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A STUDY OF EGYPTIAN BENTONITE BY X-RAY DIFFRACTION METHOD

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Abstract. Newly discovered Egyptian bentonite was subjected to detailed methods of X-ray diffraction analyses. The oriented aggregate method was used in the preparation of the sample for the examination of clays. The advantage of this method is that the enhancement of diagnostic (001) reflections allows them to be seen when the mineral is present in small proportions. X-ray quantitative analysis was carried out using an internal standard method. The raw mineral was composed of bentonite clay and free silica. It is a mixture of 67% bentonite and 33% free silica. The results were compared and discussed in terms of the patterns of widely distributed bentonites from the U.S.A., Germany and the USSR.

Material and Sample Preparation

Three samples from different localities were obtained from Fayoum, Egypt. The mineral was finely ground to pass 200 mesh B.S.S. The raw minerals are not pure clay and for this reason the following method was adopted to concentrate the clay mineral.

Following Brown,[1] oriented aggregates were made by centrifuging a 1%-suspension of clay. The supernatant liquid was decanted after centrifuging and clay was removed and dried at 40–50°. This is much quicker and simpler than allowing the suspension to evaporate to dryness in a vacuum desiccator and appears to give stronger, more cohesive aggregates.

For photographic techniques oriented aggregates, in the form of rod-shaped specimens were obtained by coating externally a thin capillary about 0.3 mm dia and allowing it to dry. Thin strips were also made by gentle pressure with razor blade. These strips were mounted on a thin glass fibre and rotated in a powder camera. The strips were exposed to boiling glycerol vapours and the patterns were again recorded. This treatment helps to form stable complexes.[2,3] Randomly oriented powders of the samples under investigation and standard bentonites were made by loosely packing the powder in a thin capillary which is rotated in a powder camera.

For the diffractometer techniques oriented flakes were mounted on the specimen holder. The specimen in its holder was exposed to glycerol vapours. The general reflections were studied by using randomly arranged powders.

Photographic diagrams and recording charts were taken for the specimens preheated to 120°. Humidity in camera or diffractometer sample chamber was controlled.[3]

Measurements Technique. X-ray qualitative analysis was carried out for the samples under investigation. Unicam powder camera (19-cm dia), Straumanis camera, stabilized X-ray Philips generator and diffractometer were used in these studies with filtered copper radiation. The photographs or the recording charts by diffractometer showed the diffraction patterns of bentonite together with free silica, (α-quartz). The reflection at d 3, 18Å could be plagioclase and at d 4.11Å might be cristobalite. As soon as the substances were identified, X-ray photographs and charts were taken for the most typical bentonite from Wyoming, Germany andthe USSR, (Figs. 1b–d,). The intensities and the spacings of the X-ray diffraction lines on the resulting charts were compared (Figs.1a–d, Table 1). These latter charts were used as a final check in the process of identification. The intensities were measured qualitatively by designating each lines as strongest (st,) strong (s), medium (M), weak,(W), faint (F), very faint (VF), and very very faint ($\bar{V}F$). An X-ray pattern was recorded for the sample treated with glycerol (Fig. 2).

X–ray quantitative analysis was carried out using the diffractometer. The internal standard method[4] was used in this analysis. Following Alexandar and Klug,[5] fluorite (CaF_2) was found to be suitable internal standard. A calibration curve was prepared from mixtures of quartz and calcium carbonate of known composition, each mixed with enough fluorite to make the weight fraction of fluorite in each composite sample equal to 0.20. The percentage of quartz in raw materials was obtained by measuring the ratio I_Q/I_F for a composite sample containing the unknown and the same proportion of standard as was used in the calibration. I_Q is the intensity of the d 4.26Å. line of quartz and I_F is the intensity of the d 3.13Å line of fluorite. The percentage of quartz determined by this method equal to 30.4, 31.8 and 35.4 for the three samples under investigation. The average value was evaluated and was found to be equal to 32.8%. In other words Egyptian bentonite was composed of montmorillonite mineral and quartz with 67.2 and 32.8% respectivley.

Discussion

No information on X-ray diffraction data or patterns of Egyptian bentonite was recorded previously. From information on the chemical composition and differential thermal analysis or thermobalance analysis,[6] a body of knowledge has been built which enables the different mineral groups to

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Table 1. X-Ray Powdered Patterns of Egyptian Bentonite Compared with Typical Bentonites.

Egyptian bentonite		Wyoming bentonite		German bentonite		USSR bentonite		Type
dÅ	I/I_o	dÅ	I/I_o	dÅ	I/I_o	dÅ	I/I_o	
11·38	st	11·4	st	10·52	st	11·56	st	B
8·78	VF	8·8	VF			9·60	VF	B
5·80	M	5·8	W					B
4·48	S	4·48	S	4·48	S	4·48	S	B
4·29	F	4·28	F	4·30	F	4·28	F	B
4·24	M	4·26	W	4·27	W	4·25	M	Q
4·11	VF	4·08	VF	4·08	VF	4·11	VF	C
3·48	W	3·49	W	3·48	W	3·48	F	B
3·33	S	3·36	W	3·37	S	3·34	M	Q
3·18	M	3·18	M	3·18	M	3·18	M	P
2·90	F	2·91	VF	2·92	VF	2·92	VF	B
2·53	M	2·54	M	2·55	M	2·53	M	B
2·47	F	2·48	W	2·47	W	2·49	M	B
2·43	VF			2·46	F	2·46	VF	Q
2·26	VF			2·27	VF	2·30	VF	Q
2·22	V̄F	2·22	VF	2·23	VF	2·23	VF	B or Q
2·11	V̄F			2·12	VF	2·12	V̄F	Q
1·80	VF	1·81	V̄F	1·80	V̄F	1·80	V̄F	Q or B
1·68	W	1·68	W	1·67	VF	1·69	V̄F	B
1·64	V̄F			1·63	V̄F	1·65	VF	Q
1·53	F			1·54	VF			Q
1·48	M	1·49	S	1·49	M	1·49	M	B
1·36	V̄F							Q
1·30	F	1·29	F	1·30	F	1·30	F	B
1·24	VF	1·24	F	1·24	F	1·24	F	B
1·11	V̄F	1·12	V̄F	1·12	V̄F	1·12	V̄F	B
0·98	V̄F	0·98	V̄F	0·98	V̄F	0·98	V̄F	B
0·88	V̄F	0·86	V̄F	0·86	V̄F	0·87	V̄F	B

st strongest, S strong, M medium, W weak, F faint, VF very faint, V̄F very very faint, O zero, B bentonite, Q quartz, P plagioclase, C cristiobalite

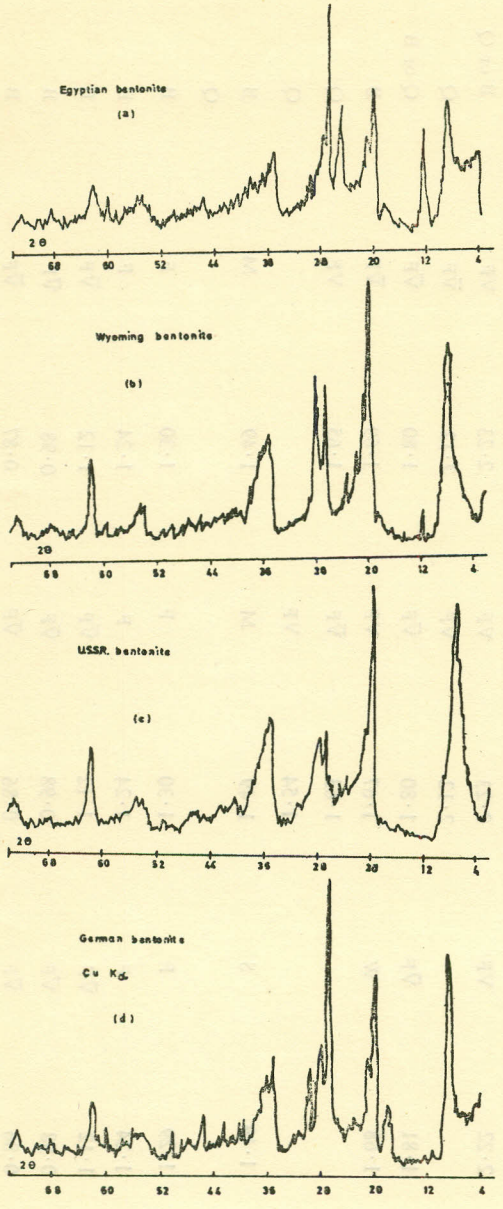

Fig. 1

be recognized. All these results were considered as a whole to see how far they complement or support each other. For as much information as possibile about a clay more than one pattern is needed. Powder patterns, i.e. patterns of unoriented specimens in addition to (00l) reflections, give the (hk), and (hkl) lines which are helpful. The best single test, however, is an oriented aggregate pattern. From it the probable components may be deduced and the number of subsquent patterns needed to complete the identification can be greatly reduced.

X-ray photographs and diffractometer studies of unoriented specimens showd that the Egyptian bentonite was composed of a mixture of montmorillonite mineral and free silica. The free silica exists as α-quartz. The results were compared with bentonites which occur widely distributed in the Western

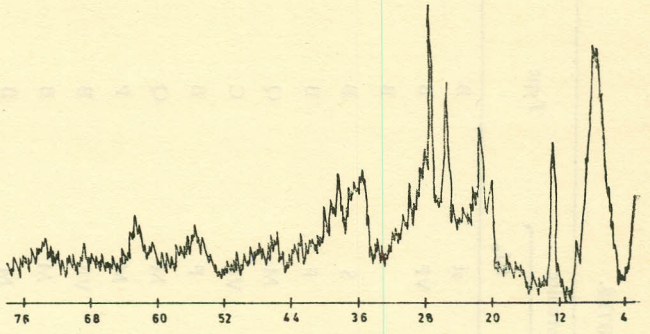

Fig. 2

TABLE 2. BASAL REFLECTIONS FROM GLYCEROL BENTONITE COMPARED WITH WYOMING BENTONITE.

001	Egyptian bentonite d (Å) calcd	Wyoming bentonite d(Å)
001	17·3	17·4
002	8·81 VF	8·85
003	5·90	5·90
004	4·30	4·33
005	3·51	3·450
006	2·94	2·950
007	2·51	2·520
008	—	2·213
009	1·86 VF	1·967
0,0,10	1·70 VF	1·770
0,0,11	—	1·609
0,012	—	1·476
0,0, 13	—	1·361
0,0, 14	—	1·264

TABLE 3. SPACING OF hK REFLECTIONS OF EGYPTIAN BENTONITE COMPARED WITH WYOMING BENTONITE.

hK	Egyptian bentonite (d Å) calcd	Wyoming bentonite[3](d Å)
11, 02	4·48	4·61
13, 20	2·53	2·56
22, 04	2·22	2·221
31, 15, 24	1·68	1•692
33, 06	1·48	1·492
26, 40	1·30	1·289
35, 17, 42	1·24	1·244
08, 44	1·11	1·120
19, 53, 46	0·98	0·9705
39, 60	0·88	0·8942

U.S.A. Germany and the USSR (Table 1, Fig. 1 a–d) Fig. 1a showed the pattern of Egyptian bentonite for powdered material dried at 120°. In comparison with other bentonites the pattern showed a small shift for the long spacing lines. This shift may be attributed to the fact that the long spacing line and its higher orders vary with water content.[7] For this reason the sample was treated with glycerol to form stable complexes, [2,3] (Fig 2, Table 2). The general reflections were studied by using randomly arranged powder and compared with patterns of standard bentonites. The (00l) and (hk) reflections were examined compared with reflections of Wyoming bentonite[3] (Tables 2 and 3). Egyptian bentonite has higher percentage of free silica than Wyoming and U.S.S.R. bentonites and it has less silica than German bentonite. This conclusion was observed and guided with the relative intensities of the patterns of quartz. Finally the reflection at d 3.18Å could be plagioclase and at d 4.11Å might be cristobalite.

X-ray quantitative analysis was carried out using internal standard method. This method is restricted to sample in powder form and has been widely used for the measurement of quartz content of industrial dust. It must be mentioned here that oriented aggregate method makes quantitative estimation impossible, but even alleged unoriented specimen of clays are usually partially oriented. In choosing diffraction lines to measure we must be sure to avoid overlapping or closely adjacent lines from different planes. CaF_2 (d 3.13Å) was used as an internal standard to estimate α-quartz (d 4.26Å) in the raw mineral. X-ray quantititave analysis showed that Egyptian bentonite is a mixture of 67.2% (average value) of montmorillonite clay and of 32.8% of free silica.

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