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FABRIC OF KAOLINITE AND ILLITE FLOCCULES

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Abstract—A scanning and high voltage electron microscope study of the fabric of laboratory sedimented moist uncompressed kaolinite and illite floccules reveals an abundance of face–face flake orientation in the porous clay network. Clays were flocculated in the laboratory in both distilled water and slightly saline (1 g/l. NaCl) water using various clay concentrations. Floccules taken from the moist clay mass were prepared for study by freeze-drying and impregnation with polyethylene glycol.

There is little difference in the fabric of kaolinite flocculated in distilled or slightly saline water. The fabric is dominated by a 3-dimensional network of twisted chains of face–face oriented flakes having the appearance of a stair-stepped cardhouse. Illite floccules in distilled water also consist of abundant face–face oriented overlapping flakes. However, in salt water there is a more even mixture of fabrics—edge-to-face flocculation of individual platelets and also stepped clusters of face-to-face oriented flakes, the latter being more abundant.

It is suggested that under the experimental conditions the double layer of each clay particle is compressed resulting in an increase in the importance of van der Waals forces of attraction. As a result flakes approach each other and rotate into a parallel or subparallel position. The resultant dominant fabric is that of a stepped cluster of overlapping flakes.

INTRODUCTION

INVESTIGATION of the original fabric of flocculated clay should help in understanding changes that natural sediments undergo upon compaction (a phenomena of interest to sedimentologists) and in determining reasons for certain soil properties (features of interest to geological engineers). Since very few direct observations with the electron microscope have been made of the original undisturbed clay flake orientation in flocculated clay, it was felt that an investigation of this type would be of value. This paper presents electron micrographs of the fabric of laboratory prepared unconsolidated kaolinite and illite floccules.

Idealized drawings have been made of hypothetical models of clay floccules (Schofield and Samson, 1954; Van Olphen, 1963, p. 94) and photographs have been published of the fabric of recent sediment assumed to have been flocculated when deposited. It is not surprising, however, that very little attention has been paid to photographing the original fabric of isolated loose floccules themselves due to the difficulty of sample preparation and the lack of appropriate research tools. With the development of the scanning electron microscope and high voltage electron microscope the tools have become available to take electron micrographs and to test the validity of the commonly accepted clay floccule models.

Many students of clay fabric have limited their research to the investigation of clay flake orientation in natural sediment, which may have undergone some consolidation and clay flake reorientation. Others have based their conclusions upon observations of flocculation phenomena and have proposed hypothetical models inferred from these results. The earliest significant discussion of clay fabric was that of Karl Terzaghi in 1925. He discussed mainly the structure of cohesive soils in which he felt cohesion was due to adhesion between adjacent minerals. Fine and coarse grained muds were believed to be composed of a loose aggregate of grains resembling cells of a honeycomb.

Goldschmidt (see Rosenqvist, 1959) expressed a view that flaky minerals are arranged in a cardhouse structure in sensitive clays. Casagrande (1940) also presented a theory of a honeycomb structure in soils. Idealized drawings by Lambe (1953) showed the microstructure of clays and suggested also a cardhouse fabric in marine clay. Tan (1958) presented an idealized drawing of clay fabric which showed a network of edge–face oriented flakes in clayey sediment. In fabric research, no actual photographs were taken of clay flakes in undisturbed sediments until Rosenqvist (1959, 1963) published electron micrographs of replicas of freeze-dried samples of undisturbed marine Oslo blue clay which led him to support the validity of the cardhouse arrangement in undisturbed sediment.

The electron microscope also proved a valuable tool to a number of other students of the fabric of Pleistocene and recent unconsolidated sediment

(O'Brien and Harrison, 1967; Gillott, 1969). Pusch (1962, 1966, 1968) studied in the electron microscope ultra-thin sections of methacrylate and carbowax impregnated soft natural clays. He concluded that the structure of flocculated sediment is characterized by a network of large particles tied together by small particles arranged in groups or chains. His hypothetical floccule (Pusch, 1962, p. 53) showed individual flakes oriented in both a edge–face and edge–edge manner. Bowles (1968) studied marine sediment from the Gulf of Mexico using techniques similar to Pusch's. He stated that it is unreasonable to conclude that the microstructure of undisturbed marine sediment resembles only a cardhouse or a honeycomb although it does appear to resemble more closely Terzaghi's honeycomb idealization. Later work by Bowles, Bryant and Wallin (1969) suggested that unconsolidated sediment may also be composed of packets of parallel oriented particles, although their results were inconclusive.

Van Olphen (1963, pp. 94–95) summarized modes of particle association in flocculated clay suspensions and appropriate terminology which is a useful frame of reference in this discussion. The phenomenon of flocculation is observed when particles in suspension begin to stick together upon collision, and the agglomerates grow in size and settle rather quickly. Three different modes of particle association may occur: face–face, edge–face, and edge–edge. The term "aggregated" is suggested for the face to face association of several flakes. Thus individual flakes may be flocculated in an edge–edge and edge–face association, while aggregates (each composed of face–face associated flakes) may also be edge–edge or edge–face flocculated. These models resemble the typical clay cardhouse. Later the author will elaborate upon the validity of these models as a result of his study.

PROCEDURE

Two different sample preparation techniques were used to study floccule fabric:freeze-drying and polyethylene glycol No. 6000 impregnation. The freeze-dried samples were observed directly in the scanning electron microscope and ultrathin sections of the impregnated samples studied with the high voltage electron microscope. A freeze-drying technique has also been used to study the fabric of bentonite muds by Borst and Shell (1970). Moist samples were frozen on aluminum specimen stubs cooled with liquid nitrogen and then observed with the SEM. X-ray analysis revealed random orientation present in the freeze-dried clay, therefore, they concluded, that in quick freezing ice formed in the vitreous state rather than as crystals, thereby preventing change in the fabric. Halberstadt *et al.* (1969) studied samples of silica and gelatin gels prepared by a liquid nitrogen freeze-dried technique and concluded "the gels were not damaged by this procedure and the silica structure remained essentially intact". Gillott (1969) has suggested, however, ice crystals have more chance to form if moist samples are immersed directly in liquid nitrogen. To minimize the formation of ice crystals, he proposed freezing samples by immersion in a vessel containing isopentane surrounded by liquid nitrogen in an outer container, resulting in rapid freezing at a temperature below −130°C. Gillott mentioned that since sublimation is normally carried out at higher temperatures, it is hoped that the fabric of the clay prepared in this manner impedes recrystallization sufficiently to prevent serious damage from the growth of ice crystals.

The material used for impregnation was similar to that used and described by Mitchell (1956) as Carbowax. He studied the fabric of impregnated wet clays as observed in thin sections and concluded that the technique gave thin sections that contained particles in the same position as in the original wet clay. Thus this author concluded that this technique would be a useful method to check the validity of clay fabric studied by the freeze drying technique.

The clay fabric was observed to be very similar in clay prepared by these two different techniques, suggesting (1) that if any changes are produced they are identical for both alternative preparation procedures; or (2) neither preparation technique significantly altered the original floccule structure. The latter is assumed to be the case based primarily upon fabric similarities of the same clays prepared by the widely different techniques. Interpretations are based upon the validity of this assumption.

Freeze-drying technique

Samples of Georgia kaolinite (Hydrite PX) and Grundite illite were flocculated in distilled and slightly saline water. 50 g of kaolinite and illite each were stirred into separate beakers each containing a liter of distilled water and then mixed in a blender for 5 min. Observation of the clay suspension under a binocular microscope indicated complete disaggregation; however, upon standing large floccules formed. 100 g of kaolinite and illite were each stirred into separate jars containing distilled water and shaken by hand for 10 min. 10 g of kaolinite and 5 g of illite also were dispersed in 500 ml of distilled water (containing 20 ml dil. NH_4OH) and mixed for 5 min in a blender. The latter suspensions stood for one day in the

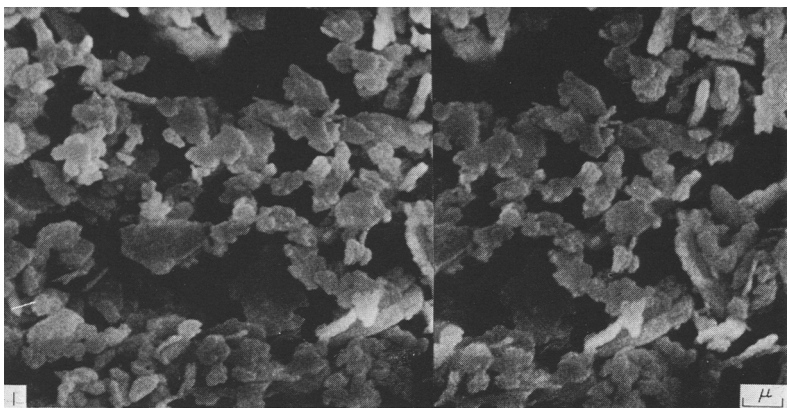

Fig. 1. Stereo-pair electron micrograph of freeze-dried flocculated Georgia kaolinite (50 g/l. clay concentration in distilled water). Stepped face–face and edge–face flocculation is present. Scale 8000 ×.

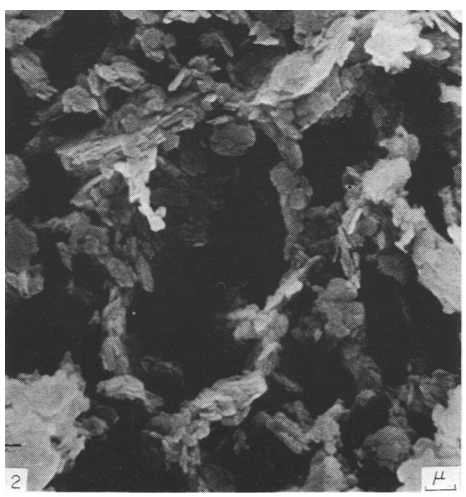

Fig. 2. Scanning electron micrograph of freeze-dried flocculated Georgia kaolinite (100 g/l. clay concentration in distilled water). Note long twisted chains of face–face flocculated flakes. Scale 6000 ×.

Fig. 3. Scanning electron micrograph of freeze-dried flocculated Georgia kaolinite (100 g/l. clay concentration in distilled water). Points *x* and *y* show face–face oriented flakes: Point *z* shows an area of edge–face flocculation. Scale 15,000 ×.

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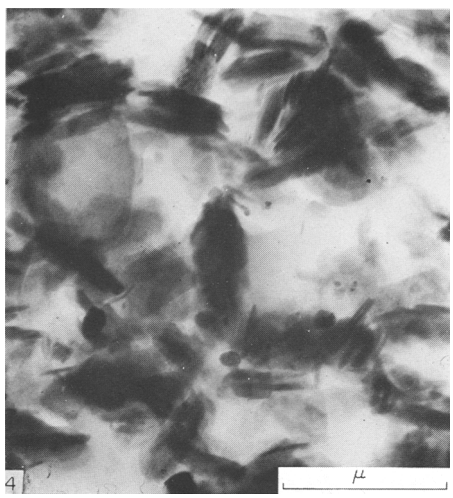

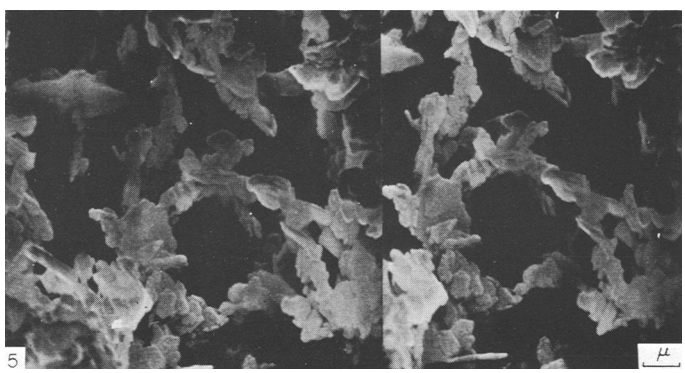

Fig. 4. High voltage electron micrograph of polyethylene glycol impregnated flocculated Georgia kaolinite (50 g/l. clay concentration in distilled water). Notice porous fabric of the flocculated clay. Scale 33,000 ×.

Fig. 5. Stereo-pair scanning electron micrograph of freeze-dried flocculated Georgia kaolinite (10 g/l. clay concentration in salt water, 1 g/l. NaCl). Note stepped clusters of face–face oriented flakes in the center of photograph. Scale 8000 ×.

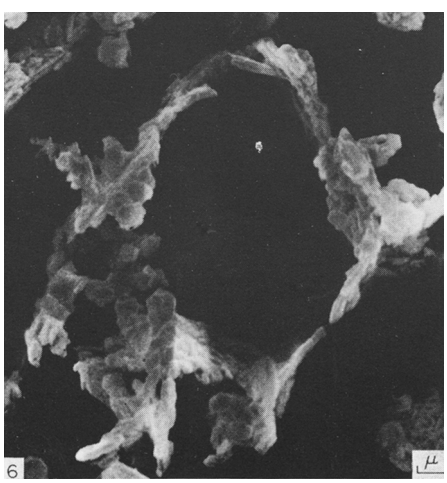

Fig. 6. Scanning electron micrograph of freeze-dried flocculated Georgia kaolinite (10 g/l. clay concentration in salt water, 1 g/l. NaCl). Notice long chains composing the floccule sides. Scale 6000 ×.

Fig. 7. High voltage electron micrograph of polyethylene glycol impregnated flocculated Georgia kaolinite (10 g/l. clay concentration in salt water, 1 g/l. NaCl). Scale 45000 ×.

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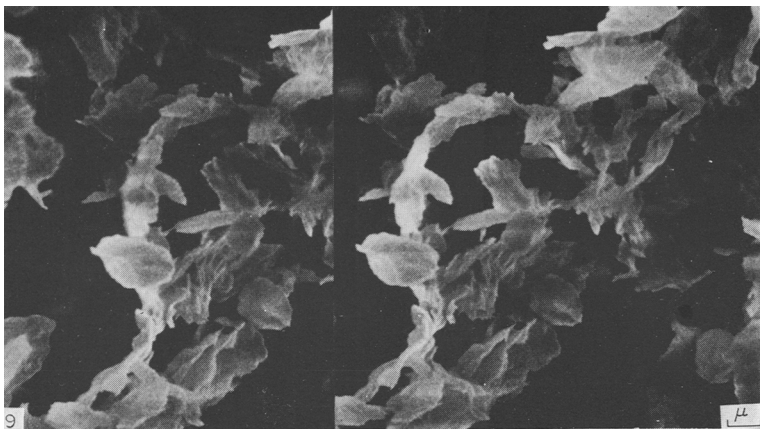
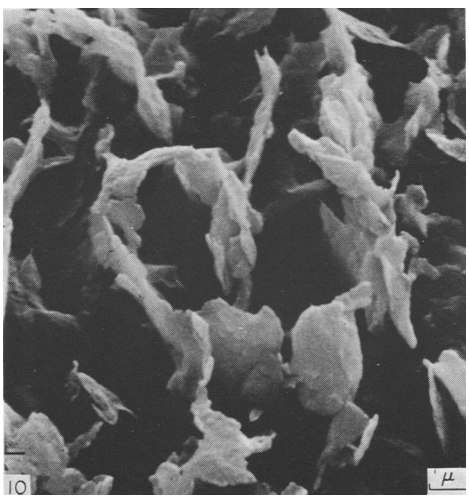


Fig. 9. Stereo-pair scanning electron micrograph of flocculated grundite illite (50 g/l. clay concentration in distilled water). Scale 6000 ×.



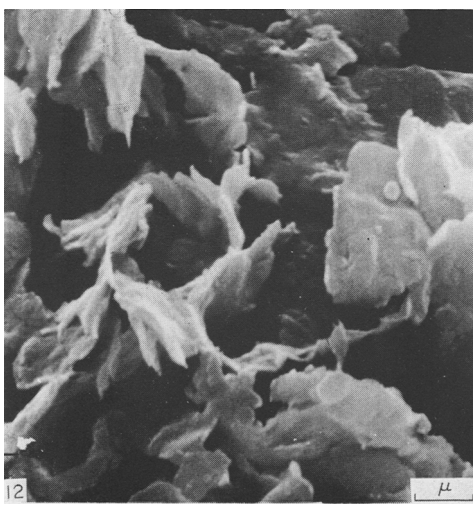


Fig. 10. Scanning electron micrograph of freeze-dried flocculated grundite illite (100 g/l. clay concentration in distilled water). Scale 8000 ×.

Fig. 12. Scanning electron micrograph of freeze-dried flocculated grundite illite (100 g/l. clay concentration in distilled water). Scale 10,000 ×.

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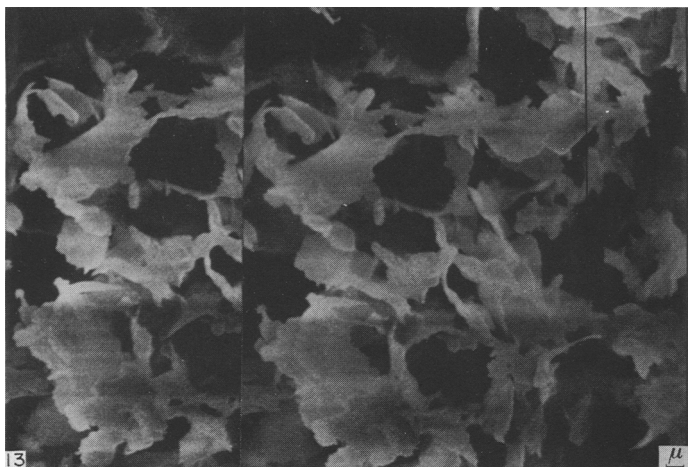

Fig. 13. Stereo-pair scanning electron micrographs of freeze-dried flocculated grundite illite (5 g/l. clay concentration in salt water, 1 g/l. NaCl). Scale 8000 ×.

Fig. 14. High voltage electron micrograph of polyethylene glycol impregnated flocculated grundite illite (5 g/l. clay concentration in salt water, 1 g/l. NaCl). Scale 12,800 ×.

Fig. 15. High voltage electron micrograph of polyethylene glycol impregnated flocculated grundite illite (5 g/l. clay concentration in salt water, 1 g/l. NaCl). Scale 12,800 ×.

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Table 1. Clay and NaCl concentrations used in fabric study

Clay concentration (g/l.)		NaCl concentration (g/l.)	pH
Georgia kaolinite	50	0 (distilled water)	5·0
	100	0 (distilled water)	7·8
	10	1	8·4
Grundite illite	50	0 (distilled water)	7·1
	100	0 (distilled water)	4·5
	5	1	10·2

laboratory and were observed to be well dispersed. Then each clay suspension was mixed with an equal amount of NaCl solution so that the final concentration equaled 1 g NaCl per liter of distilled water. Table 1 summarizes final clay and salt concentrations plus pH values of the final suspensions used in the experiments.

A drop of each flocculated clay suspension was then deposited on a small 7 mm dia. aluminum disc producing a sphere. Each sample was frozen quickly by being submerged slowly into a container of liquid isopentane (temperature −140°C) surrounded by liquid nitrogen in an outer container. Then frozen samples were put into a cold vacuum chamber and the ice removed by sublimation under a vacuum of approximately 7×10^{-1} mm Hg for 4 hr. During this time the chamber was kept at −15° to −20°C in a salt–ice mixture. The dried powdery samples were removed and gold shadowed (200–300 Å thick) to prepare them for scanning electron microscope study. A JEOL, JSM-2 microscope was used in this study.

Polyethylene glycol technique

The same flocculated clay suspensions described in the previous section (Table 1) were also used in preparing the clay for the high voltage electron microscope investigation. Others (Martin, 1966; Quigley and Thompson, 1966), have used polyethylene glycol No. 6000 for X-ray diffraction analysis of moist clay fabric. The flocculated clay masses were poured into small beakers and allowed to settle for 1 hr. Most of the clear overlying water was siphoned off, leaving only a few millimeters of clear water at the sediment–water interface. Then warm liquid polyethylene glycol No. 6000 (melting point 55°–59°C) at 70°C was carefully poured down the side of each beaker so as not to stir up the clay. Beakers were put into an oven, loosely covered with aluminum foil lids and kept at 70°C for over 2 weeks, during which time the moist clay became entirely impregnated with the wax. Then the beakers were removed and the wax impregnated samples were allowed to harden at room temperature. Thin sections (1 μ thick) were prepared by using a microtome, and mounted on 200 mesh copper screens for viewing.

Because of the thickness of the sections, it was necessary to use for viewing a 500 kV high voltage electron microscope developed by the Shimadzu Seisakusho Co. of Kyoto, Japan (Kobayashi *et al.* 1964). This unit allowed greater beam penetration with minimal sample damage. The author observed that even at 500 kV the impregnated samples were stable in the beam and considerable fabric detail was revealed.

RESULTS AND DISCUSSION

Fabric of kaolinite floccules

Experimental results obtained by this author suggest the existance of edge–face and face–face flocculation produced in the kaolinite samples. Generally, the gross fabric of kaolinite floccules formed in distilled water consists mainly but not exclusively, of domains or groups of face–face flocculated flakes arranged in a stair-step manner in each domain, which are joined to each other commonly in an edge–face manner (Figs. 1 and 2). This fabric corresponds to the idealized "edge-to-edge, edge-to-face and aggregated" model suggested by Van Olphen (1963, p. 94). Randomly oriented stepped clusters showing the top and side views (Fig. 3 points *x*, *y*) of aggregates of face–face attached kaolinite flakes and edge–face orientation of domains (point *Z*, Fig. 3) are also observed. Kaolinite clay prepared by the impregnation technique also shows edge–face and face–face flocculation (Fig. 4). Figure 4 also demonstrates that flocculated kaolinite fabric actually does consists of a porous network of flakes. Furthermore, results indicate little difference in the gross fabric of kaolinite floccules formed in distilled water with a high clay concentration and in salt water with a lower clay concentration (compare Figs. 5 and 1; Figs. 6 and 2; Figs. 7 and 4). The fabric in both cases consists of abundant stepped clusters of face–face flocculated flakes with some zones of edge–face oriented platelets all randomly arranged in a very porous 3-D network. It is suggested that this fabric may be a consequence of the compression of the double layer of each flake under experimental conditions thus allowing van der Waals forces of attraction to increase in importance, hence the promotion of face–face orientation.

The experiments with flocculated kaolinite suggest two important conclusions: (1) under conditions of either a large clay concentration in distilled water or low clay concentration in an electrolyte solution, repulsive forces between the flakes seem to be minimized so that the flakes can attract to each other mainly in a face–face manner,

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primarily because van der Waals attractive forces predominate; (2) and most importantly, a 3-D gel network of twisted interlocking chains of face–face oriented flakes is built up in an attempt to establish equilibrium in the system; resulting in an agglomeration of kaolinite flakes with a large void ratio (Fig. 8).

Fabric of illite floccules

Illite floccules show various types of fabric existing in the same sample. The difference in fabric compared to kaolinite may be due to the shape and crystallinity of the clay minerals involved. Kaolinite platelets are flat, hexagonal-shaped, and highly crystalline. Grundite illite flakes are commonly curved, have an irregular outline, and have less well developed crystallinity. Borst and Keller (1969) show this morphology in their electron micrographs and attribute the warping of the basal surface of illite to interstratification with montmorillonite. The curving of the flakes is important to the illite fabric study since it seems to influence the orientation of the flakes in a floccule.

Figures 9 and 10, show a sub-parallel to parallel flake orientation apparent in illite floccules formed in distilled water. Figures 9 and 10 may indicate face to face flocculation (as in the case of kaolinite). However, this may only be an apparent orientation due to the curvature of the illite platelets. It is also possible that the flakes flocculated in an inclined edge–face manner and give the impression of parallel orientation (Fig. 11). This orientation could also be interpreted as simply reflecting the attraction of a positive clay edge for a negative surface.

Other samples of sub-parallel to parallel flake orientation were observed, however, which suggest more positively a primary face–face orientation. Figure 12 shows stepped clusters of curved illite flakes standing perpendicular to the photo plane and arranged in a pinwheel fashion (compare to Fig. 11). The close packing of the parallel flakes and their stair-step arrangement suggests they were originally associated in this face–face orientation and not in an edge–face manner in the moist floccule.

Freeze-dried fabric of salt water flocculated illite is also seen in Fig. 13. Figure 13 reveals typical edge–face cardhouse orientation and also an overlapping face–face fabric present in the same small area. The same sample prepared by the polyethylene glycol treatment also shows a mixture of fabrics; individual edge–face orientation (Fig. 14) and domains consisting of face–face fabric (Fig. 15).

Results of the illite experiments suggest a mixture of fabrics present in the same sample and

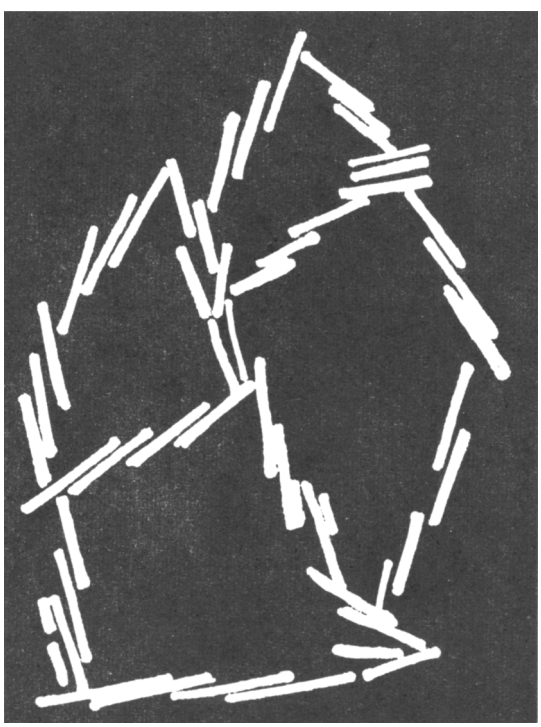

Fig. 8. Stairstep cardhouse fabric of flocculated kaolinite.

Fig. 11. Model of illite floccule in distilled water. Model is based upon the interpretation of illite fabric in Fig. 10.

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very little difference in the fabric of floccules formed under saline and salt free conditions.

The author feels that the same explanation used to describe the reasons for the fabric of kaolinite floccules could also apply to illite floccules. The main difference being that due to the curvature of illite, the parallelism of face–face flocculated flakes is not as well developed as in kaolinite. After the illite flakes contact each other, they tend to assume a more parallel position due to van der Waals attractive forces but they cannot become perfectly parallel due to their curvature. Therefore, they assume an orientation slightly different from the flat kaolinite flakes.

CONCLUSION

This study of the fabric of clay floccules prepared by different experimental techniques and studied by means of scanning and high voltage electron microscopes reveals that edge–face and face–face flocculation is common in clay aggregates. There appears to be little difference in the fabric of either kaolinite or illite floccules formed in saline or salt free water. Experiments indicate a slightly saline solution (NaCl 1 g/l.) promotes flocculation and produces a fabric similar to distilled water flocculation. It is suggested that possibly under the experimental conditions the double layer was compressed with a resultant increase in importance of the van der Waals forces of attraction. In this situation flakes would approach each other in a parallel (in the case of flat flakes like kaolinite) or sub-parallel position (in the case of warped illite flakes) and become oriented mainly in stepped clusters.

Generally the idealized honeybomb model proposed by Terzaghi in 1925 seems to be more accurate than the cardhouse model proposed by Casagrande (1940), Tan (1958) and others. In no samples studied, however, was the fabric as simple as that shown in the model by either Tan or Terzaghi. The closest similarity to the Tan model was seen in illite flocculated in salt water (Fig. 13) although other areas in the same sample show abundant face–face orientation, similar to the Terzaghi model.

The main contribution of the experiments is the light shed on the gross fabric of flocculated clay sediment. An uncompressed flocculated kaolinite and illite mass does consist of a very porous network of randomly oriented flakes or clumps of flakes. Naturally in such a system it is difficult to isolate one floccule, since it is part of an extended network. However under conditions described it is suggested that this one unit in kaolinite commonly appears to be composed of numerous face–face flocculated flakes arranged in a cluster in a stair-step fashion. These domains, in turn, may be oriented at all angles and attached in an edge–face manner to other domains. The sides of this one unit in illite, however, may be composed either of one flake or several stepped face–face oriented flakes.

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Résumé—L'étude par microscopie électronique à balayage et sous haute tension de la texture de floculats de kaolinite et d'illite, humides, non comprimés et sédimentés au laboratoire, révèle une grande abondance de plaquettes orientées face à face dans le réseau argileux poreux. Les argiles ont été floculées au laboratoire à la fois dans l'eau distillée et dans de l'eau légèrement saline (1 g/l. NaCl), à différentes concentrations en argile. Les floculats prélevés dans la masse argileuse humide ont été préparés pour l'étude ultérieure, par cryodessiccation et imprégnation au polyéthylène glycol.

Il y a peu de différences entre la texture de la kaolinite floculée dans l'eau distillée et celle de la kaolinite floculée dans l'eau légèrement saline. La texture est essentiellement constituée par un réseau tridimensionnel de chaines torsadées de plaquettes orientées face à face, ayant l'apparence d'un paquet de cartes incomplètement étalé, en marches d'escalier. Les floculats d'illite obtenus dans l'eau distillée sont constitués également par un grand nombre de plaquettes orientées face à face, qui se chevauchent. Cependant, dans l'eau salée, il y a un mélange plus uniforme de textures dues à la floculation de plaquettes individuelles bord à face et aussi d'agrégats en marches d'escalier dûs à des plaquettes orientées face à face, ces derniers étant plus abondants.

Il est suggéré que dans les conditions expérimentales de ce travail, la double couche de chaque particule d'argile est comprimée, ce qui entraîne un accroissement de l'importance des forces d'attraction de Van der Waals. En conséquence, les plaquettes se rapprochent mutuellement, et tournent pour se mettre dans une position parallèle ou subparallèle. La texture dominante qui en résulte est celle d'agrégats en marches d'escalier, formés de plaquettes qui se chevauchent.

Kurzreferat—Eine Untersuchung mittels Abtastungs- und Hochspannungselektronenmikroskopie des Gefügen von im Laboratorium abgeschiedenen, feuchten, unverdichteten Kaolinit- und Illitflocken zeigt eine flächenmässig gegenüberliegende Orientierung der Blättchen in der porösen Tonstruktur an. Die Tone wurden im Laboratorium in destilliertem Wasser sowie in schwach salzhaltigem (1*g/l*, NaCl) Wasser unter Verwendung verschiedener Tonkonzentrationen ausgeflockt. Die aus der feuchten Tonmasse genommenen Flocken werden durch Gefriertrocknung und Imprägnierung mit Polyäthylenglykol für die Untersuchung vorbereitet.

Der Unterschied zwischen dem in destilliertem Wasser ausgeflockten Kaolinit und dem aus schwach salzhaltigem Wasser ist gering. Vorherrschend im Gefüge ist ein dreidimensionales Netzwerk verdrehter Ketten von flächenmässig gegenüberliegend orientierten Blättchen, die das Aussehen eines stufenartigen Kartenhauses geben. Die Illitflocken im destillierten Wasser bestehen ebenfalls grösstentails aus flächenmässig gegenüberliegenden, überlappenden Blättchen. In Salzwasser hingegen findet sich eine ausgeglichenere Mischung von Gefügen: Kante-gegen-Fläche Flockung von Einzelblättchen und stufenartige Büschel von flächenmässig gegenüberliegenden Blättchen, wobei letztere überwiegen.

Es wird als möglich angesehen, dass unter den Versuchsbedingungen die Doppelschicht jedes Tonteilchens zusammengepresst wird wodurch sich eine Zunahme im Umfang der van der Waalsschen Anziehungskräfte ergibt. Demzufolge nähern sich die Blättchen einander und drehen sich in eine parallele oder nahparallele Position. Das sich ergebende, vorherrschende Gefüge weist stufenförmige Anhäufungen sich überlappender Blättchen auf.

Резюме — Исследование в сканирующем и высоковольном электронных микроскопах строения осажденных в лаборатории влажных, не подвергавшихся действию давления каолинитовых и иллитовых хлопьев обнаружило преобладание в пористом глинистом каркасе ориентировки частиц типа поверхность к поверхности. Глины в различных концентрациях подвергались флокуляции в лабораторных условиях как в дистиллированной воде, так и в слабом растворе

NaCl (1 г/л). Агрегаты, выделенные из влажной глинистой массы, были приготовлены для выусшивания замораживанием и насыщения полиэтилен-гликолем.

Наблюдались лишь незначительные различия в строении каолинита, подвергшегося флокуляции в дистиллированной воде и в слабом растворе NaCl. Структура такого каолинита представляет трехмерный каркас из скрученных цепей, состоящих из ориентированных поверхность к поверхности чешуек; каркас имеет вид ступенчатого карточного домика. Агрегаты иллита в дистиллированной воде также состоят из преимущественно ориентированных поверхность к поверхности перекрывающихся чешуек. Однако в растворе NaCl наблюдалась более однообразная смесь структур типа ребро к поверхности отдельных пластинок, а также ступенчатые группы ориентированных поверхность к поверхности частиц, причем последних было больше.

Предполагается, что в условиях эксперимента двойной слой каждой глинистой частицы сжимается в результате увеличения влияния ван-дер-ваальсовых сил притяжения. Вследствие этого частицы сближаются и занимают параллельные или субпараллельные положения. Результирующая преобладающая структура представляет собой ступенчатую группу из перекрывающихся пластинок.