

USE OF THE (02,11) X-RAY DIFFRACTION REFLECTIONS TO IDENTIFY CLAYS

Key Words—Heat treatment, Identification, Kaolin, Mica, Smectite, X-ray powder diffraction.

During a study of local soil clays, we obtained X-ray powder diffraction traces that were characterized by the absence of all basal reflections, but with the prism reflection (02,11) corresponding to a spacing of about 4.47 Å, as shown in trace 1, Figure 3. The absence of basal reflections makes the interpretation of the traces very difficult if not impossible. As is known (Grim, 1968), however, the (02,11) reflections of chlorite and vermiculite are at larger spacings than those of kaolin-, mica-, and smectite-group minerals, giving rise to the possible use of these reflections for identification purposes. Thus, if the observed spacing is less than 4.47 Å, chlorite and vermiculite could be eliminated from consideration, leaving only kaolin-, mica-, and smectite-group minerals to be considered.

The kaolin-group minerals break down on heating for 1 hr at 550°C (Brindley, 1972). Therefore, a reflection at about 4–5 Å would disappear on heating if it belongs to a kaolin-type structure. If a treatment could be devised that would affect this prism reflection differently for the two remaining groups, smectites and

micas, a useful method for clay identification would be generated.

We have found that heating a clay sample well mixed with 10% sodium carbonate for 2 hr affects none of the reflections of hydrous mica, but destroys those of montmorillonite. We have noted this behavior for 3 montmorillonites and 2 illites. The diffraction traces, obtained using Ni-filtered $\text{CuK}\alpha$ radiation for a local montmorillonite from Cerro Bandera (Peinemann *et al.*, 1972) and the Fithian illite are shown in Figures 1 and 2.

The different treatments received by the clays are described in the legends of the figures. It may be seen from the traces that the normal reflections of montmorillonite and illite are unaffected by heating the sample with 10% CaCO_3 at 550°C. Adding NaCl to the CaCO_3 , however, reduced the intensity of the reflections of montmorillonite. Furthermore, when the clay

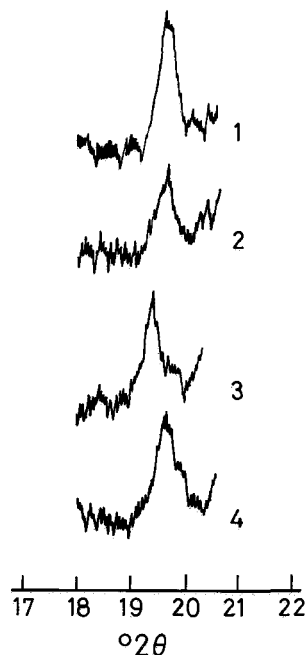


Figure 1. X-ray powder diffraction traces of Fithian illite: (1) calcined at 550°C, (2) calcined in the presence of Na_2CO_3 , (3) as in (2) but then washed with 0.1 M HCl, and (4) calcined in the presence of CaCO_3 ($\text{CuK}\alpha$ radiation).

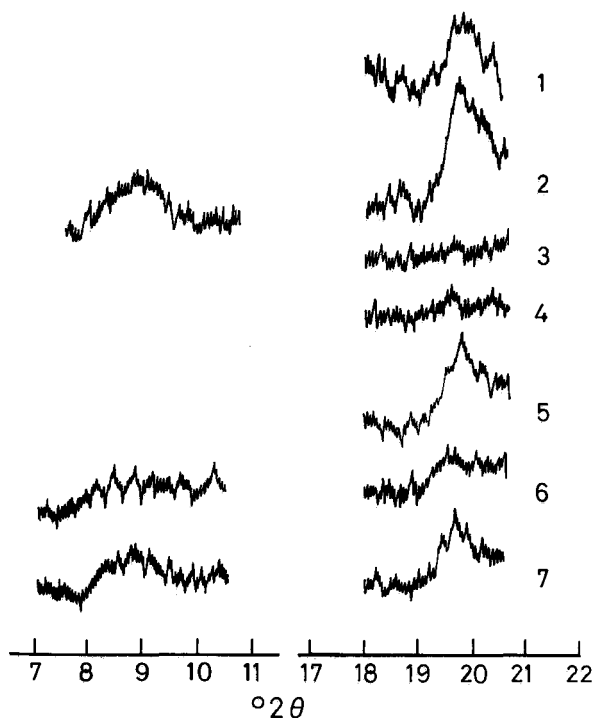


Figure 2. X-ray powder diffraction traces of Cerro Bandera, Argentina, montmorillonite: (1) air dry, (2) calcined at 550°C, (3) calcined in the presence of Na_2CO_3 , (4) same as in (3) but then washed with 0.1 M HCl, (5) calcined in the presence of CaCO_3 , (6) calcined in the presence of 10% CaCO_3 and 10% NaCl, and (7) same as in (6) but then washed with 0.1 M HCl ($\text{CuK}\alpha$ radiation).

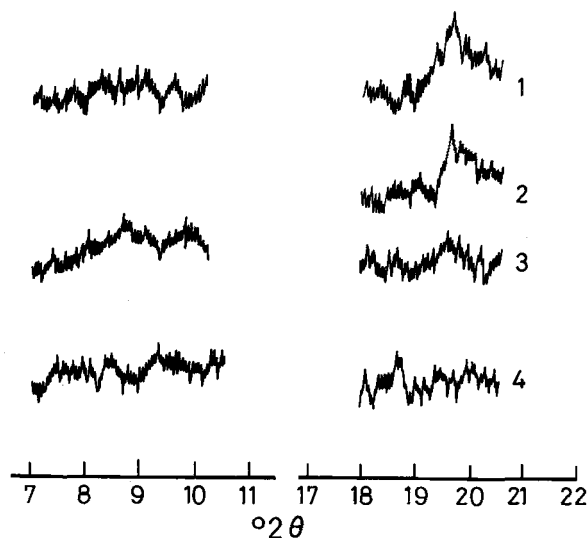


Figure 3. X-ray powder diffraction traces of an Argentinian soil clay: (1) air dry, (2) calcined at 550°C, (3) calcined in the presence of CaCO_3 and then acid washed, and (4) calcined in the presence of Na_2CO_3 ($\text{CuK}\alpha$ radiation).

was washed several times with 0.1 M HCl and then with water, the montmorillonite reflections regained their normal intensities, indicating that the products of the heat treatment probably interfered with the diffraction of the X-rays and were removed by the acid treatment. The acid treatment, however, was ineffective for the montmorillonite receiving a Na_2CO_3 -heat treatment suggesting that the montmorillonite struc-

ture was destroyed by heating at 550°C in the presence of Na_2CO_3 .

As shown in Figure 3, the reflections of the soil clay were not affected by calcination at 550°C, but disappeared as a result of the sodium carbonate treatment, indicating that this soil clay likely has a montmorillonite-type structure. In addition, the CaCO_3 -heat treatment (see trace 3, Figure 3) produced a reflection at 10 Å, similar but less intense than that found in the calcined montmorillonite (trace 2, Figure 2).

The above method may be especially useful for identifying clays which show few reflections in their XRD traces. The one spacing which usually is present when others are not is that due to the (02,11) reflections (Sudo and Hayashi, 1956).

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