VERMICULITE AS A **MEMBER IN MIXED-LAYER MINERALS** B_{γ}

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ABSTRACT

X-ray diffraction and differential thermal analysis data are presented for colloid fractions from a soil and shales containing a vermiculite and a montmorillonoid; a chlorite and a vermiculite-chlorite mixed-layer mineral; and a montmorillonoid and a vermiculile-chlorite mixed-layer mineral. The minerals were identified by heating the clay fractions at various temperatures and by treating them with ethylene glycol and/or ammonium chloride.

INTRODUCTION

One of the first references made to a type of "mixing" of clay minerals not consisting of discrete particles but of an interstratification of parallel layers of separate minerals was in 1934 by Gruner, who described hydrobiotite as an interlayering of approximately equal amounts of mica and vermiculite_ Since that time the existence of a vermiculite-chlorite-like structure has been proposed by Hendricks and Jefferson (1938) and discussed by Barshad (1948), Mathieson and Walker (1952), and Stephen (1952). Stephen described both micavermiculite and chlorite-vermiculite mixed-layer minerals occurring in the soils of the Malvern Hills.

A survey of the mineralogical composition of the colloid fractions from the soils of the semi-arid and desert regions in South Africa (according to the classification of van der Merwe, 1941) indicated the rather common occurrence of mica-vermiculite mixed-layer minerals in the lower levels of the soil profiles (van der Merwe and Heystek, in press). In the upper levels illite and illite-montmorillonoid mixed-layer minerals predominated.

In this paper x-ray diffraction and D.T.A. data are presented for a chloritevermiculite mixed-layer mineral occurring in a shale deposit used by a buildingbrick manufacturer, and for comparison reference is made to a vermiculite from a weathered diabase_

It is hoped that the preliminary investigation of these shales will be continued at a later date to obtain additional and more complete data on their clay minerals and specifically the chlorite-vermiculite mixed-layer mineral.

VERMICULITE IN WEATHERED DIABASE

A diabase occurring between a shale and a quartzite layer in the Pretoria Series was found to be weathered to a great depth, and samples were collected at one-foot intervals down to 50 feet. Examination of the clay fractions $\langle \langle 2\mu \rangle$ was carried out by means of x-ray diffraction using filtered cobalt radiation and a Philips high-angle diffractometer. In addition differential thermal curves

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were obtained on all the samples; only one curve (clay fraction from sample collected at 50 feet) is recorded in Figure 1.

The following clay minerals were identified in the clay fractions throughout the profile:

kaolinite also present
The clay fraction from the soil sample collected at 50 feet contained essentially vermiculite as shown by the basal spacing of 14.4 A on the x-ray diffraction diagram (Fig. 2) with some montmorillonite (a small peak appeared at 18.6 A in addition to one at 14.2 A when the sample was treated with ethylene glycol). The presence of vermiculite was further verified when the 14 A peak was shifted to 11.2 A after the sample was boiled in a normal ammonium chloride solution (Walker, 1951). When this latter sample was also treated with ethylene glycol a peak due to montmorillonite appeared at 19 A. Chlorite was eliminated when the sample was heated at 300 $^{\circ}$ or 500 $^{\circ}$ C before being x-rayed; the 14 A basal peak shifted to 9.57 A. The 7.2 A peak is therefore most probably due to kaolinite.

A CHLORITE~VERMICULITE MIXED-LAYER MINERAL

Amongst a group of clays and shales used as raw material for building brick and that were investigated for their ceramic properties and mineralogical composition were some shales that melted at a low temperature without bloating. Fourteen samples of these shales collected near Germiston were fractionated, and the minerals present in the clay fraction $\left(\langle 2\mu\right)$ determined. The x-ray diffraction data from two of these samples, namely Primrose no. 1 and Primrose no. 5, are represented in Figure 3 and their differential thermal curves in Figure 1.

The x-ray reflections of the clay fraction prepared from the Primrose no. 1 sample give a chlorite-type diagram with reflections at 7.1 A and 14.3 A and a high basal spacing at 30 A. The spacings are not affected by treatment with ethylene glycol; this excludes the possibility that a montmorillonoid or "swelling chlorite" may be present. After the sample is heated at 500° C and also 600° C the 14.3 A peak is replaced by two peaks at 13.9 A and 11.8 A indicating minerals of a chloritic and a chloritic-vermiculitic mixed-layer nature (approximately equal amounts of chlorite, 14 A, and vermiculite, 9.5 A, interlayered) respectively. Furthermore, when an unheated sample was boiled in a normal ammonium chloride solution and then x-rayed, peaks appeared at 14.2 A (chlorite) and 12.6 A (chlorite-vermiculite mixed-layer, 14 \overline{A} -11.2 A).

The data indicate that essentially two crystalline phases are present in the Primrose no. 1 sample, namely a chloritic phase and a regular interlayering of approximately equal amounts of a chlorite and a vermiculite.

The basal x-ray reflections of the Primrose no. 5 sample are similar to those of Primrose no. 1 except for a smaller 7.1 A peak. After this material was heated to 500° or 600° C, the chlorite was eliminated and the 14.3 A peak disappeared. The strong basal reflections of 24 A and 11.85 A can again be interpreted as

FIGURE 1. - D.T.A. curves of clay fractions containing a vermiculite-chlorite mixed-layer or vermiculite clay mineral.

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FIGURE 2. - Basal x-ray reflections of a vermiculite and a montmorillonoid in a weathered diabase.

FIGURE 3. - Basal x-ray reflections of a clay fraction containing a vermiculite-chlorite mixed·layer mineral.

representing a regular interlayering of about equal amounts of chloritic (14 A) and vermiculitic (9.5 A) components. This also explains the presence of the 12.6 A peak on the x·ray diffraction pattern of material that was treated with ammonium chloride.

The peak occurring at 9.5 A after the samples are heated at 500° to 600° C is consistent with data for vermiculites but is most probably due to a montmoriIlonoid. The evidence for the presence of the latter is the peak at 14.2 A after the sample was boiled in an ammonium chloride solution and the peak at 19 A after the material was treated with ethylene glycol.

In the Primrose no. 5 sample, the predominant minerals seem to be a montmorillonoid and a chlorite-vermiculite mixed-layer mineraL

The other samples collected at the shale pits of the building-brick manufacturer at Primrose near Germiston contained various combinations of chloritic, montmorillonitic, chlorite-vermiculite mixed-layer, and mica minerals. It is hoped that further sampling may make it possible to obtain relatively pure specimens of the mixed-layer mineral for detailed study.

During this investigation samples no. 1 and no. 5 were heated to temperatures of 300° , 400° , 500° , 600° , and 700° C for periods of as much as 24 hours before x-ray examination; after these samples were heated to 300° and 400° C very diffuse x-ray diffraction patterns were obtained (Fig. 3). This was not true for untreated samples or samples heated to 500° , 600° , or 700° C. At the moment it can only be proposed that this diffuseness may be due to the very rapid rehydration shown by dehydrated vermiculite when exposed to normal atmospheric conditions even after heating to 500° C as reported by Walker (1951). This, however, was not true for the vermiculite fractionated from the weathered diabase, where sharp x-ray diffraction patterns were obtained from samples heated to temperatures as low as 300° C (Fig. 2).

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