

The decomposition of biotite in the soil.

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[Read April 9, 1949; Clay Mineral Group meeting at Leeds.]

A NOTICEABLE feature of many of the soils of north-east Scotland is the presence in them of glistening yellow flakes of decomposed biotite. The light-reflecting property of this material catches the eye even though the flakes themselves are often of very small size.

An analogous process to the breakdown of biotite as reported below occurs in the soil-clays of the area, and the present study was undertaken primarily in order to throw light on the mineralogical nature of the clay fractions of the soils, since the large amounts of amorphous material associated with the soil-clay minerals renders less satisfactory the direct approach. The mineralogy of the soil-clays will be dealt with in a separate communication.

REVIEW OF THE LITERATURE.

The changes taking place in the decomposition of biotite have interested mineralogists for many years. Prior to 1934, the terms 'hydrobiotite' (1, 2, 3) or 'vermiculite' (4, 5, 6) were often loosely applied to altered biotite in various stages of decomposition, but in that year Gruner (7) suggested precise definitions on structural grounds and thereby excluded many specimens previously called by these names.

At an early stage, it was established that the weathering of biotite causes an increase in the optic axial angle, a decrease in specific gravity and refractive indices, the disappearance of pleochroism, and a marked colour change from black through golden-yellow to silver and white (8, 9, 10). At the same time, the decomposition was found to be accompanied by a loss of iron, preceded by the oxidation of ferrous to ferric, loss of magnesium, potassium, and sodium, and a gain in water. If the decomposition proceeds far enough, the final product under natural conditions appears to be a kaolinite (9, 11, 12, 13), but at an intermediate stage vermiculite (12), chlorite (14), hydrobiotite (15), or other products (14) may be formed.

A chemical study by Denison, Fry, and Gile (16) of the decomposition of biotite in the soil showed similar trends and indicated that the degree

of alteration is related to the particle size; and Humbert and Marshall (14), studying the genesis of soils from igneous rocks, noted that penetration of water molecules by cracks along cleavage planes in biotite could be followed under the microscope by means of the traces of oxidation left behind.

Hydrothermal alteration of biotite as opposed to natural weathering also tends ultimately to give a kaolinitic end-product (17), although the formation of montmorillonite has been reported on the basis of electron microscope examination (18). The artificial breakdown of the mineral in the laboratory also gives a product with the composition of kaolinite (19, 20, 21, 22), although in this case, if the treatment is sufficiently vigorous, the end-product consists of white flakes of amorphous silica (23, 24, 25). Under the anaerobic conditions obtaining on the sea-floor, glauconite appears to form from biotite (26, 27), but whether this is an ultimate product or merely a stage in the process of breakdown is not clear.

The corresponding changes taking place in the weathering of muscovite have also been examined (16, 28, 29), and although there is no marked colour change in this case, the chemical composition as with biotite tends towards that of kaolinite. It is not known what intermediate minerals, if any, are formed in the weathering of the heptaphyllite. The relative rates of decomposition of muscovite and biotite in the soil appear to be about the same (16), and in spite of some disagreement (6), it is probable that both are among the most resistant of all rock-forming minerals to weathering (21, 30, 31).

SOURCE OF THE MATERIAL.

Most of the weathered biotite flakes examined were obtained from a soil profile in Glen Buchat, Aberdeenshire. The depth of the profile excavated was about 100 cm., and the distribution of flakes of decomposed mica appeared to be fairly even both as regards frequency and range of colour in the various layers of the profile. The very small grain-size of most of the weathered flakes made it difficult to obtain pure samples of uniform material, and attention was therefore concentrated on collecting the few comparatively large flakes which were available. The majority of the flakes obtained were about 1 mm. square and perhaps 0.1 mm. thick, but one or two flakes about ten times this size were found. Various shades of brown and yellow were noted, most of them with a glistening lustre although a few were dull and coated with a brown iron oxide, later shown to be goethite.

A sample of the fresh biotite was obtained from a nearby outcrop of bed-rock—a hypersthene-gabbro in which local segregations of biotite occasionally occur. One of these segregations exposed in a small gully excavated by rain-water was also sampled. This material, which consists of dark brownish crystals showing a yellow lustrous appearance when broken along the cleavage planes, appears to be highly weathered, and crumbles readily in the hand.

EXPERIMENTAL.

X-ray data.—A broad separation of the profile material on the basis of colour was made into three main groups consisting of: (a) glistening yellow flakes; (b) duller buff-coloured flakes; and (c) dull brownish flakes heavily coated with goethite. Representatives of the second and third groups were very scarce relative to group (a) which, although it consisted of a considerable range of shades of yellow, was not subdivided further after a preliminary X-ray examination had been made.

X-ray powder photographs of the fresh biotite and the weathered material from the gully differed in certain respects, particularly in the width of the strong basal reflection at 10 Å. This reflection, sharp in the fresh biotite, is notably broadened in the weathered material and its centre shifted from 10.0 Å. to 10.4 Å. The diffraction pattern of the glistening yellow flakes which make up the bulk of the profile material is rather similar to that from the gully; whereas the buff-coloured flakes give an additional basal reflection in the region of 14 Å. as well as at 10 Å.; and the dull brown material, after most of the goethite had been removed by washing in water, gave a sharp reflection at 14 Å. and no indication of one at 10 Å.

On the basis of these results the various stages were placed provisionally in the order:

Order of Weathering.	Description.
0	Fresh black biotite.
1	Dark brown flakes from gully.
2	Glistening yellow flakes from soil profile.
3	Dull buff flakes from soil profile.
4	Dull brownish flakes from soil profile.

The changes taking place in the shapes and positions of the second-order basal reflections during the weathering process are shown in fig. 1, and the complete diffraction data are listed in table I.

TABLE I. X-ray powder data on fresh and weathered biotites from Glen Buchat, Aberdeenshire.

1.		2.		3.		4.		5.		6.	
<i>d.</i>	<i>I.</i>										
—	—	—	—	—	—	13.7	4d	13.6	6	14.0	10
10.0	10	11.0	8	10.9	8	11.8	6d	11.8	3d	—	—
—	—	9.8		9.8		9.8		9.5		—	
—	—	—	—	—	—	—		7.0		1d	7.0
4.60	3	4.56	4	4.57	4	4.57	5	4.56	4	4.56	4
4.23	2	4.23	2	4.17	2	4.17	10	4.15	10	4.15	7
3.70	0.5	3.69	1	3.69	1	—	—	—	—	—	—
3.53	0.5	—	—	—	—	—	—	3.53	1	3.50	4
3.34	10	3.35	10	3.36	10	3.36	4d	3.37	2	3.34	5
3.02	0.5	—	—	3.03	0.5	—	—	—	—	—	—
2.80	0.5	2.82	0.5	—	—	—	—	—	—	2.80	1
2.73	2	—	—	2.73	2	2.68	4	2.69	4	2.69	2
2.63	8	2.61	5	2.61	10	2.62	6	2.62	6	2.62	2
2.51	3	2.52	2	2.51	1	—	—	—	—	2.53	2
2.44	6	2.43	4	2.42	8	2.43	8	2.44	8	2.44	5
—	—	—	—	—	—	—	—	—	—	2.37	2
2.31	1	—	—	—	—	—	—	—	—	—	—
2.27	2	2.27	2	2.28	2	2.24	3	2.24	4	2.25	2
2.23	1	—	—	2.23		—	—	—	—	—	—
2.175	4	2.17	2	2.16	6	2.17	4	2.17	4	2.19	2
2.119	0.5	2.11	0.5	—	—	—	—	—	—	—	—
2.002	4	1.99	2	2.00	6	2.00	2d	2.00	2d	2.01	0.5d
1.909	2	—	—	1.92	2	1.92	0.5d	—	—	—	—
1.860	1	—	—	—	—	—	—	—	—	—	—
1.810	2	1.81	4	1.80	4	1.79	2	1.80	2	1.81	0.5d
1.741	1	—	—	—	—	—	—	—	—	—	—
1.716	2	—	—	—	—	1.712	5	1.715	5	1.713	4
1.672	6	1.67	3	—	—	1.686	1	—	—	—	—
1.541	8	1.532	6	1.526	10	1.533	5	1.534	5	1.532	4
1.524	2	—	—	—	—	—	—	—	—	—	—
1.501	1	—	—	1.505	1	1.502	1	1.505	2	—	—
1.476	2	—	—	—	—	—	—	—	—	—	—
1.463	0.5	—	—	1.462	2	—	—	1.468	0.5	—	—
—	—	—	—	—	—	1.446	0.5d	1.450	0.5	1.449	1
1.435	2	—	—	—	—	—	—	—	—	1.415	1
1.363	4	1.370	2	1.35	2	—	—	—	—	1.373	0.5
1.330	3	—	—	—	—	—	—	—	—	—	—
—	—	1.322	1	1.322	2	—	—	—	—	1.316	1d
1.313	3	—	—	—	—	—	—	—	—	—	—
1.304	4	—	—	—	—	—	—	—	—	—	—
1.284	1	—	—	—	—	—	—	—	—	—	—
1.275	2	—	—	—	—	—	—	—	—	—	—
1.255	2	—	—	—	—	—	—	—	—	—	—

d in Ångström units; *I*, relative intensity estimated visually; Fe- $K\alpha$ radiation; goethite lines present in columns 4, 5, and 6; d=diffuse.

1, fresh biotite. 2, first stage of weathering. 3, second stage of weathering. 4, third stage of weathering. 5, as 4, saturated with glycerol. 6, fourth stage of weathering.

It appears that the first and second stages of weathering are structurally not greatly different from the fresh biotite, since their diffraction patterns differ from it chiefly in respect of the sharpness of the basal reflections. At the same time, considerable colour changes have taken place, from black through dark brown to various shades of yellow, the most extreme examples of which, viz. bright golden-yellow and very pale yellow, give almost identical powder patterns and hence are grouped together in the second stage.

The dull buff-coloured flakes representing the third stage show a diffuse reflection at 14 Å. in addition to the 10 Å. When this material is mixed with glycerol, the 14 Å. reflection becomes appreciably stronger relative to the 10 Å., and weak higher orders appear at 7 Å. and 3.5 Å. This reaction is very interesting because it suggests that *single* layers of glycerol molecules are in certain cases capable of entering between the layers of a 'double-layer' type of mineral (cf. the reaction of glycerol with montmorillonite and halloysite (32)). The buff flakes of the third stage are always associated with goethite, which occurs between the cleavage planes of the flakes and appears when an apparently clean flake is broken along the cleavage.

In the fourth stage the goethite also occurs as a heavy coating on the outsides of the flakes, increasing their apparent size considerably. The flakes of this stage show no sign of a 10 Å. reflection and the appropriate diagnostic tests (33) indicate that the 14 Å. reflection is due to vermiculite. There is no sign of chlorite. None of the flakes examined show any

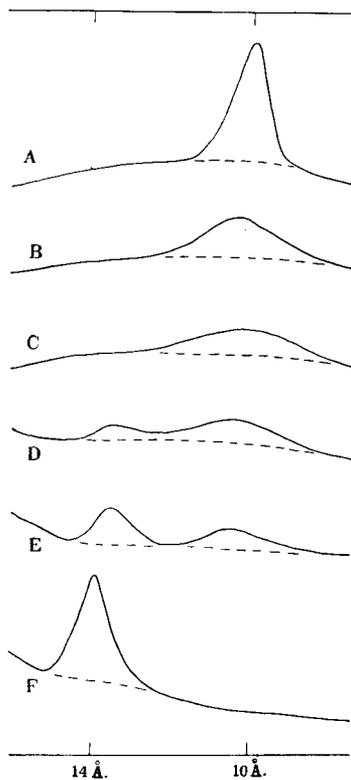


FIG. 1. The shapes of the 10 Å. and 14 Å. reflections at various stages during the weathering of the Glen Buchat biotite. A, fresh biotite; B, first stage of weathering; C, second stage of weathering; D, third stage of weathering; E, third stage after treatment with glycerol; F, fourth stage of weathering (vermiculite).

further increase in basal spacing beyond about 14.2 Å., and it is probable that, if decomposition proceeds farther, it consists in the destruction of the double-layer type of lattice, perhaps with the formation of kaolinite which is present in the clay fractions of the soil.

Optical data.—The α indices of the fresh and weathered biotites were determined by Lindberg's method (34).¹ The indices of each individual stage of the weathering were found to be somewhat variable, due to the degree of alteration to which a particular flake or portion of a flake had been subjected. Not only do different flakes from an apparently uniform sample show variations, but the outside edge of a flake may give a slightly lower value than the core. Data are listed in table II and show that the process of weathering is marked by a general reduction in the indices.

TABLE II. Optical data on the fresh and weathered biotites.

	α .	β, γ .	$\gamma - \alpha$.
Fresh biotite	1.621	1.678	0.057
First stage of weathering	1.624	1.679	0.055
Second stage of weathering	1.598	1.644	0.046
Third stage of weathering	—	1.635	—
Fourth stage of weathering	—	1.630	—

Attention may also be drawn to the difference in refractive index between the first and second stages which is notable in view of the similarity of their powder patterns. A slight increase in the size of the optic axial angle occurs in the first stage of weathering, but thereafter the angle reverts to the size of that in the fresh biotite which is small. Associated with these changes, there is a constant diminution in the definition of the interference figure which is consistently biaxial negative with the acute bisectrix nearly normal to the cleavage planes, as in biotite.

Chemical and physical data.—The data reported in this section are restricted by the scarcity of the profile material. Samples of the fresh biotite and the weathered material from the gully were purified by hand-picking under a lens followed by electromagnetting, after which the material appeared quite pure under the microscope, and X-ray photographs of the analysed samples, over-exposed so as to show up any very faint lines, gave no indication of any of the likely contaminants. The chemical analyses of these samples are given in table III, and so far as they go show trends consistent with those reported by previous

¹ This method consists in sprinkling a number of grains on to sodium silicate in a tacky condition, allowing to dry, and then using those which chance to stand vertically for the measurement of the α index.

workers. Thus, the high proportion of ferrous oxide in the fresh material is almost completely changed to ferric at the first stage; and in addition, magnesia and potash show significant losses balanced by gains in silica

TABLE III. Chemical analyses of the fresh and weathered biotites.

	1.	2.
SiO ₂	34.97	38.18
TiO ₂	5.64	4.79
Al ₂ O ₃	15.30	15.88
Fe ₂ O ₃	2.81	18.88
FeO	21.23	2.99
MnO	0.16	0.15
MgO	8.83	4.92
CaO	1.09	1.22
Na ₂ O	0.27	0.46
K ₂ O	7.17	3.66
H ₂ O+	3.03	6.70
H ₂ O-(105° C.)	0.31	2.38
	100.81	100.21

1. Fresh biotite, Glen Buchat, Aberdeenshire; anal. G. Robertson.
2. First stage of weathering; anal. G. Robertson.

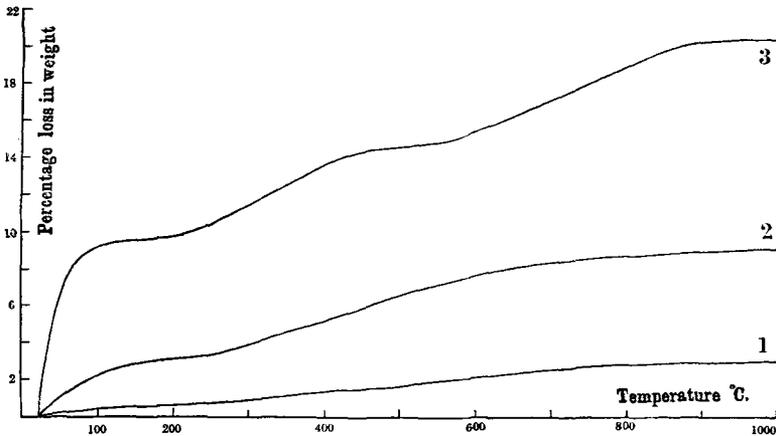


FIG. 2. Cumulative dehydration curves of: 1, fresh biotite, Glen Buchat; 2, first stage of weathering; 3, vermiculite from West Chester, Pennsylvania.

and water. The dehydration data (fig. 2) emphasize the difference in water content between the fresh and weathered biotites, and the curve for a true vermiculite (33) is included for purposes of comparison.

The cation exchange capacity of the fresh biotite (9 milliequivalents per 100 g.) increases on weathering. At the first stage the value is

23 milliequivalents per 100 g., and the very high capacity of hydrothermal vermiculite (33, 36, 40) indicates that this figure probably continues to increase as the weathering proceeds.

Exfoliation tests were made on a number of the weathered flakes both by heat (4) and hydrogen peroxide (37), the latter being much more effective in most cases. The brown flakes of the first stage exfoliated to about ten times their original size with the peroxide treatment and turned a golden-yellow colour. This colour change appears to be wholly due to the mechanical prizing apart of the flakes since there is no associated change in X-ray diffraction pattern or optical properties. The subsequent stages expanded hardly at all with heat or peroxide treatments, and it is probable that they had already largely exfoliated in the soil. The range of yellows represented in stage 2 of the weathering appears to be due at least partly to the degree of exfoliation undergone by the individual flakes; and after exfoliation the dark-brown flakes of the 'first stage' become very similar in appearance to those of the 'second stage'. Ruthruff's (38) exfoliation test for hydrobiotite gave negative results in all cases.

DISCUSSION.

At Glen Buchat the great majority of the weathered biotite flakes in the soil profile are at a relatively early stage of decomposition. A considerable degree of stability at this (the second) stage of soil weathering is suggested by its apparent similarity to the material of Denison, Fry, and Gile (16), who, in a chemical examination of eleven soil profiles, found biotite to be altered to about the same extent in all cases with a potash content approximating 4%. At Glen Buchat X-ray diffraction enables the process of weathering to be traced beyond this point to the development of a vermiculite and perhaps, by further breakdown, to kaolinite. The final stage, however, is less certain, and in this section we shall consider the process only as far as the vermiculite stage.

It appears from the chemical data that magnesia at first tends to be lost as the weathering proceeds and this is borne out to some extent by other investigations (15, 16). Most published analyses of vermiculites (7) show a high percentage of magnesia but refer exclusively to vermiculites produced by hydrothermal agencies rather than by weathering in the soil. It has not been possible to determine directly whether the soil vermiculite also contains large amounts of magnesium or whether the octahedral positions are occupied by other ions. The large amounts of goethite associated with the advanced stages of decomposition indicate

that much of the iron after its oxidation is removed from the crystal lattice, and replacement of these ions by magnesium from soil solutions rich in this constituent is not unlikely (39).

The (060) reflection on the diffraction pattern of the soil vermiculite, moreover, occurs at 1.53 Å. which is generally regarded as being in the trioctahedral¹ region, and this seems to indicate that the octahedral positions are occupied by divalent ions such as magnesium. The position of the second-order basal spacing of the soil vermiculite is also of interest since it has been shown that the replacement of exchangeable magnesium by other ions in hydrothermal vermiculite may alter the structure of the water layers and hence the height of the unit cell (33, 36, 40). In the diffraction pattern of the soil vermiculite, the occurrence of this reflection at 14 Å. is compatible with the occupation of the exchange positions by magnesium ions although certain other ions, e.g. calcium, cannot be ruled out.

Although these considerations seem to leave the way open to acceptance of the chemical identity of soil and hydrothermal vermiculites, the refractive indices of the two types differ appreciably. Thus the values of the Glen Buchat vermiculite (table II) are significantly higher than those given by Shannon (35) (γ 1.545–1.573) for hydrothermal vermiculite, and seem to indicate a difference in composition between soil vermiculite and vermiculite of hydrothermal origin. It is plain that the final answer to this question must await direct chemical evidence.

Initially, the process of weathering appears to lie in the replacement of the most accessible potassium ions by water molecules which are of much the same ionic size. As this replacement continues towards the interiors of the flakes, the binding between the silicate layers loosens and the spacing of the basal planes increases slightly. Concomitant changes within the silicate layers, such as oxidation of ferrous iron, substitution of hydroxyl for oxygen, and loss of magnesium, take place in order to balance the lattice charges. In this condition, which corresponds to the 'second stage' of weathering, the flakes appear to be fairly stable; but eventually further leaching of potassium from interlayer positions together with replacement of iron in the interiors of the silicate layers by magnesium or other ions from percolating waters reduce the attractive interlayer forces and permit the entry of double layers of water molecules. This latter phenomenon spreads through the crystal layer by layer,

¹ Following the usage of Stevens (41), trioctahedral refers to layer minerals in which all possible octahedral positions are filled, and dioctahedral to those in which only two out of three such positions are occupied. In dioctahedral minerals the (060) reflection usually occurs at about 1.50 Å.

giving first a mixed-layer structure in which increasing numbers of layers become of the vermiculite type.

Just prior to the expansion of an individual layer by natural processes in this way, it seems that glycerol molecules can penetrate to some extent between adjacent silicon-oxygen surfaces and cause expansion. In the case of montmorillonite, glycerol molecules can displace water molecules from between the silicate layers and form a double glycerol layer (32); but in weathered biotite the expansion does not go beyond about 14 Å., so that only one layer of glycerol molecules can be accommodated between a pair of silicate layers. The shortage of material again makes itself felt at this point where the opportunity of further investigation would be particularly welcome; possible alternative lines of approach are, however, available, since a similar phenomenon is found to occur in NH_4 -saturated vermiculite (33) and in the 'vermiculite' of the soil clays.

The profile examined is of an intermediate drainage type in which the vertical leaching process is probably fairly active throughout the year. In the clay fractions of the soils of the area, breakdown of a biotite-like clay mineral under free drainage conditions proceeds as above to a vermiculitic product. In very wet conditions, however, i.e. under a permanent or fluctuating water-table, it is not certain that the end-product would be the same, since in the clay fractions of very poorly drained or wet soils of basic origin the weathering of the 'clay biotite' proceeds directly to the formation of a mineral with a freely expanding lattice like montmorillonite,¹ although in other respects its diffraction pattern is similar to the vermiculitic product of the drier soils.

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¹ A mineral taken from the sand fraction of a Banffshire soil and which may be of this kind has been examined by Drs. Muir, Mitchell, Hart, and MacEwan of the Macaulay Institute (unpublished work).

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