Clay Flake Orientation in Flocculated Illite- An Electron Microscope Study*

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Introduction

This paper presents electron micrographs showing the fabric of uncompressed illite clay floccules. The purpose of the research was to test the validity of the honeycomb model (Terzaghi, 1925) and cardhouse model (Tan, 1958) as applied to the actual fabric of laboratory prepared flocculated clay.

Illite was chosen for this study since it is the dominant clay mineral in most argillaceous sediments. Grundite illite flakes which were used were observed to be curved slightly, and possess an irregular outline. Borst and Keller (1969) attribute the warping of the basal surface of grundite illite to interstratification with montmorillonite. The curving of the flakes is important to this fabric study since it influences the orientation of flakes in an illite floccule.

Procedure

The clay was flocculated under both slightly saline and salt-free conditions. The resultant floccules were observed in both a scanning and ultra high-voltage electron microscope. A freeze drying technique was used to prepare the illite floccules for SEM viewing. One hundred grams of illite were mixed in 1 litre of distilled water and completely disaggregated. Flocculation occurred due to the high clay concentration. In the saline-flocculation experiment, 5 grams of illite were first dispersed in a litre of distilled water containing 5 ml. of NH_4OH. After complete dispersion was insured, this solution was mixed with a salt solution--final concentration being illite 5 grams/litre and NaCl 1 gram/litre. Drops of the flocculated masses were put onto small sample holders and submerged into liquid isopentane at -145°C. Frozen samples were then put into a vacuum chamber and the ice removed by sublimation. The dried powdery clay skelton was then gold shadowed for viewing.

The same samples were also used for study with the high voltage (500KV) transmission electron microscope. To prepare the clays for study with this unit, they first had to be impregnated with polyethylene glycol No. 6000. Flocculated clay was allowed to settle to the bottom of a 100 ml. beaker and the cleared water was siphoned off. Then the molten polyethylene glycol No. 6000 was slowly poured over the clay so as not to mix it. The samples were put into an oven at 70°C and cured for two weeks. When removed, the wax hardened and 1μ-thick microtome thin sections were prepared for viewing directly in the microscope. Martin (1966) has used this technique for x-ray diffraction analysis of moist clay fabric and concluded it is quite adequate to give a representative view of the undisturbed clay fabric. The reader will observe that the illite fabric (Figures 1-4) prepared by both freeze-drying and impregnation is very similar thus suggesting that each technique is useful in preserving the gross original fabric of the floccules.

Fabric of illite floccules

Figures 1-2 show the typical fabric produced by freeze-drying illite flocculated in distilled water. The major observation is that there is an open network of flakes oriented at various angles. Flakes appear to be aggregated into domains or clusters consisting of much face to face orientation (Figure 2). More individual face-edge orientation is seen in certain areas in the salt-flocculated illite clay masses (Figure 3); however, other areas in the same samples revealed an abundance of face-to-face flocculation (Figure 4). It may be possible that the face-to-face flocculation seen in all samples may be only an apparent orientation due to the curvature of the flakes. The flakes could have flocculated in an edge-face manner but inclined at a very small angle to each other (not at 90° to 45° as seen in some cardhouse models) and still give the general impression of parallel or sub-parallel flakes due to their curvature. This orientation may simply reflect the attraction of the positive clay edge for a negative flake surface as described by Tan (1958). Alternately, it may be possible that one is observing actual stepped face-to-face flocculation similar to that found to exist in freeze-dried kaolinite floccules (O'Brien and Suito, 1969). It does seem possible that under the laboratory conditions of flocculation (high clay concentration or an electrolyte solution) the double layer on each clay flake may be compressed, thus increasing in importance van der Waal forces of attraction--a likely mechanism to promote face-to-face flocculation.

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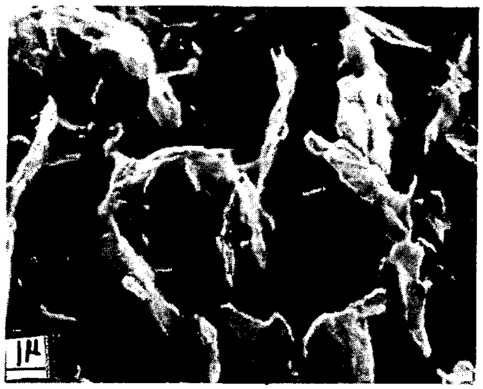

Figure 1 - Scanning electron micrograph of freeze-dried grundite illite floccules. Flocculated in distilled water. x 8000.

Figure 2 - Scanning electron micrograph of freeze-dried grundite illite floccules. Flocculated in distilled water. x 10000.

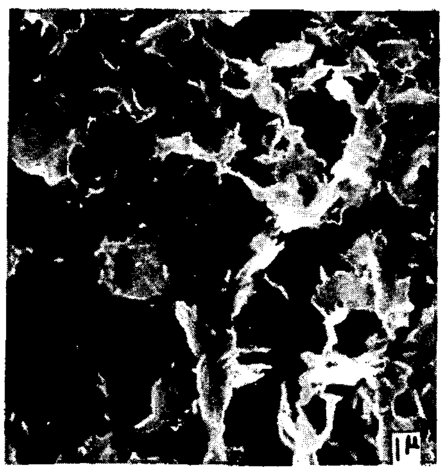

Figure 3 - Scanning electron micrograph of freeze-dried grundite illite floccules. Flocculated in salt water (Na Cl 1 gm/l.) x 6000.

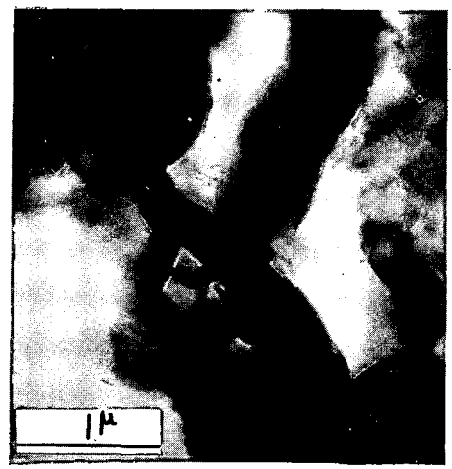

Figure 4 - High voltage transmission electron micrograph of flocculated grundite illite impregnated with polyethylene glycol. Flocculated in salt water (Na Cl 1gm/l.)x28000

Conclusion

The fabric of uncompressed illite floccules formed under saline and salt-free conditions in the laboratory consists of an open porous network mainly consisting of randomly oriented domains or clusters of apparent stepped face-to-face flocculated flakes. The exact reasons for the parallel or sub-parallel orientation of the flakes are not known, although this orientation may indicate the importance of van der Waal attractive forces under the experimental conditions. The results of these experiments are in agreement with the gross fabric assumed by others to be present in the honeycomb or cardhouse models of clay floccules; however, detailed study indicates more face-to-face flocculation than previously assumed.

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