

CHEMICAL AND X-RAY EXAMINATION OF AN ASBESTOS-LIKE MINERAL OCCURRING IN HINDUBAGH

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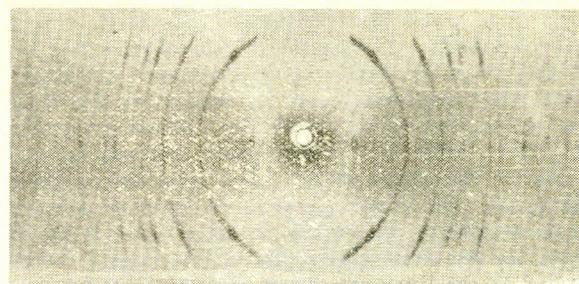
A fibrous asbestos-like mineral from Hindubagh has been examined chemically and with the help of X-ray diffraction patterns. It has been found to consist of 50% brucite ($Mg(OH)_2$), 40% dolomite ($MgCO_3CaCO_3$) and at most 10% chrysotile asbestos. The chrysotile component has been identified by means of fibre and powder X-ray patterns of the residue obtained from acid-treatment of the original sample.

A sample of fibrous material, reportedly asbestos, collected from Hindubagh, was received through the courtesy of the Geological Survey of Pakistan for a study of its possible uses in the asbestos industry. The bulk of the sample consisted of well-formed long fibres in rock formations. The fibres were greenish-white, silky smooth and lustrous, their texture varying from soft to brittle hard (the brittle hard material being predominant). Because various varieties of asbestos, each suited for specific uses, are found to occur in nature, it was considered essential, in the first instance, to identify and classify the mineral.

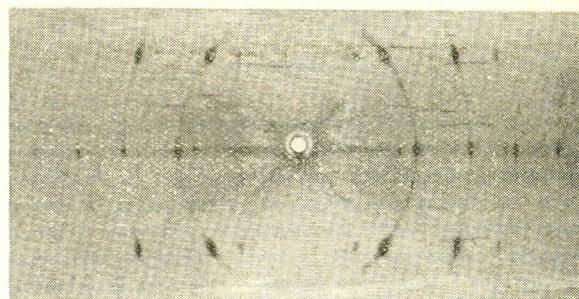
Axial-length Measurements along Fibre-axis

In order to apply H.V. Anderson and G.L. Clark's X-ray method¹ for the classification of asbestos (based on axial-length measurements along fibre axis and the weight lost by variously treated samples), X-ray patterns of hand-picked fibres were taken with a Cylindrical Bernal camera, using filtered Cu K radiation. The patterns thus obtained are reproduced in Figs. 1(a) and 1(b), Fig. 1(a) being the pattern obtained with the untreated fibres, while Fig. 1(b) shows the pattern of the fibres after heating at 1000°C. for 2 hours. Both these patterns show sharp powder lines, super-imposed upon which are several strong and fairly sharp diffraction spots arranged on layer lines, indicative of a good fibre structure oriented around the axis. In Table 1, the values of the axial lengths along the fibre axis, as calculated from these two patterns and the weights lost with standard chemical treatments are compared with the nearest values from the standard data given by Anderson et al.,¹ picrolite showing the highest loss on treatment, and chrysotile having the next highest in the list.

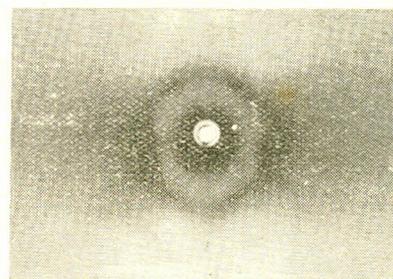
Examination of the data of Table 1 suggests that the mineral under study is essentially different from any of the listed varieties of asbestos. This was confirmed by the X-ray pattern taken after leaching with 1:1 hydrochloric acid for 12 hours.



(a)



(b)



(c)

Fig. 1.—X-ray fibre patterns of the asbestos-like mineral taken with a normal Bernal camera, using filtered Cu K radiation: (a) untreated fibres, (b) fibres after heating at 1000°C for 2 hours, and (c) fragment from residue after leaching with 1:1 HCl for 12 hours.

TABLE 1.—AXIAL LENGTHS ALONG FIBRE AXIS OF THE UNTREATED AND TREATED SAMPLES, TOGETHER WITH WEIGHT LOSSES: COMPARED WITH SOME STANDARD DATA.

Treatment given	Weight percent loss on treatment			Identity period (axial length) along fibre axis		
	Picrolite	Unknown	Chrysotile	Picrolite	Unknown	Chrysotile
1. Untreated sample	—	—	—	~ 5 A	3.14A	5.06A±0.14
2. Heat-treated sample at 1000°C. for 2 hrs.	35.1	35%	17.3±6.0	—	2.96A	—
3. Acid-treated sample (leached in 1:1 HCl for 12 hours)	85.9	95%	55.6±2.7	—	—	—

While the bulk of the material dissolved in the acid solution, some fragments of the residue gave the very poor pattern shown in Fig. 1(c), which is conspicuously different from the first two and has four faint spots, yielding an axial length of $4.8 \pm 0.2A$, which would agree with that of chrysotile. This suggests that, whereas the bulk of the material is not identifiable as asbestos, it probably contains a small fraction (about 10%) of some form of asbestos. A more detailed chemical and X-ray analysis was therefore undertaken to identify these and other possible components of the sample.

Chemical Analysis

A representative fraction of about 5 lbs. was taken from the bulk sample and crushed in a jaw crusher. By repeated crushing and sampling (by the quartering method), a finely-ground representative sample was obtained. This was oven-dried at 110°C. and then chemically analysed. The mean results of half a dozen analyses are given in Table 2. The deficit of 3.3% can be accounted for by alkali metals, etc.

TABLE 2.—CHEMICAL ANALYSIS OF THE UNTREATED SAMPLE

MgO	41.1%	Fe ₂ O ₃	3.8%
CaO	10.9%	Loss on ignition	36.7%
SiO ₂	4.2%	Total	96.7%

Generally the different varieties of commercial asbestos fibres vary in their SiO₂ content² from 37-62%, and thus the significantly low SiO₂ content of the sample would at best account for about 10% asbestos. Further, magnesium and calcium are present in appreciable quantities, and the loss on ignition is quite high, and would correspond to the presence of a carbonate or a hydrate

in appreciable quantities. The carbonate radical was detected by hydrochloric acid treatment, but its quantitative estimate was not carried through. Thus magnesium carbonate or hydroxide may be the major phase present in the sample.

X-ray Powder Patterns

For a complete identification of the actual component phases, recourse was again taken to X-ray diffraction methods. One of the phases can be identified as brucite, Mg(OH)₂, from the powder lines on the fibre pattern of the untreated sample (Fig. 1(a)). Furthermore, the fibre pattern of the heat-treated sample (Fig. 1(b)) also gave sharp powder lines, which correspond to MgO. A powder photograph of the representative sample was next taken with filtered Cu K radiation in a 9-cm. diameter powder camera. This photograph is reproduced in Fig. 2, along with comparison patterns of brucite, of asbestos, and of a 1:2 hydrochloric acid treated sample. In Table 3 are given the d-values of the lines of the experimental pattern together with those of comparison standards.

After accounting for the brucite lines on the experimental pattern, some prominent lines remain outstanding, those with d-values of 2.89, 2.19,



Fig. 2.—X-ray powder patterns of the mineral and of comparison standards, taken with 90 mm. dia. camera using filtered Cu K radiation: (a) standard sample of Brucite (Mg(OH)₂), (b) untreated mineral, (c) Chrysotile asbestos, and (d) residue from acid treatment of mineral.

2.01A (all visible in Fig. 2) being quite strong. A search in the A. S. T. M. card index³ showed that they could correspond with the strong lines of dolomite ($MgCO_3 \cdot CaCO_3$), the complete powder data of which was found to account for most of the outstanding lines (cf. Table 3). A semi-quantitative estimate from the intensities of the powder lines indicates a ratio of 5:4 for brucite and dolomite. Allowing 10% for any other phases (asbestos, etc.), this gives 50% brucite and 40% dolomite, which leads to an estimate of 40% for

magnesium oxide and 12% for calcium oxide, in good agreement with the chemical data of Table 2.

It may here be mentioned that the presence of magnesite ($MgCO_3$) bands in the asbestos samples have been reported by investigators in the Geological Survey of Pakistan. Therefore, the powder data of the pattern of magnesite and its hydrates, as given in the A.S.T.M. card index, was also looked up but they were not found to fit the experimental pattern at all.

Brucite and dolomite account for nearly all the lines of the pattern, and only a few weak and faint lines remained unexplained. Amongst these outstanding lines, a line at $d=7.5\text{\AA}$ and the other at $d=3.67\text{\AA}$ were quite significant; they correspond with the strongest lines of chrysotile, already identified in the fibre pattern of Fig. 1(c), and the complete data of whose pattern also agreed well with the remaining powder lines. This phase, however, must be present only in small quantities, and so in order to confirm its presence in the original sample, attempts were made to separate it from the bulk material.

The Chrysotile Component

Hydrochloric acid (1:2) successfully separated out the chrysotile component as a residue (weighing about 6% of the total weight of material treated by the acid) without disturbing the lattice of this phase. The powder pattern of this sample, also reproduced in Fig. 2, is seen to be very diffuse. Nevertheless, it compares well with the standard asbestos (chrysotile) pattern (Table 3).

The mean of three separate analyses of this acid-treated sample are given in Table 4. The deficit of 2.2% can be accounted for by alkali metals etc.

The rather high value of SiO_2 , which should be about 44% for chemically pure chrysotile, is attributable to free silica present along with the extracted fibres. The figure for magnesium is a little low, perhaps due to attack of 1:2

TABLE 3.—COMPARISON OF THE POWDER PATTERNS OF THE UNTREATED AND ACID-TREATED FIBROUS MINERAL WITH THOSE OF VARIOUS STANDARDS.

Untreated sample	Std. data of Mg(OH) ₂ (brucite)	Std. data of MgCO ₃ ·CaCO ₃ (dolomite)	Std. data of chrysotile	Sample treated with HCl
—	—	—	8.0 (40)	7.94 W
7.52 W	—	—	7.3 (90)	7.10 M
4.80 S	4.76(60)	—	4.93(10)	—
—	—	—	4.46(60)	4.43 M
3.67 WF	—	3.71(2)	3.64(100)	3.63 W
—	—	—	—	3.37 F
2.89 S	—	2.89(100)	—	—
2.66 W	—	2.68(2)	—	—
2.52 W	—	2.55(2)	—	—
—	—	—	2.44(70)	2.49 M
2.36 S	2.37(100)	—	—	—
—	—	2.40(13)	—	—
2.19 M	—	2.19(40)	—	—
2.05 F	—	2.06(2)	—	—
2.01 M	—	2.02(20)	—	—
1.84 F	—	1.85(2)	—	—
1.79 S	1.79(80)	1.80(40)	—	—
1.57 M	1.57(70)	1.57(2)	—	—
1.54 W	—	1.55(10)	1.52(90)	1.54 M
1.49 WM	1.49(60)	1.50(1)	—	—
1.46 F	—	1.47(6)	—	—
1.43 F	—	1.42(1)	—	—
1.38 F	—	1.39(8)	—	—
1.37 M	1.37(60)	—	—	—
1.33 F	—	1.34(2)	—	—
1.30 W	1.31(60)	1.30(1)	—	—
1.26 F	—	1.27(1)	—	—
1.23 F	—	1.24(2)	—	—
1.20 F	—	1.20(1)	—	—
1.18 F	1.18(50)	1.17(1)	—	—
1.11 F	—	—	—	—
1.10 WF	—	—	—	—
1.09 WF	1.09(20)	—	—	—
1.03 W	1.03(40)	—	—	—
1.00 W	1.00(60)	—	—	—

TABLE 4.—ANALYSIS OF RESIDUE FROM ACID-TREATMENT.

SiO ₂	51.3%	MgO	25.4%
Fe ₂ O ₃	1.1%	Combined water	17.7%
Al ₂ O ₃	0.9%	and CO ₂ (traces)	—
CaO	1.4%	Total	97.8%

hydrochloric acid, pure chrysotile containing 35% (approximately) magnesia. Thus the chemical analyses lend some support to the conclusion that the acid treated residue is chrysotile.

Conclusion

From the foregoing study, it is concluded that brucite and dolomite represent the major phases in the sample, while chrysotile (asbestos) forms only a small fraction (<10%) of the total weight of the material. Thus these three phases complete the list of the constituents of the sample.

Regarding the origin of the brucite (magnesium hydroxide), it may be conjectured that it is formed as a decomposition product of asbestos, as noted by earlier workers⁴—"The mineral is found with magnesite and dolomite in serpentine as a result of the decomposition of magnesium silicates".

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