

# Identification of Kaolinite and Metahalloysite in Tropical Soils

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Lateritic soils develop under tropical conditions of high temperature and high precipitation that is often seasonal. The primary silicates are quickly broken down, and the extensive precipitation causes the quick removal in solution of any alkalis and alkaline earths. Aluminum and iron tend to be enriched in soil horizon, and the influence of material rock frequently disappears. The organic matter is very rapidly decomposed so that it does not accumulate. It is well known that dominant clay mineral of lateritic soils is kaolin minerals, and other accessory minerals are gibbsite and iron oxide minerals. Which species of kaolin minerals occurs in such soils is interesting and important in studying the properties and the genesis of tropical soils, consequently.

We are often confronted with some difficulties in identifying clay minerals in soils, especially, differentiation of (1) vermiculite from montmorillonite, (2) kaolin minerals from chlorite, and (3) kaolinite from metahalloysite. Walker<sup>1)</sup> differentiated vermiculite from montmorillonite. Wada et al.<sup>2),3)</sup> presented the differentiation of kaolin minerals from chlorite by intersalation with potassium acetate. Oinuma et al.<sup>4)</sup> identified kaolin minerals

and chlorite in sedimentary rocks by the combination of several experimental techniques.

The author<sup>5)</sup> attempted to work on the differentiation of kaolinite from metahalloysite both of which have 7 Å basal spacing, by applying electron microscopy, X-ray diffraction method, infrared absorption spectroscopy, and thermal analysis. Kaolinite and metahalloysite samples as the minerals from geological deposit were the fraction less than 2 $\mu$  of Georgian Kaolin and Hongkong Kaolin, respectively. Soil minerals were obtained from the clay fraction of the soils listed in Table 1.

## Electron microscopy

Electron micrograph of kaolinite and metahalloysite obtained from geological deposit is shown in Plates 1 and 2, respectively, and that of the minerals occurring in soils is represented in Plates 3 and 4. These electron micrographs were taken by direct magnification of 10,000 at 75 kV after the specimen was suspended in distilled water and placed on a mesh covered with thin collodion film.

The shape of kaolinite and metahalloysite particle is hexagonal plate and tubular, respectively (Plates 1 and 2), which is well

Table 1. Soil samples

	Locality	Soil type and horizon	Texture	Parent material	Land utilization
Kaolinite	Ammy, Southern Vietnam	Diluvial Soil, Ap (0~30 cm)	SiL	River deposit	Paddy field
Metahalloysite	Kedung halang, Bogor, West Java	Reddish-brown Latosol, Blg (27~46 cm)	HC	Old fan deposit	Paddy field



Plate 1. Electron micrograph of kaolinite from geological deposit

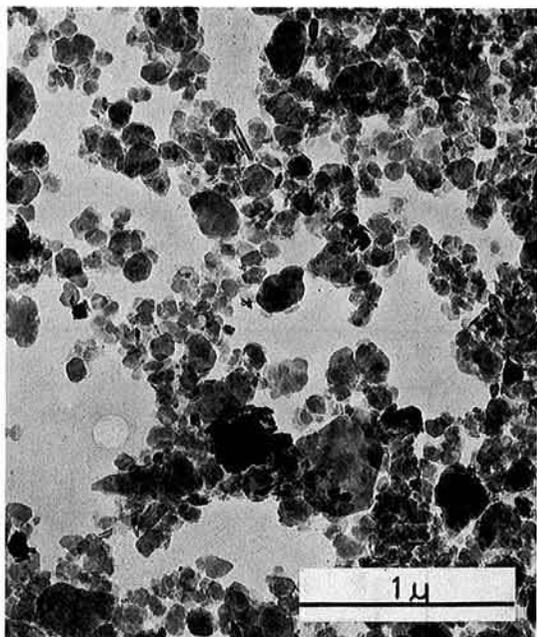


Plate 3. Electron micrograph of kaolinite from soil



Plate 2. Electron micrograph of metahalloysite from geological deposit.

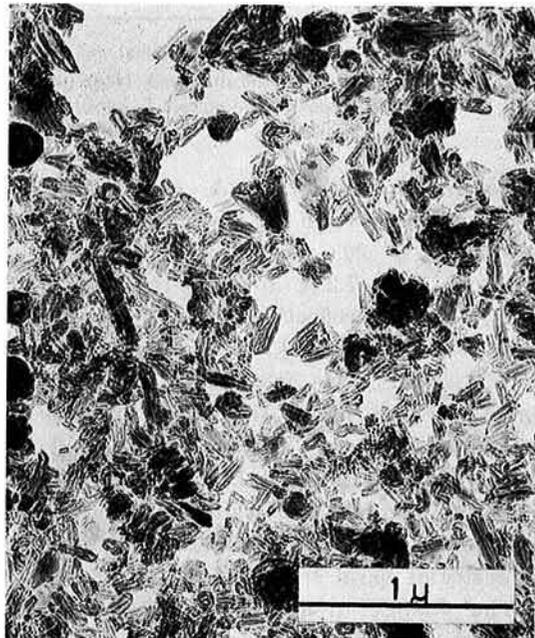


Plate 4. Electron micrograph of metahalloysite from soil

known. On the other hand, the particles of soil minerals also show same shape although their size are generally smaller than the formers (Plates 3 and 4). A little amount of onion-

like particles coexisted with metahalloysite particles in Plate 4 is halloysite.

Electron microscopy is surely very effective to differentiate kaolinite from metahalloysite

in soils, but much skill is needed to prepare specimen and to take electron micrograph properly.

### X-ray diffraction method

X-ray diffraction pattern of kaolinite and metahalloysite by powder method is shown in Fig. 1. These were obtained with Cu-K $\alpha$

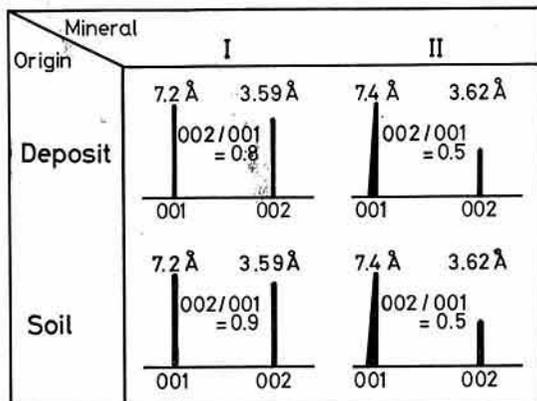


Fig. 1. Position and intensity of basal spacing of kaolin minerals obtained from geological deposits and soils. I, kaolinite; II, metahalloysite

radiation at 40 kV, 20 mA, slit system of 1° (DS, SS)–0.3 mm (RS), and a scanning speed of 2° (2 $\theta$ )/min. Basal spacing of kaolinite was 7.2 Å for 001-reflection and 3.59 Å for 002-reflection, irrespective of deposit or soil mineral. The intensity ratio, I(002)/I(001) was 0.8 or 0.9 in kaolinite. On the other hand, the basal spacing of metahalloysite was 7.4 and 3.62 Å, and intensity ratio was 0.5 in both origin mineral. Kaolinite and metahalloysite could be differentiated by measuring the position and relative intensity of basal spacings.

The change of basal spacing of kaolin minerals in soils by hydrazine intercalation-intersalation is shown in Fig. 2. The reflection at 7 Å of soil kaolinite was hardly changed, while that of metahalloysite was shifted to 14 Å, by the treatment with hydrazine-potassium acetate. The basal spacing was shifted to 10 Å partially in kaolinite and com-

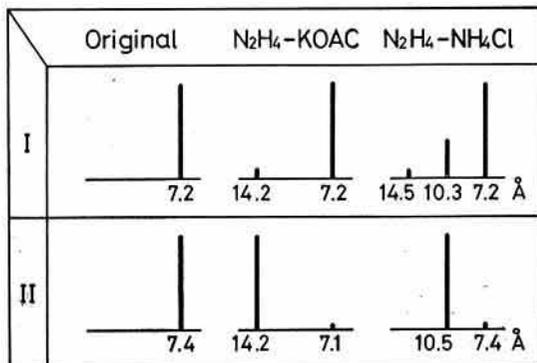


Fig. 2. Changes of 7 Å basal spacing of kaolinite and metahalloysite in soils by treatment with hydrazine-potassium acetate and -ammonium chloride. I, kaolinite; II, metahalloysite

pletely in metahalloysite with hydrazine-ammonium chloride system. Wada et al.<sup>3),6)</sup> already reported that kaolinite in deposit tends to resist against intersalation. This method is only effective to identify metahalloysite, consequently.

### Infrared absorption spectroscopy

The infrared absorption band associated with O–H stretching vibration of structural hydroxyl group in kaolin minerals appears in the range of wave number from 3,700 to 3,600 cm<sup>-1</sup>, and splits to three or four band in kaolinite and two in metahalloysite, irrespective of deposit or soil origin as shown in Fig. 3. Accordingly, kaolinite and metahalloysite can be differentiated by observing this infrared absorption band. Coexistence of some amount of gibbsite and 2:1-type minerals did not interfere the differentiation of kaolinite and metahalloysite by this method. Only one band associated with O–H stretching vibration of structural hydroxyl group in other clay minerals such as montmorillonite, vermiculite, mica and chlorite is situated near 3,620 cm<sup>-1</sup>, and the band of gibbsite that is frequently detected in lateritic soils splits to five bands at 3,630, 3,530, 3,450, 3,400 and 3,380 cm<sup>-1</sup>. In gibbsite, the band at 3,630 cm<sup>-1</sup> was not so strong, and only two

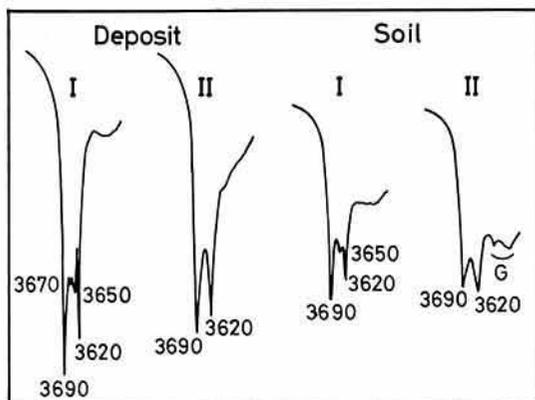


Fig. 3. Infrared absorption band associated with O-H stretching vibration of structural hydroxyl group in kaolin minerals obtained from geological deposits and soils. I, kaolinite; II, metahalloysite.

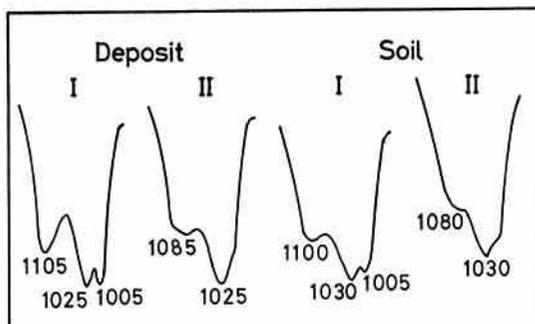


Fig. 4. Infrared absorption band associated with Si-O stretching vibration of kaolin minerals in geological deposits and soils. I, kaolinite; II, metahalloysite

bands at  $3,530$  and  $3,450\text{ cm}^{-1}$  were strong. The bands shown by G in Fig. 3 are associated with gibbsite.

The absorption band near  $1,000\text{ cm}^{-1}$  is associated with O-H stretching vibration of kaolin minerals, and splits to three bands in kaolinite and two in metahalloysite, irrespective of deposit or soil minerals (Fig. 4), which also would be applied to the differentiation of kaolinite and metahalloysite.

The infrared absorption spectrum was measured by tablet method with potassium bromide. The tablet was prepared by pressing a mixture of 1 or 0.5 mg of specimen with 200 mg of potassium bromide at  $400\text{ kg/cm}^2$ .

## Thermal analysis

It was difficult to differentiate kaolinite and metahalloysite by means of differential thermal analysis and thermogravimetry although these techniques are very effective to differentiate kaoline minerals from other clay minerals. kaolin minerals from other clay minerals.  $550^\circ\text{C}$  and a sharp exotherm near  $900^\circ\text{C}$  in differential thermal analysis. The weight loss of kaolin minerals in the temperature region from  $400$  to  $600^\circ\text{C}$  is about 14 per cent, and far larger than other clay minerals.

## Conclusion

The combination of X-ray diffraction method, infrared absorption spectroscopy, electron microscopy and hydrazine intercalation-intersalation is most effective to identify kaolinite and metahalloysite occurring in the clay fraction of soils. If it is difficult to employ all techniques mentioned above together, kaolinite and metahalloysite might be identified by combining two techniques which are easily available.

This method for the differentiation of kaolinite and metahalloysite is surely effective to be applied in the case of lateritic soils of which dominant clay mineral is kaolin minerals, but is hardly applicable to soils with large amount of 2:1-type minerals and others coexisting.

## References

- 1) Walker, G. F.: Differentiation of vermiculite and smectites in clays. *Clay Miner. Bull.*, **3**, 154-163 (1957).
- 2) Wada, K.: Lattice expansion of kaolin minerals by treatment with potassium acetate. *Amer. Mineral.*, **46**, 78-91 (1961).
- 3) Wada, K. & Yamada, H.: Hydrazine intercalation-intersalation for differentiation of kaolin minerals from chlorites. *Amer. Mineral.*, **53**, 334-339 (1968).
- 4) Oinuma, K., Kodama, H. & Kobayashi, K.: chlorite. *Nendo Kagaku (J. Clay Sci. Soc. Japan)*, **3**, 179-193 (1963).

- 5) Kitagawa, Y.: Identification of kaolinite and metahalloysite occurring in soils. *Nendo Kagaku (J. Clay Sci. Soc. Japan)*, 14, 90-97 (1974).
- 6) Wada, K.: Use of salt complex in differentiation of halloysite from kaolinite. *Nendo Kagaku no Shinpo (Abs. Adv. Clay Sci., Tokyo)*, 2, 263-272 (1960).