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Pharmaceutical Investigation of Selected Alberta Bentonites I

Geology and Identification

By ARTHUR J. ANDERSON† and ELMER M. PLEIN

Bentonite samples were collected from a number of Alberta deposits. Of these, 23 were indexed in table form to show location and other information dealing with the parent beds. On a selected suite of samples, identification as bentonites was confirmed by differential thermal and X-ray diffraction techniques.

EARLY INFORMATION concerning bentonite deposits in western Canada for the most part is found as brief reports in publications of the Canadian government (1–4). Of particular value in this respect are the comprehensive surveys of Spence (5) and Spence and Light (6).

In 1949 the discovery of major oil reserves in Alberta brought an unprecedented demand for the high-swelling type of clay. With no known reserves of suitable quality in Canada, all supplies, of necessity, were imported from the United States. In recent years, however, intensive subsurface exploration programs carried out by commercial interests resulted in the location of a number of reserves considerably better in quality than those previously described. Of these, deposits uncovered at Rosalind and Onoway were considered sufficiently important to warrant commercial development. Mines and processing plants were established at both locations with production designed to meet the needs of the petroleum and associated industries.

Our interest in the material arose when claims were made that certain beds contained bentonite comparable in some respects to that obtained from the Black Hills region of Wyoming and South Dakota. A subsequent inquiry revealed that insufficient information was available to allow for an adequate pharmaceutical evaluation of bentonites from any of the known Alberta deposits. For that reason, a study was undertaken with a view to determining the presence of reserves of pharmaceutical grade clay in the province.

GEOLOGY

Byrne (7) states that in Alberta, thin beds of bentonite are fairly common throughout the Cretaceous and Tertiary eras. However, thick accumulations have been reported only from the upper Cretaceous, with the Edmonton formation undoubtedly being the most important for future prospecting (Fig. 1). The occurrences found in other levels of the upper Cretaceous as well as in the Paskapoo member of the Tertiary are considered to have little commercial importance.

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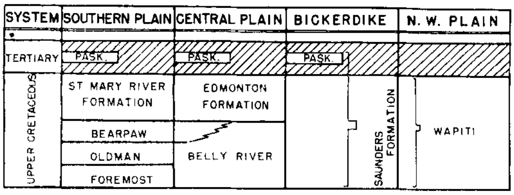

Fig. 1.—Stratigraph table of bentonite-containing formations in Alberta. *, Quaternary; Pask., Paskapoo.

In general, the Edmonton formation consists of poorly indurated shales and clays mixed with feldspathic siltstones and sandstones. Beds of relatively pure bentonite usually are found as lenses of varying widths. Coal seams are plentiful, and iron concretions are sometimes encountered.

EXPERIMENTAL

Collection and Preparation of Samples.—Since bentonite beds are known to vary widely in properties within short distances, a number of core samples are normally taken from each deposit to obtain an appraisal of clay quality throughout the occurrence. In this study, such a program was considered impractical because of transportation difficulties and the limited facilities available. Instead, every effort was made to obtain clean material, at varying depths below the surface of the deposit. In some instances a trenching procedure was employed to prevent contamination with surface sand. Several portions were gathered from different parts of each deposit and mixed to form a composite.

It was recognized that such samples were still not truly representative. For this reason, subsequent laboratory examination was considered to give only a general indication of the quality of the parent beds.

Twenty-nine samples were examined in the course of this work (Table I). Clays collected during field trips averaged between 8 and 10 lb. in weight, while samples received from other sources varied widely in size from 200 Gm. to 9 Kg. Coarse contaminants were removed by hand in the laboratory and the remaining material was dried in an oven at 90°. Each sample was reduced to a fine powder (200 mesh), mixed well to obtain a uniform blend, and then stored in a suitable glass, metal, or polythene container.

Identification of Clays.—Even though most of the samples used in this study were obtained from bentonite deposits which previously had been described by professional geologists, all clays were again identified as bentonite by qualified mineralogists either during or after collection. Recognized field tests were employed for this purpose.

After laboratory investigation had been conducted for some time it was felt that instrumental identification of the better and more interesting samples would be desirable. Arrangements were then made to have a suite of selected clays subjected to analysis by differential thermal and X-ray diffraction techniques.

Differential Thermal Analysis.—In recording the thermograms of the clay samples, a heating rate of 15° per min. was maintained over the entire temperature range (Figs. 2 and 3). Arrangement of the thermocouples, design of the block, and other details of the equipment used are described by Berkowitz (8).

TABLE I.—INDEX OF BENTONITE SAMPLES STUDIED

Sample[a]	Geologic Formation	Location	Map Coordinates
1	Edmonton	Rosalind[b]	Lsd. 15, Sec. 31, Tsp. 42, R. 17, W. 4
1A	Edmonton	Rosalind	Lsd. 15, Sec. 31, Tsp. 42, R. 17, W. 4
2	Edmonton	Rosalind	Lsd. 15, Sec. 31, Tsp. 42, R. 17, W. 4
3	Edmonton	Rosalind	Lsd. 15, Sec. 31, Tsp. 42, R. 17, W. 4
4	Edmonton	Drumheller	N.W. 1/4, Sec. 14, Tsp. 29, R. 20, W. 4
5	Edmonton	Beynon	S.E. 1/4, Sec. 32, Tsp. 27, R. 20, W. 4
6	Bearpaw	Dorothy	N.E. Sec. 3, Tsp. 27, R. 17, W. 4
7	Bearpaw	Irvine	N.W. 1/4, Sec. 30, Tsp. 11, R. 2, W. 4
8	Upper Oldman	Walsh	S.W. Sec. 28, Tsp. 11, R. 1, W. 4
9	Bearpaw	Bullshead Butte	N.E. 1/4, Sec. 2, Tsp. 8, R. 7, W. 4
10	Edmonton	Busby	N.E. 1/4, Sec. 7, Tsp. 56, R. 2, W. 4
16	Edmonton	Nevis	S.W. 1/4, Sec. 22, Tsp. 39, R. 22, W. 4
20[c]	...	...	Volclay, B.C. Grade.
21	Edmonton	Rosalind	
22	Edmonton	Rosalind	
23	Edmonton	Smoky River[d]	Lsd. 6, Sec. 35, Tsp. 69, R. 4, W. 6
24	Wapiti	Kleskun Hills	Sec. 15, Tsp. 72, R. 4, W. 6
25	Wapiti	Kleskun Hills	North Edge Secs. 21, 22, 23, Tsp. 72. R. 4 W. 6
26	Wapiti	Kleskun Hills	North Edge Secs. 21, 22, 23, Tsp. 72, R. 4, W. 6
27	Edmonton	Smoky River[d]	Lsd. 4, Sec. 2, Tsp. 70, R. 3, W. 6
28	Edmonton	Rosalind	
29	Control		Volclay, B.C. Grade
30	Edmonton	Onoway	Lsd. 9, Sec. 7, Tsp. 56, R. 2, W. 5
31	Edmonton	Onoway	Lsd. 9, Sec. 7, Tsp, 56, R. 2, W. 5
32	Edmonton	Rosalind[e]	

[a] Samples not listed were rejected after preliminary examination. [b] Rosalind beds contain several grades of clay roughly classified on a color basis: green (1, 1A, 21, 22, 28), gray (2), and yellow (3). [c] A small sample of finely ground bentonite received from the American Colloid Co. for testing. When it proved satisfactory for use as a control, a larger quantity was supplied for this purpose (sample 29). [d] Not previously reported. [e] Fines collected from vent stack of Rosalind mill.

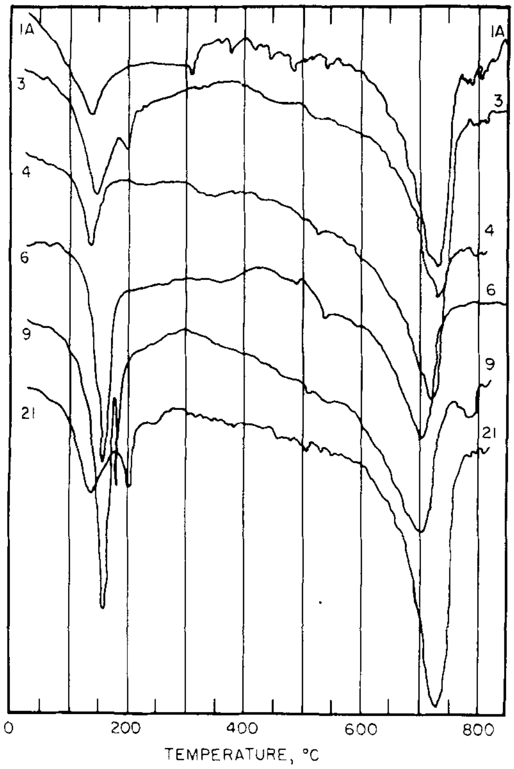

Fig. 2.—Differential thermal analysis of selected Alberta bentonites.

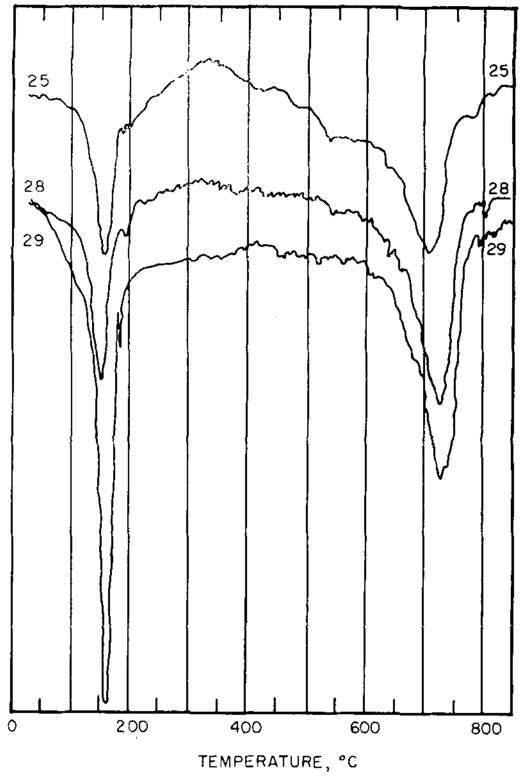

Fig. 3.—Differential thermal analysis of selected Alberta bentonites.

X-Ray Diffraction Analysis.—The clay portion of bentonite has been shown to consist primarily of montmorillonite. With the discovery that this material was actually crystalline in nature, it became possible to employ X-ray techniques to measure the characteristic angles of reflection of such rays from the atomic planes of the crystals.

A 2% (w/w) suspension of clay was prepared in a Waring Blendor (9). This mixture was transferred to a measuring cylinder and allowed to stand undisturbed for 8 hours and 10 min. By means of a pipet, 25 ml. of the suspension was then withdrawn from immediately below the surface and allowed to run into a clean Petri dish containing two glass microscope slides which were completely covered in the process. The assembly was then dried in an oven at 55–60°, and the slides removed with the aid of a razor blade to prevent chipping the oriented clay film. The prepared sample was then placed in a Phillips X-ray spectrometer and exposed to a monochromatic beam obtained through the use of a copper target and nickel filter. Scanning rate was set at 1° per min., through 40°, with the tracing recorded on a synchronized Brown strip chart recorder.

A diffraction tracing of the clay film was obtained, after which the slide was transferred to a glass desiccator and exposed to ethylene glycol vapor for 1 hour at room temperature. A second tracing was then prepared as before.

Basal (001) spacings and higher order reflections were calculated in each case and the values presented in Table II. As a matter of interest, Samples 28 and 29 were also heated to 550° for 30 min. and subsequently X-rayed. This latter procedure was undertaken to illustrate the reported collapse of montmorillonite under such conditions (10).

Table II.—X-Ray Diffraction Values of Selected Bentonites[a]

Sample No.					
1A	Untreated	11.26	4.03	3.13	..
	Glycolated	16.52	8.39	5.59	3.38
3	Untreated	11.63	4.02	3.11	..
	Glycolated	16.52	8.39	5.59	3.38
4	Untreated	10.72	4.76	3.15	..
	Glycolated	15.64	8.27	6.32	3.33
6	Untreated	11.05	3.15	..	..
	Glycolated	16.52	8.43	5.57	3.36
9	Untreated	11.19	3.13	..	..
	Glycolated	16.37	8.43	5.57	3.36
21	Untreated	11.79	4.93	3.11	..
	Glycolated	16.37	11.48	3.13	..
25	Untreated	12.11	3.11	..	..
	Glycolated	16.37	8.59	3.29	..
28	Untreated	10.59	4.80	4.01	3.14
	Glycolated	16.67	8.55	5.57	3.36
	Heated	9.46	4.75	4.04	3.16
29	Untreated	9.51	7.38	4.72	3.15
	Glycolated	15.64	8.27	6.33	3.33
	Heated	9.36	4.73	3.16	2.45

[a] Lattice spacings in Å.

DISCUSSION

The thermograms prepared in this series have generally similar shapes, although variation in certain details is observed. The initial endothermic peak caused by loss of interlayer water occurs in all cases between 140 and 160°. Where present, the notch on recovery is attributed to the presence of calcium in the lattice. High temperature exotherms are not recorded because of difficulties encountered with the apparatus when temperatures were raised much above 800°.

Each sample was X-rayed before and after treatment with ethylene glycol, with the glycolated materials giving basal spacings slightly less than the accepted value of 17 Å. (11, 12). It was shown by Mackenzie (12) that this spacing would be reduced to lower values when insufficient ethylene glycol was present to complete the complex formation. However, before discarding the data, in question, a detailed study of the tracings was undertaken by Dr. L. B. Halferdahl.[1] While several of the clays showed quartz and/or gypsum peaks he was able to classify each of the samples as bentonite containing montmorillonite as the chief clay mineral. This finding, together with the evidence gathered from the differential thermal studies, was considered as sufficient to confirm the identity of the clays under study.

Further laboratory data designed to assist in evaluation of these clays as possible pharmaceutical agents will be reported in part II of this investigation.

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Pharmaceutical Investigation of Selected Alberta Bentonites II

Limit Tests and Mechanical Analysis

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A collection of bentonite samples from known Alberta deposits was examined. Limit tests as described in the B.P. and U.S.P. were completed on each. Additional procedures were undertaken to assist in evaluation of physical properties of the clays. On the basis of this work, one sample was shown to be of pharmaceutical quality, with a second sample from the same commercially important deposit being excluded from this category on a minor technicality only.

Twenty-nine samples of bentonite taken from deposits throughout Alberta were examined in the course of this work. All clays were identified as bentonite by qualified mineralogists, with verification by differential thermal and X-ray techniques being made on a selected group of interesting samples (1).

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A laboratory study of the collected materials was then made to determine the presence of reserves of pharmaceutical grade bentonite in the province. A rigorous chemical analysis was not undertaken since pharmaceutical requirements were based mainly upon the inherent physical properties of the clays. However, certain chemical procedures were completed to aid in characterization.

EXPERIMENTAL

All samples were dried at 90°, reduced to a fine powder (200 mesh) by passage through a laboratory hammer mill, and then mixed well to obtain a uni-