefulness in correlation. The shales were investigated to help evaluate the influence of depositional environment on clay composition.

The bentonites range in composition from 19 to about 90 per cent mixed layer clay, 3 to 14 per cent kaolinite, no illite or chlorite and from none to almost 80 per cent montmorillonite. The shales lack the mixed layer clay, contain from about 25 to about 50 per cent kaolinite, 40 to 75 per cent montmorillonite, small amounts of illite, and one sample contains some chlorite.

Fourier transforms of the mixed-layer clays show three component interstratifications which in three of the bentonites are within the compositional limits of 50 to 62 per cent illite, 14 to 35 per cent vermiculite and 15 to 30 per cent montmorillonite. The mixedlayer clay from the fourth bentonite contains only 20 per cent illite, 18 per cent vermiculite and 62 per cent montmorillonite which is partly present as a mechanical mixture. Calculations and combination transform peaks suggest a tendency toward a long range ordering of the components.

X-ray spectrochemical analysis proves to be promising as a rapid, non-destructive method of determining the major rock forming elements, except Na⁺ and Mg⁺, in complex clay systems. At present accuracy of this method limits it to a semi-quantitative analysis. The major chemical components of the four bentonites are quantitatively very similar. The shales contain more Fe, Mn, and Ti, and less K and Al than the bentonites.

It is concluded that mineralogically the clays may be of use in correlating these bentonites, whereas the chemical compositions are too similar to be used in differentiating them. The shales are mineralogically and chemically different from the bentonites, but similar to one another. Assuming that the shales and bentonites were deposited in similar continental environments, such an environment of deposition only slightly modifies the composition of clay minerals.

INTRODUCTION

Thick deposits of Upper Cretaceous strata in the Alberta Foothills are largely continental and difficult to correlate. During the deposition of sandstones and shales, volcanoes in the Cordillera spread layers of ash, now altered to bentonite beds, over this region. Any original chemical or mineralogical differences in the pyroclastic material may be

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preserved to some degree in the bentonites and thus aid in characterizing individual horizons for correlation purposes. The present study is primarily concerned with the clay mineralogy of four bentonites and two shales at different stratigraphic levels in one section of the Belly River Formation in the Foothills. The study was undertaken as a preliminary investigation into the problem of whether or not the clay mineral composition would aid in characterizing the bentonites.

GEOLOGIC SETTING

The bentonites and shales used in the work were from one section of the Upper Cretaceous Belly River Formation on the Bow River west of Calgary, (Fig. 1). The Belly River Formation is one of several continental tongues in the Upper Cretaceous rock of this area which pinch out and are replaced to the east by marine shales. The stratigraphic section pertinent to this study is shown in Fig. 2. The sandstones and shales of this formation were derived from the south and west and the bentonites from the then active orogenic belt to the west. (Lerbekmo, 1963).

Oriented clay samples were examined with a Norelco diffractometer and non-oriented samples with a 114.6 mm. camera using Ni-filtered CuK α , radiation ($\lambda = 1.5405$ Å). Each of the oriented samples was *x*-rayed in its untreated condition, after glycolating using the method of Brunton (1955), and after heat treatment at 475° C. for 12 hours. The unoriented samples were *x*-rayed only in the untreated state. Exposed time was 3 hours with the spindle continuously rotating.

Qualitative analysis

Standard procedures for the identification of the basal reflections from the x-ray diffraction patterns (Fig. 3) were followed. The results thus obtained are given in Table 1.

Powder camera patterns for all the bentonites show (060) to be 1.50 Å with estimated relative intensity of 80-90. According to MacEwan (1950) this value suggests that the minerals reported from the bentonites are all dioctahedral.

Semi-quantitative analysis

In determining the proportions of the various clay minerals, a method following Johns, Grim & Bradley (1954), Weaver (1958) and Byrne & Farvolden (1959) was employed. This method is based on earlier work by Bradley (1953), who experimentally determined relative peak intensities for certain montmorillonite-organic liquid complexes.



FIGURE 1.



FIG. 2. Stratigraphic section along the Bow River.



FIG. 3. X-ray diffraction patterns from two orientated clays. (a) Untreated (b) Glycolated (c) Heat Treated

			Cla	ay Mineral		
Sampl	e	Mixed Layer	Montmoril- lonite	Illite	Chlorite	Kaolinite
Bentonites	4156	×	_	_	-	×
	4154	×	×	_	_	×
	4138	×	X		—	×
	4132	×	×	—	_	×
Shales	4134		×	×	_	×
	4133	_	×	×	×	×

TABLE 1. OCCURRENCE OF CLAY MINERALS IN THE BELLY RIVER BENTONITES AND SHALES

 \times denotes occurrence of clay mineral.

- denotes absence of clay mineral.

Based on his work various authors have produced correction factors which can be applied to diffraction peak intensities so that they will attempt to represent the amount of each constituent present. Table 2 shows the intensity correction values used in this study.

Mineral	Peak	Correction Factor
	17 Å	× 1.0
	15	\times 1.3
	13.4	\times 1.5
	13	\times 1.8
"10Å" ^{1,2}	12.5	\times 1.9
	11.6	\times 2.4
	10	\times 4.0
	9.6	\times 4.7
	9.3	\times 5.7
	9.1	× 6.3
Chlorite ³	7	× 1.6
Kaolinite ⁸	7	\times 1.6

TABLE 2. PEAK INTENSITY CORRECTION VALUES

 $1''10\text{\AA}'' = \overline{\text{Montmorillonite, illite and mixed layer clays.}}$

2"10Å" values from Bradley (1953).

"Chlorite and kaolinite values were determined from Byrne & Farvolden (1959) and Weaver (1958).

Semi-quantitative estimates were made using as many of the peaks as possible from the glycolated and natural samples. The results show a range of values. Johns, Grim & Bradley (1954) and Byrne & Farvolden (1959) conclude that this method is accurate to within about five per cent. In the light of the results obtained here, it appears that the error may be considerably greater. The results given in Table 3 were taken from calculations using peaks with d(001) between 7 and 17 Å from the glycolated samples. Values obtained from peaks in this region were fairly consistent for the various runs. Calculations in the region of d(001) = 3.3 to 3.5 Å gave variable results, with kaolinite usually being greater than reported here. This is probably due to the diffraction affects of well-crystallized as opposed to poorly-crystallized material being more apparent at higher angles. The results for untreated samples vary, possibly because of irregular and inconsistent water content in the interlayers of the expanding lattice.

			% Clay			
Sample	e	Mixed Layer	Montmorillonite	Illite	Kaolinite	Chlorite
Bentonite	e 4156	86-87			13-14	
	4154	49 - 52	44-46		4-5	
	4138	19 - 33	64-78		3	
	4132	93-	-97	.	3–7	—
Shale	4134		50–57	- Trace	43–50	_
	4133	—	43	2–73	27-	-58

TABLE 3. SEMI-QUANTITATIVE CLAY DETERMINATIONS

Mixed layer analysis

The simplest method of mixed layer analysis consists of comparing *x*-ray diffraction patterns with theoretically calculated patterns, a number of which are presented by Brown & MacEwan (1950) and Brown (1961). This trial and error method has certain limitations; the number of situations for which calculated patterns are available are few; proportions and components may be estimated in this way, but manner of ordering cannot be. A more direct approach, and the one used in this study, is the Fourier transform method described by MacEwan, Amil & Brown in Brown (1961).

The Fourier transforms calculated in this work are shown in Figs. 4 and 5. On the transforms the components have been labelled A, B, C, A^2 , B^3 etc., where $A^2 = AA$ and $B^3 = BBB$. The peak heights were calculated so that the sum of the components present equals 1.

The components present, which are the same for each sample may be readily identified with the aid of the x-ray diffraction patterns and the two Fourier transforms calculated for each clay. The first characteristic observed is that the mixed layer x-ray diffraction peaks move on glycolation, indicating the presence of an expandable mineral. Secondly, on heating to 475° C. the peaks collapse to form an integral series of



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001 peaks with d(001) = 10 Å indicating the components are basically 10 Å mica-like layers. Examining the transforms of the untreated samples, two components are seen to be present, one with a spacing of 10 Å, the other with a spacing of 12.5 to 13 Å. Comparing the transforms of the untreated samples to those for the glycolated material, the 10 Å component does not move, and is thus identified as illite (A). The 12.5-13 Å component splits into two peaks, indicating that it consists of two components. The one that expands to 13.5 Å has been tentatively called vermiculite (B), and the one that expands to 16.5-17 Å is identified as montmorillonite (C).

The quantities of each component can be directly determined from the transforms by multiplying the peak height (or probabilities) by 100. The composition of the mixed layer clays determined from Figs. 4 and 5 are given in Table 4. The transform for the untreated 4132 sample is difficult to interpret, possibly because of the lack of adequate diffraction peaks in its calculation.

Sample	Treatment	% Illite	% Vermiculite	% Montmorillonite
A1 20	Glycolated	55	16	29
4100	Untreated	57		}
41 84	Glycolated	59	14	27
4194	Untreated	62		3
1190	Glycolated	50	35	15
4190	Untreated	53	47	7
4199	Glycolated	20	18	62
4102	Untreated	—		

TABLE 4. COMPOSITION OF MIXED LAYER CLAYS

The interstratification of the Belly River bentonite mixed-layer clays have been determined by the method outlined in Brown (1961). A series of peak heights for specific situations were calculated and matched to the observed peak heights, and Table 5 shows the results of such a calculation for sample 4156.

The transforms for the untreated sample (Fig. 4) shows prominent AB, AAB, ABB peaks suggesting a long range ordering of this type. Table 5 shows that the calculated values for both maximum alternation and randomness do not agree with those observed. The transforms for the glycolated sample (Fig. 4) similarly show strong combination peaks of type AC, AAC and ABB, again suggesting a tendency toward a long range ordering. The calculated values for maximum alternation and randomness in the glycolated sample do not correlate well with

the observed peaks. It appears, therefore, that the sample has a long range ordering of type AC, AAB, ACC, . . . for which calculated and observed peak heights would not be expected to agree.

	Un	treated			Glyco	olated	
Peaks	Observed Heights	Calculated Maximum	Heights	Peaks	Observed Heights	Calculated Maximum	Heights
A B A^{2} AB B^{2} $A^{2}B$ AB^{2}	.57 .43 .17 .54 .27 .37 .61	.57 .43 .14 .86 .00 .65 .32	.57 .43 .32 .49 .19 .42 .32	A B C A ² AB B ² -AC A ³ -AC C ² -A ² B AB ² -A ² C A ⁴ -ABC	.55 .16 .29 .25 .11 .79 .28 .00 .70 .29	$\begin{array}{c} .55\\ .16\\ .29\\ .10\\ .32\\ .58\\ .02\\ .48\\ .20\\ .17\end{array}$	$\begin{array}{r} .55\\ .16\\ .29\\ .30\\ .18\\ .35\\ .26\\ .23\\ .17\\ .25\end{array}$

 TABLE 5. OBSERVED AND CALCULATED FOURIER TRANSFORM PEAK HEIGHTS FOR SAMPLE 4156

Examination of the Fourier transforms for the untreated sample 4154 (Fig. 4) shows high peaks at AB and ABB similar to sample 4156. The calculated and observed heights of the transform for the glyoclated sample (Table 6) do not show good agreement. The transform (Fig. 4) has high combination peaks at AC, AAC and ABB, again suggesting a long range ordering.

	Un	treated			Glyco	lated	
		Calculated	Heights			Calculated	Heights
	Observed	Maximum			Observed	Maximum	
Peaks	Heights	Alternation	Random	Peaks	Heights	Alternation	Random
A	.62	.62	.62	A	.59	.59	.59
B	.38	.38	.38	B	.14	.14	.14
A^2	.19	.24	.38	C	.27	.27	.27
AB	.50	.76	.47	A^2	.19	.18	.35
B^2	.00	.00	.15	AB	.05	.28	.17
A^2B	.62	.68	.41	B^2-AC	.81	.54	.34
AB^2	.15	.23	.25	A ³ -BC	.31	.05	.29
				$C^2 - A^2 B$.00	.22	.22
				A^2C-AB^2	.76	.43	.32
				A4-ABC	.36	.02	.26

 TABLE 6. OBSERVED AND CALCULATED FOURIER TRANSFORM PEAK HEIGHTS

 FOR SAMPLE 4154

The transform of the untreated sample 4138 (Fig. 5) has large combination peaks at AB, AAB and ABB, again suggesting long range ordering. Neither of the calculated series of peaks in Table 7 show good agreement with those observed. Similarly, the transform (Fig. 5) of the glycolated sample has large composite peaks at AB, AC, AAB, AACand ABB, and neither of the calculated series of peaks show good agreement with the observed values.

It is probable that the mixed layer clay in sample 4138 has a long range ordering of the type AB, AC, AAB, AAC, ABB, ...

	Unt	reated			Gl	ycolated	
		Calculated	Heights			Calculated	Heights
Peaks	Observed Heights	Maximum Alternation	Random	Peaks	Observed Heights	Maximum Alternation	Random
A	.53	.53	.53	A	.50	.50	.50
\tilde{B}	.47	.47	.47	B	.35	.35	.35
A^2	.15	.06	.28	C	.15	.15	.15
ĀB	.47	.94	.50	A^2	.09	.00	.25
B^2	.38	.00	.22	AB	.32	.91	.35
A 3	.00	.01	.15	B^2-AC	.43	.30	.27
A^2B	.31	.10	.38	A 3-BC	.04	.00	.24
AB^2	.52	.42	.36	$C^2 - A^2 B$.21	.35	.55
B^3	.15	.00	.10	A^2C-AB^2	.46	.40	.41
				A4-ABC	.03	.21	.21

 TABLE 7. OBSERVED AND CALCULATED FOURIER TRANSFORM PEAK HEIGHTS FOR SAMPLE 4138

TABLE 8. OBSERVED AND CALCULATED FOURIER TRANSFORM PEAK HEIGHTS FOR SAMPLE 4132

Glycolated								
Calculated Heights								
Peaks	Observed Heights	Maximum Alternation	Random	Mechanical Mixture				
A	.20	.20	.20	.20				
B	.18	.18	.18	.18				
C	.62	.62	.62	.62				
A^2	.31	.00	.04	.20				
ÂB	.05	.00	.09	.00				
$B^2 - AC$.21	.40	.57	.18				
A ³ -BC	.20	.36	.23	.20				
$C^2 - AB$.61	.24	.39	.62				
$AB^2 - A^2C$.46	.06	.16	.00				
A4	.00	.00	.00	.20				

The transform of the untreated sample 4132 was not calculated because interpretation of the observed transform (Fig. 5) is in doubt. Examining the transform of the glycolated sample, the high combination peaks at CC + AAB and ABB + ACC stand out. None of the calculated arrangements shown in Table 8 agree with the observed peaks. It must, therefore, be assumed that a long range ordering of the type AAB, ABB, AAC, ... is present.

This arrangement, however, does not account for the large amount of component C (montmorillonite) present. It therefore appears that a large part of this component is present as a mechanical mixture.

X-RAY SPECTROCHEMICAL ANALYSIS

Instrumentation

Recent developments in instrumentation have enabled fluorescence equipment to analyse for elements as light as Mg (Z = 12). Thus all major rock forming metals, with the exception of Na, may now be quantitatively determined by this rapid, non-destructive method. However, with the sample preparation used it has been found that Mg is not detectable below about 10 per cent, so it is not reported in the materials studied. A standard Norelco vacuum-path spectrograph was used in this work.

Sample preparation

Several techniques for sample preparation are presently available (Claisse, 1960). For the present study a technique used in the Department of Geology at the University of Alberta was employed. The samples used for standards were ground for $1\frac{1}{2}$ to 3 hours in acetone with a mechanical mortar to sufficiently reduce the partical size. The samples for analysis were separated from the field samples by sedimentation techniques, and thus only 2μ to $\frac{1}{2} \mu$ size material was present. About $1\frac{1}{2}$ grams of sample were diluted 1:1 with borax, and this mixture ground in a mechanical mortar with acetone for 15 to 20 minutes. The preparation was then put in a $1\frac{1}{4}$ inch diameter cylinder, backed with 1 to 2 grams of borax, and compressed at 15,000 lb. for three minutes.

Calibration

In x-ray fluorescence analysis, quantities are not measured directly, but must be determined by intensity comparisons between the unknowns and standard samples that have been previously analysed by other techniques. An initial scan is made to ascertain which elements are present and this is followed by detailed measurement of the individual peak heights.

Due to interference effects (Molloy, 1959; Claisse, 1960), standard samples used for calibration should be as close in composition and structure to the unknowns as possible. This is especially true for the complex compounds encountered in clay analysis. A series of clays analysed by wet chemical techniques by Kerr *et al.* (1951) for API (American Petroleum Institute) Research Project 49, along with one bentonite and one shale analysed in the Department of Geology at the University of Alberta, and a number of synthetic samples were used in preparing the calibration curves. The instrument operating conditions are given in Table 9.

Elem	ent	Crystal	Peak U	lsed Order	Peak Location °20	Con Type	unter Voltage	Pulse H Analy Base W Line V.	leight yser /indow V.	Recording ¹ Conditions
	26		- K 1	1						
Fe	-0	LiF	$\tilde{\mathbf{K}}$ 2	ĩ	57.5	Sci.	800			512 - 1 - 8
	25		K 1	2						
Mn		ADP	K2	2	16.47	F.P.	1300	5.0	3.0	1-1-8
	22		K 1	2						
Τi		ADP	$\mathbf{K} 2$	2	32.10	F.P.	1310	4.0	4.0	4-1-41
	20		K 1	2						
Ca		ADP	K 2	2	48.26	F.P.	1400	15.0	5.0	2-1-8
	19		K 1	1						
K		ADP	${ m K}~2$	1	11.06	F.P.	1430	15.0	10.0	32 - 1 - 2
	14		K 1	1						
Si		ADP	$\mathbf{K} \ 2$	1	54.15	F.P.	1430	6.0	6.0	4-1-4
	13		K 1	1						
Al		ADP	$\mathbf{K} 2$	1	73.33	F.P.	1430	4.5	5.5	2-1-8

TABLE 9. OPERATING CONDITIONS FOR X-RAY SPECTROCHEMICAL ANALYSIS*

Sci. = Scintilation, F.P. = Flow Proportional, Amplifier gain 0, Gas flow 0.25 SCFH. NOTE: When using PHA, circuit must be on differential. When PHA is wide open, circuit is on integral and base line voltage is zero.

*Basic x-ray unit operated at 50 KV, 40 MA.

¹Scale factor — multiplier — time constant.

Correction for the absorption of Si radiation by Al

Although the samples were diluted 50 per cent by weight with borax it appeared that Al was absorbing some portion of the energy emitted by Si.

An attempt has been made to correct for the Al absorption effect. The silicon peak height was run on a set of SiO_2 -Al₂O₃ samples and

plotted against per cent Al₂O₃ present. As there would be no correction at 0% Al₂O₃ and at 0% SiO₂ a straight line between these points will represent a silicon calibration with no interference from Al. A curve through the plots of Si peak height against % Al₂O₃ will show the effect of the Al absorption and lie beneath this straight line. A curve for correcting the intensity of the Si peaks (Fig. 6) was constructed. Thus by analysing for Al before Si it is possible to correct the height of the Si peak for Al absorption, and such a correction was made in this study.

The Al absorption of Si radiation while decreasing the Si peaks may be expected to simultaneously enhance the Al peaks. A duplicate set of Al_2O_3 -SiO₂ and Al_2O_3 -CaCO₃ samples showed that this affect is not measurable.



FIG. 6. Correction curve for Si absorption by Al.

Analysis of the bentonites and shales

Analysis of the unknown samples consisted of taking peak heights and determining the corresponding quantities of oxides from the calibration curves. In the case of Si, the Al absorption correction was made before the percentage was determined from the curve. The results have been given in terms of oxides in order to be consistent with accepted methods of presenting rock analyses (Table 10).

		Bentonites				
	4156	4154	4138	4132	4134	4133
SiO ₂	49.5%	51.5%	56.5%	50.5%	53.0%	58.5%
Al ₂ O ₃	37.5	27.0	22.5	24.0	16.0	16.5
Fe ₂ O ₈	3.85	2.80	4.20	3.90	6.60	7.00
CaO	.66	.85	.97	1.25	1.32	.98
K ₂ O	4.65	4.65	3.90	3.05	2.30	2.50
TiO ₂	.17	.17	.35	.15	.55	.49
MnO	.03	.02	.02	.05	.09	.10
Total	96.36	86.99	88.44	82.90	81.86	86.07

TABLE 10. CHEMICAL COMPOSITIONS DETERMINED BY X-RAY SPECTROCHEMICAL METHOD

Comparison of fluorescence analysis with wet chemical analysis

The wet chemical analyses of two of the bentonites are compared with the fluorescence analyses of the same samples in Table 11. More detailed comparisons given by Chodos & Engel (1961) suggest that as a semi-quantitative analysis, this rapid non-destructive x-ray fluorescence method of analysis will be useful in geological research. The techniques used in fluorescence analysis are in the early stages of

	Sampl	le 4156	Samp	le 4138
	Wet* Chemical	Fluores- cence	Wet Chemical	Fluores cence
SiO ₂	52.34	49.5	52.73	56.5
Al ₂ O ₃	24.98	37.5	23.43	22.5
Fe ₂ O ₃ †	4.22	3.85	4.34	4.20
CaO	.54	.66	.80	.97
K₂O	4.45	4.65	3.61	3.90
MgO	2.3		2.36	
TiO ₂	.19	.17	.37	.35
MnÖ	.01	.03	.01	.02
Na ₂ O	.24		.24	—
P_2O_5	.10		.09	
H_2O+	6.63		6.16	
$H_2O -$	4.19	_	5.86	—
Total	100.19		100.00	

TABLE 11. COMPARISON OF WET CHEMICAL ANALYSIS AND FLUORESCENCE ANALYSIS FOR TWO BENTONITES

*Analysis done by A. Stelmach in the Rock Analysis Laboratory University of Alberta.

†Total Fe is reported as Fe₂O₈.

- No Analysis.

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development and may be expected to become more refined and accurate in the next few years, especially with respect to sample preparation and the establishment of more accurate calibration curves.

Conclusions

One of the objects of this study was to establish criteria for characterizing the bentonites. The results obtained show that there are no major differences in the clay minerals of the bentonites. However, the lack of montmorillonite as a separate mineral in sample 4156, and its presence in the other three samples along with the higher percentage of kaolinite in 4156 may help to distinguish this bentonite from the other three. The analysis of the mixed layer clavs show that samples 4156 and 4154 contain similar amounts of illite, montmorillonite, and vermiculite. In contrast, sample 4138 has twice as much vermiculite as the first two, and less of both illite and montmorillonite; and sample 4132 has over twice as much montmorillonite as any of the others. This prevalence of interstratified montmorillonite (and correspondingly small amount of illite) along with a large amount of mechanically mixed montmorillonite will probably distinguish bentonite 4132 from the others. Otherwise, the type of interstratification is almost identical in all the samples. All four bentonites are chemically very similar.

The two shales are significantly different from the bentonites both mineralogically and chemically. Both shales lack mixed-layer clays, and contain much larger portions of kaolinite, and smaller portions of illite. Sample 4133 also contains some chlorite. Chemically the two shales are very similar to each other (despite a considerable difference in color), and contain more Fe, Mn and Ti, and less K and Al than the bentonites.

Assuming that the environment of deposition of the sediments used in this study was fairly constant, it appears that the source and not the environment was the dominant factor in determining the clays present. The bentonites, which are chemically very similar and only slightly different mineralogically, suggest similar volcanic sources. Whether the differences noted can be used to distinguish the bentonites must await further work on other samples to see if the differences persist laterally or vary as a result of local environmental conditions. As the shales were deposited in an environment very similar to the bentonites, differences in the clay minerals are due to different source material. The fact that the clays of the bentonites and shales were deposited in a similar environment and have retained significant compositional differences supports the conclusion of Weaver (1958a, 1958b, and 1959) that the environment of deposition only slightly modifies the composition of clay minerals.

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