

# OCCURRENCE OF A TABULAR HALLOYSITE IN A TEXAS SOIL

*by*

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## ABSTRACT

A substantially homogeneous mineral fraction from a Katy soil profile in Fort Bend County, Texas, consists of a tabular kaolin group mineral with the physical and chemical properties characteristic of a nearly dehydrated halloysite, retaining only a small excess of water.

THE Katy soils, of which fine sandy loam is the principal type, are Planosols with thick A horizons. They occur on the interior margin of the Gulf Coast Prairie, 94° to 97° W. longitude, 29.5 to 30.5 N. latitude.

The clay for this study was obtained from a profile near Fulshear in the northern end of Fort Bend County near the geographic center of the area of the series occurrence, in southeastern Texas. Katy fine sandy loam and the closely associated Waller and Hockley soils are the principal soil series of the undulating interior part of the Gulf Coast Prairie, locally called the Hockley Prairie. The area of some 2 million acres which they comprise is largely cultivated. Katy fine sandy loam and closely associated Waller soils make up approximately 42,000 acres or 7.7 per cent of Fort Bend County. The total acreage of Katy fine sandy loam in Texas is estimated at 750,000 acres.

These soils have developed from slightly acid or weakly calcareous sandy clays of early Pleistocene age—Lissie formation. Though the clay material under study occurs throughout the profile, the crystallinity increases with depth; and for this reason, the clay fraction of the C1 horizon at a depth of 64–84 in. is chosen here for detailed description.

The fine sandy loam texture provides 32 per cent of  $< 2\mu$  fraction of which 2–0.2 $\mu$  material in the Pt shadowed photomicrograph in Plate 1A illustrates that quartz grains and clay mineral aggregates tend to have been evenly graded. It is presumed that the kaolinization of a component of this coarse clay fraction followed the accumulation of the sediment.

Dispersion of the whole soil permits collection of an abundant clay

fraction, with as much as 65 per cent of the  $< 2\mu$  material concentrated in the equivalent settling size fraction between 0.2 and  $0.1\mu$ . Plate 1B is a Pt shadowed electron micrograph from a concentrated dispersion of the  $< 0.2\mu$  fraction, and Plates 2A and 2B are, respectively, an unshadowed electron micrograph from a more dilute suspension and the electron diffraction diagram of a corresponding field. The frequent distinct hexagonal profiles and exclusively  $hk0$  type diffraction rings of Plate 2 establish the tabular morphology of the clay fraction. Lateral dimensions appear from the micrographs to be only about one magnitude greater than thicknesses, and the pseudohexagonal set of prism rings indicate a 060 spacing of  $1.50\text{\AA}$ .

The chemical analyses of the  $< 0.2\mu$  fraction are cited in Table 1. It approximates a molecular formula of  $2\text{SiO}_2 \cdot \text{R}_2\text{O}_3 \cdot 2.6\text{H}_2\text{O}$ . Firing to  $800^\circ\text{C}$  deepens the color of the clay fraction only moderately, and clearly

TABLE 1.—CHEMICAL ANALYSES OF  $< 0.2\mu$  FRACTION  
IN KATY FINE SANDY LOAM

	Air dry Wt. %	Cation Composition
H <sub>2</sub> O	16.63	
SiO <sub>2</sub>	42.79	4.00
Fe <sub>2</sub> O <sub>3</sub>	8.25	0.58
TiO <sub>2</sub>	0.88	
Al <sub>2</sub> O <sub>3</sub>	29.28	3.27
MgO	1.10	0.15
CaO	1.52	0.16
Total	100.45	
Base exchange capacity		60 meq/100 g.
CaO content		55 meq/100 g.

less than would be the case if the nearly 10 per cent Fe<sub>2</sub>O<sub>3</sub> were all free oxide. It is presumed that iron, much apparently in ferrous state, is a component of the clay crystallization. The determined base exchange capacity could only possibly be accounted for by significant presence of divalent ions to provide layer charge.

Oriented slide aggregate preparations, when examined by X-ray diffractometry, yielded records related exclusively to the layer thickness periods. They are typical for the mixed system resultant from random incidence of excess water layers modifying a fundamental 7 Å kaolin group mineral period. The condition is essentially that described by Brindley (1951, p.55) in his volume on *X-ray Identification and Structure of Clay Minerals*. For the air-dried state, the extraneous water provides a modification of the first order feature on the low angle side, and of the second order feature

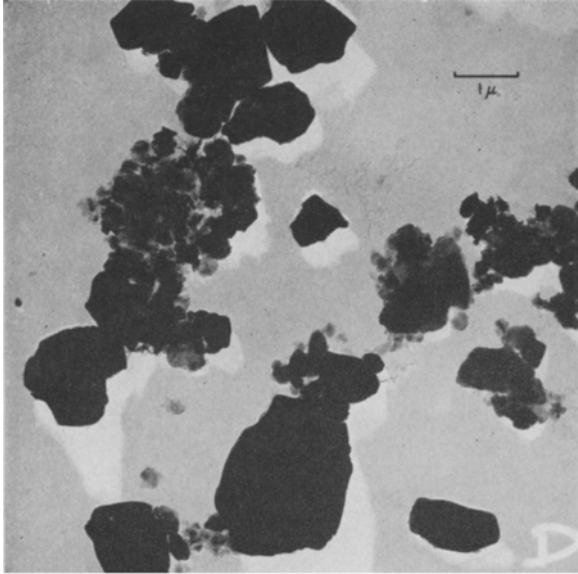


PLATE 1A.—Platinum shadowed electron micrograph of 2-0.2 $\mu$  clay from the Katy soil C1 horizon.

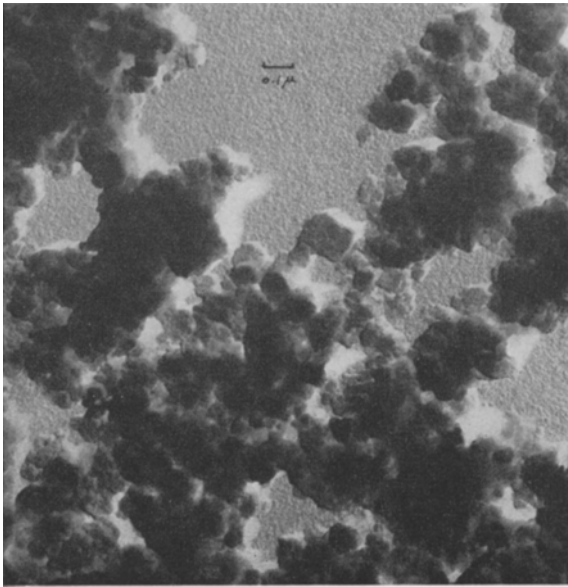


PLATE 1B.—Platinum shadowed electron micrograph of <0.2 $\mu$  fraction recovered from dispersion of the <2 $\mu$  clay.

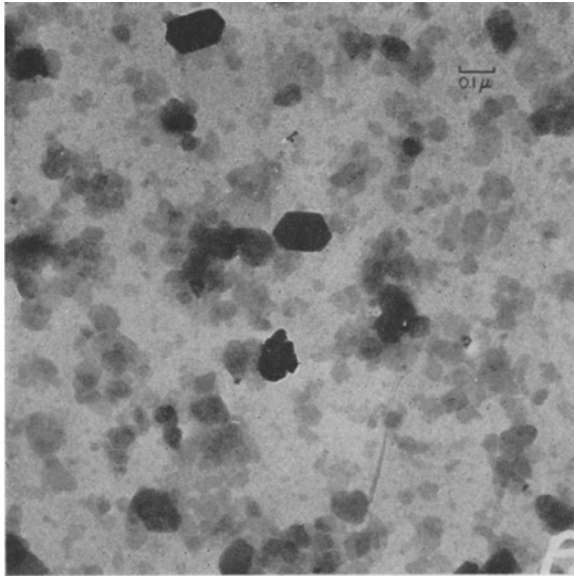


PLATE 2A —Unshadowed electron micrograph of  $<0.2\mu$  Katy soil fractions collected from a dilute dispersion.

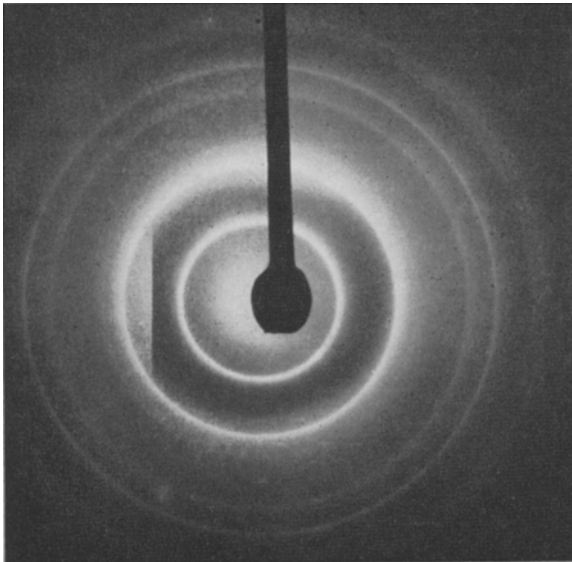


PLATE 2B.—Electron diffraction diagram from a large field of particles collected as for Plate 2A. The rings, reading successively from the center are: 1.0; 1.1; 2.0; 2.1; 3.0; 2.2 and 3.1 by hexagonal designation, with 3.0 equal to the conventional 060 monoclinic *b*-axis node.

on the high angle side. Upon glycolation the modification to the first order feature becomes more prominent, and to the second order feature less prominent. The records reproduced in Fig. 1 illustrate the relationships. The first maxima in both the water-solvated and the glycol-solvated states indicate an average of one layer in five in the solvated condition. The sharpened second maximum in the glycolated state is a consequence of the near coincidence of a second node for a 7.2Å (halloysite) layer with a third node for a 10.8Å (glycol-halloysite) layer.

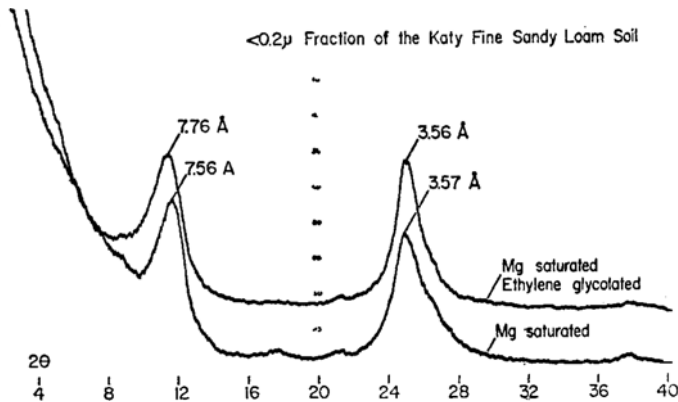


FIGURE 1.—X-ray diffractometer records of diffraction from an orientated aggregate of  $<0.2\mu$  Katy soil fraction before and after solvation with ethylene glycol.

The only morphology which can afford exclusively  $hk0$  diffraction features when oriented aggregates are viewed normal to the aggregate flake and exclusively  $001$  features when the same aggregates are examined by X-ray diffractometry is that of tabular individuals. For any aggregate of the familiar tube morphology of halloysite, either technique provides mixed sets of the two kinds of diffraction features.

The structural aspects inferred from diffraction data are supported by differential thermal and infrared adsorption records. The DTA trace shown in Fig. 2 includes a low-temperature water effect, the characteristic dehydroxylation feature for a kaolin mineral, and the characteristic high temperature exotherm. The attenuation of the exotherm is the consequence of the base saturations noted.

The infrared record (Fig. 3) consists of two major O-H stretching frequency absorption bands at  $3690$  and  $3620\text{ cm}^{-1}$ , with less important absorption between them; a broad antisymmetric stretching absorption band at about  $3430\text{ cm}^{-1}$  and a deformation band at  $1640\text{ cm}^{-1}$  for molecular water; and an O-H flexion band at  $915\text{ cm}^{-1}$  for the hydroxyls bound to two aluminums. The first three and the latter features are all

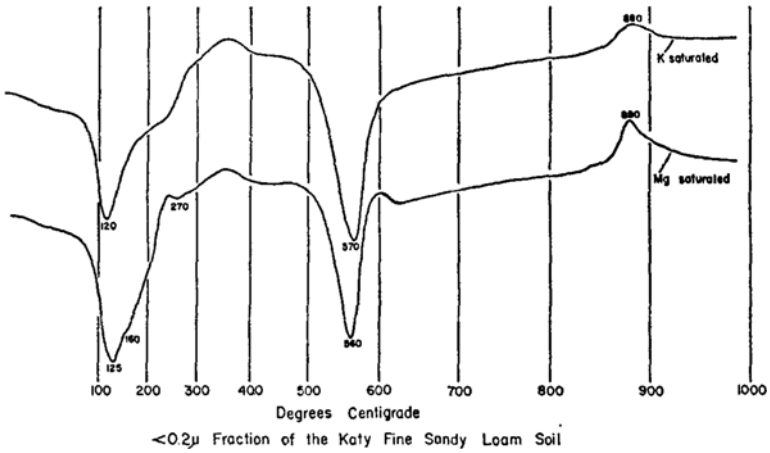


FIGURE 2.—Differential thermal analysis records for  $K^+$  and  $Mg^{2++}$  saturated  $<0.2\mu$  fractions of Katy soil clay.

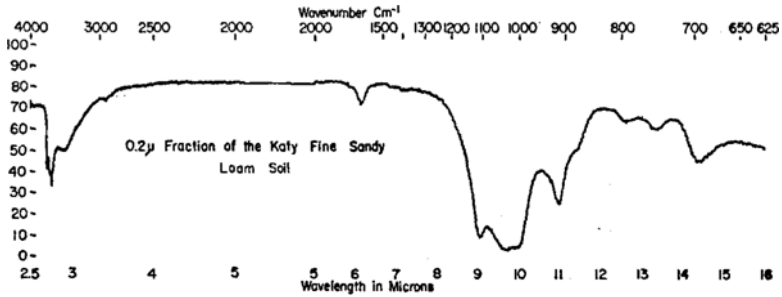


FIGURE 3.—Infrared absorption spectrum of Katy soil clay in a KBr pellet.

known vibrations in a kaolin mineral assemblage. An additional feature, a flexion in the curve at about  $870\text{ cm}^{-1}$ , has a frequency comparable with that for which flexion of OH for a hydroxyl bound to one aluminum and one iron has been suggested (Serratosa, 1960). The other features are all typical for the tetrahedral and Si-O-Al features characteristic of the kaolin minerals. No extra feature attributable to the presumed iron content is apparent, other than the cited modified OH flexion frequency.

The several avenues which have been explored in seeking characterization of the described soil clay seem to lead in concert to the conclusion that a tabular habit for the halloysite condition of the kaolin mineral structural unit exists. The presence of iron (and/or magnesium) in the structure is indicated by the analysis, and presumably by the habit and by the infrared

absorption character. The lateral dimensions of several hundredths of an angstrom greater than those of ideal kaolinite are qualitative in agreement with observations on layer silicates by Radoslovich (1962, p.620) which include cases where well-crystallized layer minerals with iron populations near 1 in 6 are a few hundredths of an angstrom greater in  $b_0$  parameters than are their low iron analogous.

## REFERENCES

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