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CHEMICAL, DEHYDRATION, DIFFERENTIAL THERMAL AND X-RAY ANALYSIS OF SALAL BAUXITE DEPOSITS

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Chemical, dehydration, differential thermal and X-ray analysis data on four samples of mineral deposits from the Salal region (J & K) have been determined to assess their mineral compositions. All the samples appear to be kaolinitic in character, though in association with other mineral impurities, such as boehmite, diaspore and dickite, in varying proportions.

We earlier reported [1] the general physical properties of Salal bauxites. In the present paper, chemical, dehydration, differential thermal and X-ray analysis data have been determined to assess the mineral compositions.

Experimental methods and procedures

i) Chemical analysis

Chemical analysis of the samples was carried out with the ultimate method.

ii) Dehydration

One gram of material (– 120 ASTM) was heated in a platinum crucible at 100° until weight constancy. The percentage loss in weight was calculated and the process was repeated in steps of 100° up to 1400°. All weights were recorded while the samples were hot. The heating rate was 10 deg/min.

iii) Differential thermal analysis

An automatic Mettler TA-I set-up with a platinum sample holder and a platinum–rhodium thermocouple was used for curve recording. Mineral powder (– 120 ASTM) was used and the experiment was carried out in static air at a heating rate of 10 deg/min.

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iv) X-ray analysis

X-ray diffractograms of the mineral samples (−300 ASTM) were taken with a Phillips PW-1410 diffractometer sung Cu-K_α radiation.

Observations and discussion

Chemical analysis

The chemical composition data (Table 1) indicate that in B—I and B—II the alumina content is as high as 66%, with only 13—15% of silica. In B—III and B—IV the percentage of alumina is lower, while that of silica is higher. The oxides of iron and titanium total about 5% in B—I and B—II, 4% in B—III and merely 3% in B—IV. Alkali and alkaline earth metal oxides are present only in minor amounts. The ignition loss in all cases lies around 14%.

Table 1 Chemical analysis of samples (weight %)

Constituents	B—I	B—II	B—III	B—IV
L. O. I.	14.73	14.50	14.38	13.80
SiO_2	15.14	13.01	31.26	43.57
Fe_2O_3	3.32	2.20	2.20	1.81
Al_2O_3	64.03	66.38	49.53	38.35
CaO	0.24	0.37	0.42	0.31
MgO	0.15	0.20	0.20	0.15
Na_2O	0.33	0.23	0.16	0.16
K_2O	0.06	0.07	0.17	0.56
TiO_2	1.76	2.78	1.54	1.11

Although it is difficult to predict mineral compositions from chemical analysis data, it appears that B—I and B—II are highly aluminous in character as compared to B—III and B—IV. When coupled with the ignition losses of the samples and their non-swelling behaviour, this observation indicates their kaolinitic character. B—IV appears to be predominantly kaolinite, particularly in view of its silica—alumina ratio.

Dehydration analysis

The dehydration curves (Fig. 1) are all nearly flat up to 300/400°, indicating that the loss of water in these samples is very small at such low temperatures. This feature is usually shown by kaolinites. Indeed, most of the dehydration takes place between 400 and 600/700°, the region in which the curves show a very steep rise. However, the exact temperature for the loss of lattice water from OH varies only slightly, suggesting that the particle size and degree of crystallinity of the samples are of the same order. Beyond 600/700°, the curves once again show a very gradual rise up to around 800°. It appears that the samples still retain about 2 to 3% of lattice water, which is released only gradually beyond 600/700°, and that the dehydration is in all cases practically complete by around 900/1000°, beyond which the curves become almost flat up to 1400°, the maximum temperature of the experiment. The very low losses of weight at the initial temperatures, and the S-shaped dehydration features, are indicative [2, 3] of the kaolinitic nature of the samples.

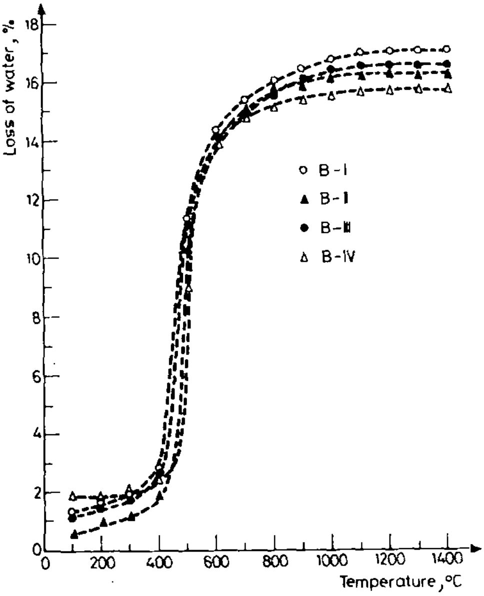

Fig. 1 Dehydration characteristics

Differential thermal analysis

The differential thermal curves (Fig. 2) show a small initial endothermic reaction at about 110°, indicating that a small amount of water, which may be present between the layers of the mineral, possibly due to some irregularities in the arrangement of the mineral units, is removed at around this temperature. An intense and fairly sharp endothermic reaction begins at around 450° in all cases, with peaks appearing in the neighbourhood of 600°. It is in this temperature region that most of the lattice OH is removed. This observation is compatible with the dehydration behaviour of the samples. It is believed [4] that the intensity of this reaction, and hence the size of the peak and the peak temperature, depends upon the particle size of the individual sample and its degree of crystallinity. Both of size of the peak and the temperature at which it appears are lowered with the decrease in particle size or in order of crystallinity.

Following dehydroxylation at around 600°, the endothermic reactions of varying intensities indicate that the samples still retain some water, as well as some degree of order, which is lost during the higher-temperature endothermic reactions. It is gener-

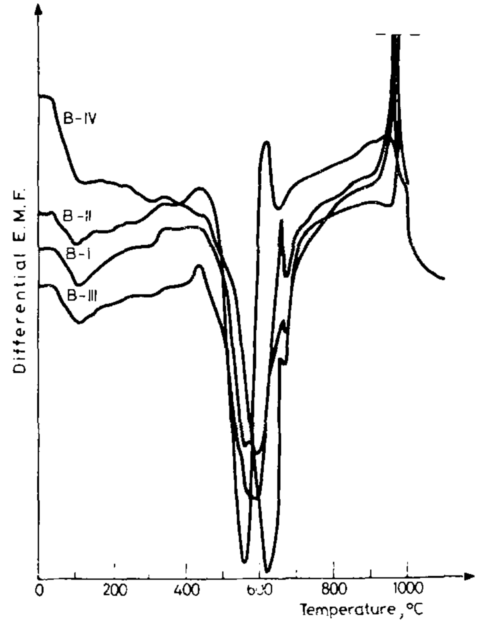

Fig. 2 Differential thermal analysis curve

ally believed that, when heated just above the temperature of complete dehydration, kaolinite is converted into the meta-kaolin phase, which still retains some degree of order. In meta-kaolin, the Si—O network for kaolinite remains largely intact, while the Al—O network is reorganized in the form of edge-shaped octahedral Al—O chains.

The fairly intense exothermic reactions observed between 960 and 980° cover a slightly wider temperature range, as an indication of their poor order of crystallinity or contamination with impurities. It is believed that the appearance of this peak for kaolinites is associated with the formation of either γ-alumina or mullite or both.

The appearance of a small endothermic reaction at around 650 to 670°, between the peak for the loss of OH and the exothermic reaction at about 950°, when coupled with a very small endothermic reaction just preceding the exothermic peak, points to the existence of some degree of crystallinity, particularly in the material of B-IV, even at such elevated temperature.

X-ray analysis

The X-ray diffraction data with the relative intensities of the peaks (Table 2) adequately demonstrate that all the samples are mixtures of kaolinite, dickite, boehmite and diaspore.

The prominent basal reflections at around 7.16, 3.56 and 2.32 Å provide sufficient evidence of the presence of kaolinite or dickite or both. While reflections with spacings of around 4.27 and 3.27 Å suggest the presence of dickite, those appearing at 4.18,

Table 2 X-ray analysis data

B–I		B–II		B–III		B–IV	
d, Å	*I*	*d*, Å	*I*	*d*, Å	*I*	*d*, Å	*I*
7.13	10	7.13	8	7.08	10	7.19	10
6.15	10	6.10	4	6.07	10	6.10	3
4.72	4	4.69	9	4.69	3	4.69	2
4.43	7	4.44	4	4.44	7	4.46	9
4.35	8	4.35	4	4.33	8	4.37	10
4.23	6	4.27	4	4.29	7	–	–
4.17	8	4.13	4	4.15	7	4.19	9
3.98	6	3.98	10	3.96	5	3.95	5
3.83	5	–	–	3.83	5	3.85	6
3.75	4	–	–	3.73	4	3.73	5
3.56	10	3.56	7	3.55	10	3.56	10
3.38	4	–	–	3.37	4	3.37	4
3.35	4	3.35	4	3.33	4	3.35	5
3.24	3	3.23	4	3.24	3	3.24	3
3.15	7	3.17	4	3.15	10	3.20	3
2.54	6	2.56	10	2.55	5	2.56	6
2.52	4	–	–	2.52	4	2.52	5
2.49	6	2.50	4	2.49	6	2.49	7
2.37	5	2.38	4	2.37	5	2.38	5
2.34	10	2.35	8	–	–	2.338	9
–	–	2.32	10	2.33	10	2.332	9
2.29	6	–	–	2.29	5	2.29	6
2.19	3	2.19	3	2.18	3	2.18	3
2.12	3	2.13	10	2.12	3	2.12	2
2.07	4	2.07	10	2.07	3	2.06	2
1.98	4	1.98	3	1.97	4	1.98	4
1.88	3	1.89	3	1.89	3	1.89	3
–	–	1.86	3	–	–	1.86	2
1.85	5	–	–	1.85	6	–	–
1.84	5	1.84	3	1.84	6	1.83	3
1.81	3	1.81	3	1.81	2	1.81	2
1.78	3	–	–	1.78	3	1.78	3
1.68	3	1.68	3	1.68	3	1.68	3
1.66	5	1.66	3	1.66	5	1.66	5
1.63	4	1.63	10	1.63	3	–	–
1.62	4	1.61	10	1.62	3	1.62	4
1.57	3	1.57	3	1.58	2	1.57	2
1.53	3	1.53	2	1.53	3	1.53	3
1.52	3	1.52	3	1.52	3	–	–
1.48	5	1.48	7	1.48	5	1.48	6
1.44	4	1.45	3	1.45	4	1.45	3

3.83 and 3.37 Å support the existence of kaolinite in the samples. In kaolinite, the observed tendency in the displacement of the positions of lines, and a single line appearing at around 4.18 Å instead of a well resolved doublet at 4.18 and 4.13 Å, and the group of lines between the (020 and 002) reflections at 4.45 Å and 3.56 Å, respectively, point to the variation in the particle size or the transition from a well crystalline to a poorly crystalline structure.

Although it is difficult to identify boehmite when this is present with mixtures of other hydrates of alumina, the first low-order reflection line (020) appearing at around 6.11 Å, and another strong reflection (021) at 3.15--3.20 Å, unmistakably indicate the presence of this mineral in the samples. In boehmite, the slight displacement in the positions of the lines may be due to the changes in the layer spacings owing to different amounts of water or foreign ions which may be entrained with the miscelles.

The presence of yet another dimorphic form of alumina hydrate, such as diaspore, is indicated by a very strong (110) reflection line at around 3.98 Å. This is well separated from the reflections for boehmite or other hydrates of alumina. Reflections such as those appearing at 4.69–4.72, 2.12–2.13, 2.06–2.07 and 1.63 Å further suggest the presence of this mineral.

Strong reflections with spacings of around 3.35 Å are indicative of the presence of free silica.

Conclusions

When supplemented with X-ray diffraction data, the results of chemical, dehydration and differential thermal studies suggest that the samples under consideration are basically kaolinitic in character, though in association with varying amounts of other mineral impurities, such as boehmite, diaspore and dickite, besides some free silica.

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Zusammenfassung — Vier Proben von Minerallagerstätten der Salal-Region (J & K) wurden chemisch sowie mittels DTA und röntgenographischer Analyse untersucht, um ihre Mineralzusammensetzung zu ermitteln. Alle Proben sind dem Charakter nach kaolinitisch, allerdings in unterschiedlichen Verhältnissen mit anderem mineralischem Material wie Boehmit, Diaspor und Dickit verunreinigt.

Резюме — Для определения минерального состава четырех образцов минералов, взятых из месторождения Салал, был использован химический, дифференциальный термический и рентгенофазовый анализ. Полученные данные показали, что все образцы являются минералами группы каолина с примесью находящихся в них бозмита, диаспора и дикита в различных соотношениях.